



UNIVERSITI PUTRA MALAYSIA

***PRODUCTION OF BIO-OIL VIA HYDROTHERMAL LIQUEFACTION OF
EMPTY FRUIT BUNCH USING ZEOLITE AND MCM-41 BASED
CATALYSTS***

NURUL SUZIANA BINTI NAWI @ MOHAMED

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By

NURUL SUZIANA BINTI NAWI @ MOHAMED

**Thesis Submitted to the School of Graduate Studies, Universiti Putra
Malaysia, in Fulfilment of the Requirements for the Degree of Doctor of
Philosophy**

June 2020

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Abstract of thesis presented to the Senate of Universiti Putra Malaysia in
Fulfillment of the requirement for the degree of Doctor of Philosophy

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June 2020

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Faculty : Science

Biomass is one of the renewable energy that has been revealing its potential as a substituent for natural fossil fuel recently. Nowadays, converting biomass via hydrothermal liquefaction appears to be another alternative to obtain valuable high energy products and chemical. In this study, oil palm empty fruit bunch (EFB) has been chosen as the source of biomass feedstock for conversion of biomass to bio-oil using HZSM-5 (zeolite) and MCM-41 (Mobil Composition of Matter number 41) as a based catalysts. The different composition of metal promoter barium (Ba), nickel (Ni), lanthanum (La) and cerium (Ce) either mono or bimetal were impregnated into zeolite (denoted as CHZSM5) and further investigated. High acidity properties of Ba and Ni modified CHZSM5 found to be the most effective in catalysing the hydrothermal liquefaction of EFB. Besides, the high surface area and volume ratio of combined two metal promoter (Ba1La2, Ba2Ni1 and Ba2La1/CHZSM5) catalysts were the reasons in the increment in the acidic sites and eventually enhanced the catalytic activity in hydrothermal liquefaction of EFB. Other than that, hierarchical zeolite has been produced with the presence of cetyltrimethylammonium bromide (CTAB) as a surfactant and *n*-alkane (*n*-hexane, *n*-octane, *n*-decane and *n*-dodecane) as swelling agent to tailor the formation of hierarchical zeolite with large pore. 1.5 molar ratios of *n*-alkane significantly reduced the size of nanocrystals formed compared to 1.1 *n*-alkanes, implying high acidity strength of hierarchical zeolite positively influenced by the size of nanocrystal and impactful in catalyst's performance. This study also revealed the ratio of silica to aluminum, Si/Al = 0.03 gave a great influence to the surface area, pore volume and pore diameter of the synthesized MCM-41 (denoted as CMCM Si/Al 0.03) and proportionally increase the acidity of the final catalyst. The addition of active metal Ni into CMCM Si/Al 0.03 rendered the greater dispersion of the crystals and at the same time induces the acidity of the catalyst. As mentioned above, high acidity

and high textural properties of the synthesised catalysts improved the quality of the obtained bio-oil in the catalytic hydrothermal liquefaction of EFB with less oxygenated compounds.



Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Doktor Falsafah

**PENGHASILAN BIO-MINYAK MELALUI PENCECAIRAN HIDROTERMA
TANDAN BUAH KOSONG MENGGUNAKAN ASAS MANGKIN ZEOLIT DAN
MCM-41**

Oleh

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Biojisim adalah salah satu tenaga boleh diperbaharui yang telah didedahkan potensinya sebagai gantian bahan api fosil semulajadi baru-baru ini. Pada masa kini, penukaran biojisim melalui pencecairan hidroterma telah menjadi salah satu alternatif untuk memperolehi produk tenaga tinggi dan bahan kimia yang berharga. Dalam kajian ini, tandan buah kosong kelapa sawit (EFB) telah dipilih sebagai sumber biojisim bahan mentah untuk penukaran biojisim kepada bio-minyak menggunakan asas mangkin HZSM-5 (zeolit) dan MCM-41 (komposisi mobil bagi jirim nombor 41). Perbezaan komposisi logam pendorong barium (Ba), nickel (Ni), lanthanum (La) dan cerium (Ce) samada secara tunggal atau dwi-logam telah di isitepuan kedalam zeolit (dilambangkan sebagai CHZSM5) dan disiasat selanjutnya. Ciri-ciri keasidan yang tinggi pada Ba dan Ni mengubahsuai CHZSM5 telah dilihat sebagai paling efektif dalam memangkin pencecairan hidroterma EFB. Di samping itu, kawasan permukaan yang tinggi dan nisbah isipadu gabungan dua logam pendorong mangkin (Ba_1La_2 , Ba_2Ni_1 and $Ba_2La_1/CHZSM5$) adalah penyebab kepada peningkatan tapak asid dan seterusnya menambah aktiviti pemangkinan dalam pencecairan hidroterma EFB. Selain daripada itu, zeolit hierarki telah dihasilkan dengan kehadiran cetyltrimethylammonium bromide (CTAB) sebagai surfaktan dan *n*-alkana (*n*-heksana, *n*-oktana, *n*-dekana dan *n*-dodekana) sebagai agen pengembang untuk membina pembentukan zeolit hierarki berliang besar. 1.5 nisbah molar *n*-alkana secara ketara merendahkan saiz nanokristal yang dibentuk berbanding dengan 1.1 *n*-alkana, menandakan kekuatan keasidan yang tinggi pada zeolit hierarki secara positifnya dipengaruhi oleh saiz nanokristal dan memberi impak tinggi kepada prestasi mangkin. Kajian ini juga mendedahkan nisbah silica kepada aluminum, Si/Al = 0.03 memberi kesan yang besar kepada luas kawasan permukaan, isipadu liang dan diameter liang MCM-41 yang disintesis (dilambangkan sebagai CMCM Si/Al 0.03) dan meningkatkan keasidan mangkin akhir secara berkadar.

Penambahan logam aktif Ni ke dalam CMCM Si/Al 0.03 memberikan penyelerakan kristal yang terbaik dan pada masa yang sama mencetuskan keasidan mangkin. Seperti yang dinyatakan di atas, keasidan yang tinggi dan sifat tekstur yang tinggi pada mangkin yang disintesis telah meningkatkan kualiti bio-minyak yang diperolehi dalam pencecairan hidroterma EFB dengan pengurangan sebatian beroksigen.



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LIST OF ABBREVIATIONS

BET	Brunauer – Emmer – Teller
CHZSM5	Commercial zeolite ZSM-5
CMCM	Calcined MCM-41
CTAB	Cetyltrimethylammonium bromide
EFB	Empty Fruit bunch
FESEM	Field Emission Scanning Electron Microscopy
GCMS	Gas Chromatography Mass Spectrometer
ICP-OES	Induced Coupled Plasma – Optical Emission Spectroscopy
JCPDS	Joint Committee of Powder Diffraction Standards
MFI	Modernite Framework Inverted
nC_6	<i>n</i> -hexane
nC_8	<i>n</i> -octane
nC_{10}	<i>n</i> -decane
nC_{12}	<i>n</i> -dodecane
SEM	Scanning Electron Microscopy
Si/Al	Silica/Alumina
TPD-NH ₃	Temperature Programmed Desorption of Ammonia
TPR	Temperature Programmed Reduction
XRD	X-Ray Diffraction
Mt	Million tonnes

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