CHARACTERISATIONS THE BIO-ACTIVE COMPOUNDS OF BIO-OIL EXTRACTED FROM RED MERANTI SAWDUST BY FAST PYROLYSIS

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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 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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Thesis submitted in fulfillment of the requirements
for the award of the degree of
Doctor of Philosophy

Faculty of Engineering Technology

UNIVERSITI MALAYSIA PAHANG

AUGUST 2018

ACKNOWLEDGEMENTS

All praises to Allah S.W.T, The Almighty God and The Lord of the Universe, The Merciful and Gracious for His mercy has given me the strength, blessing and time to complete this study. Salam to our beloved prophet, Nabi Muhammad s.a.w.

Special thanks to my sponsors, Universiti Malaysia Perlis (UniMAP) and Ministry of Higher Education (KPT) for their financial support throughout my study.

I am grateful and would like to express my sincere gratitude to my supervisor Professor Dato' Dr. Zularisam bin Ab Wahid for his germinal ideas, invaluable guidance, continuous encouragement and constant support in making this research possible. He has always impressed me with his outstanding professional conduct, his strong conviction for science, and his belief that a PhD program is only a start of a life-long learning experience. I appreciate his consistent support from the first day I applied to graduate program to these concluding moments. I am truly grateful for his progressive vision about my training in engineering technology, his tolerance of my naïve mistakes, and his commitment to my future career. I also sincerely thanks for the time spent proofreading and correcting my many mistakes.

It gives me great pleasure to acknowledge to the guidance, valuable suggestions, constructive criticism and incredible patience of my co-supervisor, Professor Datin Dr. Mimi Sakinah binti Abdul Munaim. Your scientific excitement inspired me in the most important moment of making right decisions and has significantly contributed to this thesis. Thanks you for trusting me.

My sincere thanks go to all my lab mates and members of the staff of Faculty of Engineering Technology Department, UMP, who helped me in many ways and made my stay at UMP pleasant and unforgettable. Many special thanks go to member research group for their excellent co-operation, inspiration and support during this study.

Finally, I acknowledge my sincere indebtedness and gratitude to my beloved husband and my child, my parents and sibling for their love, dream and sacrifice throughout my study and life. I cannot find the appropriate words that could properly describe my appreciation for their devotion, support and faith in my ability to attain my goals. Special thanks should be given to my friends, lab mates and postgrad roommates with their supports, comments, suggestions and knowledge, which has crucial for the successful completion of this study. For the name mentioned and not mentioned that have involved throughout my study, only Allah could pay your kindness. Last but not least, I hope this study would give a beneficial input in this field especially in conducting the future study.

ABSTRAK

Pirolisis pantas ialah satu teknologi penukaran habakimia untuk menukar biojisim kepada produk bio-minyak. Dalam kajian ini, habuk meranti merah (RMS) dipilih sebagai biojisim suapan untuk menilai potensinya menghasilkan bio-minyak melalui pirolisis pantas. Kajian ini merangkumi objektif untuk menyiasat kesan parameter proses dalam mengoptimumkan penghasilan bio-minyak dan mencirikan bio-minyak yang diekstrak. Pirolisis cepat dijalankan dalam skala-makmal reaktor lapisan terbendalir, bersama sistem yang terdiri dari pengawal suhu, siklon, pemeluwap, gas nitrogen, meter alir and pengumpul arang dan bio-minyak. Dalam mengkaji kesan keadaan pirolisis, eksperimen dijalankan mengikut pendekatan satu-faktor-pada-satu-masa (OFAT) dengan parameter yng terlibat ialah suhu, kadar alir N_2, masa tahanan dan saiz partikel suapan. Keputusan menunjukkan bio-oil mencapai hasil maksimum sekitar 56.3 % pada suhu 450 °C, kadar alir N_2 25 L/min dan masa tahanan 20 min. untuk saiz partikel suapan 0.3 mm. Dapat disimpulkan bahawa suhu adalah parameter yang paling berpengaruh untuk hasilan bio-minyak. Pencirian fizikokimia bio-minyak menunjukkan bio-minyak tidak sesuai untuk bahan bakar pengangkutan kerana tinggi kandungan oksigen. Melalui analisis spektrometri kromatografi gas (GC-MS), fenolik adalah sebatian dominan yang dikenalpasti dalam bio-minyak. Jumlah gula dalam bio-minyak ialah 12.82 % luas termasuk hasil levoglucosan adalah 8.97 % luas. Dalam menentukan kesan rawatan basuhan biojisim, RMS dibasuh dengan air dinyahion (DI) atau asid hidroklorik (HCl) cair. Kecekapan penyingkiran AAEM dengan air DI, 1.0M HCl dan 2.0 M HCl adalah 66.39%, 93.32% dan 97.28%, masing-masing. Dari analisis FTIR, rawatan basuhan telah menguatkan ikatan kimia RMS. Untuk pengeluaran bio-minyak, bio-minyak yang diekstrak dari RMS - air DI mencapai hasil maksimum kira-kira 57.2 % pada 450 °C suhu optimum. Dalam bio-minyak yang diekstrak, RMS yang dibasuh menghasilkan lebih banyak kompaun berat dan lebih banyak levoglucosan daripada RMS mentah. Dalam kajian pyrolysis RMS yang diresapi, RMS diresapkan dengan $CaCl_2$, $CaSO_4$, $FeCl_2$ atau $FeSO_4$. Antara suapan ini, RMS - $FeSO_4$ meningkatkan proses penguraian pada suhu yang lebih rendah dengan suhu penguraian maksimum telah beralih dari 361 °C bagi RMS kawalan kepada 314 °C bagi RMS - $FeSO_4$. Melalui FTIR, resapan RMS dengan $FeSO_4$ telah melemahkan ikatan kimia dalam RMS. Dalam bio-minyak yang diekstrak, ia mengandungi sebatian berat molekul berjulat besar dan menunjukkan peningkatan dalam hasil levoglucosan. Levoglucosan adalah tertinggi dalam RMS - $FeSO_4$ iaitu 40.23 % luas, dengan hasil gula sebanyak 42.24 % luas. Dalam mengoptimumkan hasil bio-minyak, rekabentuk komposit pusat (CCD) daripada kaedah pemodelan permukaan respon (RSM) digunakan untuk membangunkan model matematik dan mengoptimumkan parameter proses. Melalui model yang diramalkan, keputusan menunjukkan keadaan proses pirolysis optimum diperoleh pada suhu 480 °C, 25 L/min kadar aliran N_2 dan 24 min masa tahanan dengan 56.5 % hasil bio-minyak dan 2.11 % ralat oleh eksperimen. Kesimpulannya, RMS mempunyai potensi untuk menghasilkan bio-minyak. Dengan rawatan lanjut ke atas bio-oil untuk menyingkirkan kandungan oksigen, bio-minyak ini boleh digunakan untuk menggantikan bahan api konvensional. Rawatan terapi RMS dengan $FeSO_4$ mendedahkan bahawa proses penguraian dapat ditingkatkan pada suhu yang lebih rendah dan meningkatkan levoglucosan dalam bio-minyak. Penemuan-penemuan ini dijangka menyediakan beberapa garis panduan dalam kajian masa depan untuk menghasilkan produk tambah nilai dari sisa lignoselulosa yang lain dan seterusnya, konsep kerajaan untuk mengalihkan sisa kepada produk-kekayaan dapat dicapai.

ABSTRACT

Fast pyrolysis is a thermochemical conversion technology to convert biomass into bio-oil product. In this study, red meranti sawdust (RMS) was selected as biomass as feedstock to evaluate its potential to produce bio-oil by fast pyrolysis. The study covers the objective to investigate the parameters effect of process in optimising the bio-oil production and characterise the extracted bio-oil. Fast pyrolysis process was conducted in a bench-scale fluidized bed reactor, with the system consist of temperature controller, cyclone, condensers, nitrogen gas, flow meter, char and bio-oil collectors. In investigating the effect of pyrolysis condition, the experiments were run according to one-factor-at-a-time (OFAT) approach with the parameters involved were temperature, N_2 flow rate, retention time and feed particles size. Results showed that bio-oil achieved maximum yield about 56.3 % at 450 °C of temperature, 25 L/min of N_2 flow rate and 20 min of retention time for 0.3 mm of feed particles size. It can be concluded that the temperature was the most influential parameter for bio-oil yield. Physicochemical characterisation of bio-oil indicated bio-oil not suitable for transportation fuel due to high oxygen content. Through gas chromatography–mass spectrometry (GC-MS) analysis, phenolic was the dominant compound identified in bio-oil. Total sugars in bio-oil was 12.82 % area including levoglucosan yield was 8.97 % area. In determining the effect of washing treatment, RMS was washed with deionised (DI) water or diluted hydrochloric (HCl) acid. The efficiency of AAEM removal by DI water, 1.0M HCl and 2.0 M HCl were 66.39 %, 93.32 %, and 97.28 %, respectively. From FTIR analysis, washing treatment had strengthened the RMS chemical bonds. For bio-oil production, bio-oil extracted from RMS - DI water achieved maximum yield about 57.2 % at 450 °C of optimum temperature. In extracted bio-oil, washed RMS produced higher heavier compound and higher levoglucosan than raw RMS. In pyrolysis of impregnated RMS study, RMS was impregnated with $CaCl_2$, $CaSO_4$, $FeCl_2$ or $FeSO_4$. Among these feedstocks, RMS - $FeSO_4$ enhanced the degradation process at lower temperatures with the maximum degradation of temperature has been shifted from 361 °C for RMS control to 314 °C for RMS - $FeSO_4$. Through FTIR analysis, impregnated RMS with $FeSO_4$ has weakened the RMS chemical bond. In extracted bio-oil, it consisted large range of molecular weight compounds and showed an increasing in levoglucosan yield. Levoglucosan was the highest in RMS - $FeSO_4$ about 40.23 % area, with 42.24 % area of total anhydrosugars yield. In optimising bio-oil yield, central composite design (CCD) of response surface methodology (RSM) modelling was employed to develop mathematical model and optimise the process parameters. Through predicted model, results showed that the optimal pyrolysis process condition was obtained at 480 °C of temperature, 25 L/min of N_2 flow rate and 24 min of retention time with 56.5 % of bio-oil yield and 2.11 % of error by experiment. Conclusion, RMS has a potential to produce bio-oil. With further treatment on bio-oil to remove oxygen content, this bio-oil can be applied to substitute conventional fuel. Impregnated treatment of RMS with $FeSO_4$ reveals degradation process can be enhanced at lower temperature and increases levoglucosan in bio-oil. These findings are expected to provide some guidelines in future study to produce value-added product from other lignocellulose waste and further, the government concept of divert waste to wealth-product can be achieved.

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°C	degree Celsius
°C/s	degree Celsius per Second
cSt	centistokes
g/mol	gram/mol
kJ/kg	Kilojoule/kilogram
L/min	Litre per minute
m^3	cubic meter
min	minute
mL	mililitre
mm	millimetre
MJ/kg	Megajoule/kilogram
Mtoe	Million tons of oil equivalent
rpm	Rotation per Minute
wt. %	percentage by weight
W/mK	Watts per meter Kelvin
µm	micrometre
Y_b	bio-oil yield

LIST OF ABBREVIATIONS

AAEM	Alkali and Alkaline Earth Metal
ASEAN	Association of Southeast Asian Nations
ASTM	American Society for Testing and Materials
BC	Bio-oil Collector
C	Carbon
Ca	Calcium
$CaCl_2$	Calcium Chloride
$CaSO_4$	Calcium Sulphate
CC	Char Collector
CCD	Central Composite Design
CO	Carbon Monoxide
CO_2	Carbon Dioxide
DI	Deionised
DTG	Differential Thermogravimetric
ENSYN	Renewable Fuels and Chemical from Non-Food Biomass
$FeCl_2$	Iron (II) Chloride
$FeSO_4$	Iron (II) Sulphate
FTeK	Fakulti Teknologi Kejuruteraan
FTIR	Fourier Transform Infrared Spectroscopy
GC-MS	Gas Chromatography – Mass Spectrometry
H^+	Hydrogen Ion
H_2	Hydrogen Gas
H_2O	Water
HAA	Hydroxyacetaldehyde
HCl	Hydrochloride Acid
HHV	Higher Heating Value
ICP-OES	Inductively Coupled Plasma – Optical Emission Spectroscopy
K	Potassium
LHV	Lower heating value
Mg	Magnesium
N_2	Nitrogen Gas
Na	Sodium
NCG	Non-Condensable Gases
NO_x	Nitric Oxide and Nitrogen Dioxide

NREL	National Renewable Energy Laboratory
O	Oxygen
OFAT	One Factor at a Time
PAH	Polycyclic Aromatic Hydrocarbon
pH	Potential of Hydrogen
RMS	Red Meranti Sawdust
RSM	Response Surface Methodology
TGA	Thermogravimetric Analysis
UMP	Universiti Malaysia Pahang
Zn	Zink

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