PREPARATION AND CHARACTERISATION OF CALCIUM OXIDE FROM GYPSUM BASED COMPOUND FOR TRANSESTERIFICATION OF WASTE COOKING OIL

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MASTER OF SCIENCES

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 (Industrial Chemistry)

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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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for the award of the degree of
Master of Science

Faculty of Industrial Sciences & Technology

UNIVERSITI MALAYSIA PAHANG

JULY 2018

ACKNOWLEDGEMENTS

Thanks to God Almighty who give me His bless and strength to undertake and complete my research. I extend my deepest gratitude and thanks to my supervisor plus advisor, Associate Professor Dr. Gaanty Pragas Maniam. He assisted in a great deal in this endeavour by providing motivations, advices, guidance, support, ideas and knowledge whenever I ran into a trouble spot or had a question about my research or writing and steered me in the right the direction whenever he thought I needed it.

I also would like to extend my gratefulness and thanks to my co-supervisor Associate Professor Dr. Mohd Hasbi Ab. Rahim, who provided me with his significant knowledge and material resources for my work. In addition, I'm also grateful to Professor Dr. Jose Rajan for his very helpful guidance and Professor Dato' Dr. Mashitah Binti Mohd. Yusoff as her mere presence always motivates me.

The credit of completing my work also goes for the Faculty of Industrial Sciences & Technology office staffs laboratory staffs and Science Officers, Central Laboratory Science Officers and Institute of Postgraduate Studies staffs. I also would like to acknowledge University Malaysia Pahang (UMP) and Ministry of Higher Education (MOHE) of Malaysia for RDU Grants RDU 121207 and RDU 150322 for financial support and made this study possible.

I must express my very profound gratitude to my family for providing me with support and encouragement throughout my years of study and through the process of researching and writing this thesis between many hardships and problems. This accomplishment would not have been possible without them. Special thanks are in place to my teammates Intan Shafinaz Binti Abd Manaf, Dr. Noor Hindryawati, Irma Nurfitri, Nurul Hajar Binti Embong, Rehan Binti Zainol Abidin, Chong Kah Lun. Thank you.

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LIST OF SYMBOLS

α	Roman alphabet alpha
β	Roman alphabet beta
<	Less than
>	Greater than
≥	Greater than and equals to; atleast
°	degrees of angular arc
°C	Material temperature in degree Celsius
µm	Material length, width or height in micrometer
A_{IS}	internal standard (methyl heptadecanoate) peak area
C_{IS}	concentration of the internal standard solution, in mg/mL
V_{IS}	volume of the internal standard solution used, mL

LIST OF ABBREVIATIONS

CEU	Council of the European Union
DOE	Department of Environment Malaysia
FAME	Fatty acid methyl esters
FESEM-EDX	Field emission scanning electron microscopy-electron dispersive X-ray
FFA	Free fatty acids
FGD	Flue gas desulphuriser
FGG	Flue-gas gypsum
GC-FID	Gas chromatography-flame ionisation detector
ICP-MS	Inductively coupled plasma mass spectrometry
MeOH	Methanol
OM	Organic matter
SEM	Scanning electron microscopy
SW	Scheduled Waste
TGA	Thermogravimetric analysis
WCO	Waste cooking oil
XRD	X-ray diffraction
XRF	X-ray fluorescence
ZWS	Zero Waste Scotland
°C	Material temperature in degree Celsius
g	Material mass in gram
g/cm^3	Material density in gram per centimeter cube
g/L	Solution concentration in gram per litre
g/mol	Material molar mass in gram per mol
h	Time in hour
M	Solution concentration molar concentration
m	Material length, width or height in meter
mg/mL	Solution concentration in milligram per millilitre
mL	Solution or liquid volume in millilitre
mm	Material length, width or height in millimeter
MT	Material mass in metric tons
ppb	relative abundance of dissolved minerals in parts-per-billion

ppm	relative abundance of dissolved minerals in parts-per-million
wt. %	Weight percentage
ΣA	total peak area

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ABSTRAK

Dalam kerja ini, gipsum (kalsium sulfat dihidrat), dari syarikat bekalan kimia (gipsum gred makmal) dan sisa industri (sisa gipsum) telah digunakan untuk menghasilkan kalsium oksida (CaO). Gipsum buangan (gipsum merah) diperoleh daripada Huntsmann Tioxide (M) Sdn. Bhd. Terengganu, Malaysia. Minyak masak sisa digunakan untuk menghasilkan FAME dengan metanol melalui transesterifikasi menggunakan CaO dari sisa gipsum sebagai pemangkin heterogen. Dua kaedah yang berbeza digunakan untuk menukar gipsum kepada CaO. Dalam kaedah pertama, kaedah tindak balas dua langkah, gipsum gred makmal dan sisa gipsum digunakan secara berasingan, di mana ia dirawat secara kimia untuk menghasilkan kalsium hidroksida. Kemudian, kalsium hidroksida dikalsinasi dan diuraikan kepada CaO pada suhu yang jauh lebih rendah daripada kaedah terdahulu. Untuk kaedah kedua, kaedah penurunan karbotermal diubahsuai telah digunakan. Dua persekitaran gas statik yang berbeza telah digunakan (untuk mengkaji kesan gas yang berlainan) dan bukan aliran gas berterusan (untuk mengkaji kesan persekitaran statik) dalam kaedah yang diubah suai ini. Zink oksida pemangkin untuk proses penurunan karbotermal digunakan untuk meningkatkan CaO. Sampel penyediaan CaO telah dihantar untuk analisis XRD, TGA dan FESEM-EDX. Transesterifikasi minyak masak sisa dan metanol (MeOH) menggunakan CaO dari sisa gipsum (disediakan melalui kaedah dua langkah) sebagai pemangkin heterogen dilakukan pada suhu tetap 60°C dan menetapkan 12:1 MeOH:minyak nisbah molar; untuk menghasilkan FAME. Kesan oleh variasi jumlah pemangkin CaO (3 wt.% Dan 5 wt.%) Dan tempoh tindak balas (5h dan 7h) pada kandungan ester telah dikaji. Kandungan ester dikira mengikut EN14214 dari analisis GC-FID. Kualiti FAME yang dihasilkan dan dibersihkan dianalisis menggunakan penganalisis ICP-MS dan CHNOS. Ujian enjin dijalankan dengan menggunakan campuran B10 biodiesel yang dihasilkan dari FAME yang dihasilkan. “Diffractogram” Difraksi X-Ray dari sampel penyediaan CaO dianalisis dengan menggunakan “*Match!*” perisian dari Crystal Impact dengan “Crystallography Open Database”. Dari difraksi kaedah dua langkah tindak balas yang dilakukan pada gipsum gred makmal, ditunjukkan bahawa ketiadaan lengkap mana-mana puncak lain kecuali puncak CaO jelas untuk hampir 100% penukaran gipsum gred makmal kepada CaO. Dari diffractograms kaedah tindak balas dua langkah yang dilakukan pada gips sisa, ia dikira bahawa kandungan CaO adalah pada 86.45%. Kaedah penukaran dua langkah ini selamat membebaskan kumpulan sulfat sebagai natrium sulfat, dari gipsum yang menghasilkan gas sulfur dioksida, apabila gipsum ditukar menjadi kalsium oksida melalui kaedah pengurangan karbida. Reaksi karbothermal yang diubahsuai juga menjadi berfaedah. Gas sulfur oksida yang dihasilkan dalam laluan ini bertindak balas dengan karbon atau karbon monoksida dan dikurangkan menjadi unsur sulfur. Kandungan CaO tertinggi, 31.80% diperolehi daripada persekitaran nitrogen statik; karbon diaktifkan kepada gipsum nisbah 20:1, dengan 2 wt.% pemangkin zink oksida. Dengan menggunakan CaO dari sisa gipsum sebagai pemangkin, ia telah dikenal pasti bahawa transesterifikasi minyak masak sisa dengan methanol memberikan kandungan ester tertinggi sebanyak 97.72% untuk 3 wt% dan 5h time. Hasil ICP-MS mendedahkan bahawa sonication dan penapisan menggunakan karbon aktif diperlukan dua kali untuk mengurangkan larutan kation (terutama Ca^{2+}) untuk memenuhi kandungan “Logam kumpulan II” EN 14214 dan kandungan sulfur. Pengujian enjin menggunakan campuran biodiesel B10 yang dibuat menggunakan FAME yang dihasilkan menunjukkan bahawa karbon monoksida terancam dikurangkan sebanyak 0.03% kepada 0.05% berbanding dengan petro-diesel.

ABSTRACT

In the present work, gypsum (calcium sulphate dihydrate), from chemical supply company (laboratory grade gypsum) and industrial waste (waste gypsum) was utilised to produce calcium oxide (CaO). The waste gypsum (red gypsum) was obtained from Huntsmann Tioxide (M) Sdn. Bhd. Terengganu, Malaysia. Waste cooking oil was used to produce FAME with methanol via transesterification utilising CaO from waste gypsum as heterogeneous catalyst. Two different methods were employed to convert gypsum to CaO. In the first method, two-step reaction method, laboratory grade gypsum and waste gypsum were utilised separately, where it was chemically treated to produce calcium hydroxide. Later, the calcium hydroxide was calcined and decomposed to CaO at much lower temperatures than prior methods. For the second method, a modified carbothermal reduction method was utilised. Two different static gaseous environments were used (to study the effect of different gas) instead of continuous gas flow (to study the effect of static environment) in this modified method. The catalyst zinc oxide for carbothermal reduction process was used to increase CaO. The samples of CaO preparation were sent for XRD, TGA and FESEM-EDX analysis. Transesterification of waste cooking oil and methanol (MeOH) using CaO from waste gypsum (prepared via two-step method) as a heterogeneous catalyst was done at a fixed temperature of 60°C and fixed 12:1 MeOH:oil molar ratio; to produce FAME. Effect on ester content of CaO catalyst amount variation (3 wt. % and 5 wt. %) and reaction duration (5h and 7h) was studied. The ester content was calculated according EN14214 from GC-FID analysis. The quality of FAME produced and purified was analysed using ICP-MS and CHNOS analyser. Engine testing was conducted using B10 biodiesel blend made from FAME produced. The X-Ray Diffraction diffractograms from CaO preparation samples were analysed using *Match!* software from Crystal Impact with Crystallography Open Database. From the diffractograms of two-step reaction method done on laboratory grade gypsum, it is shown that the complete absence of any other peaks except CaO peaks is evident for almost 100% conversion of laboratory grade gypsum to CaO. From the diffractograms of two-step reaction method done on waste gypsum, it was calculated that the CaO content was at 86.45%. This two-step conversion method has safely liberated the sulphate group as sodium sulphate, from gypsum, which gives rise to sulphur dioxide gas, when gypsum is converted to calcium oxide through carbothermal reduction method. The modified carbothermal reaction proved to be advantageous as well. The sulphur oxide gas produced in this route was further reacted with carbon or carbon monoxide and reduced to elemental sulphur. The highest CaO content, 31.80% was obtained from the static nitrogen environment; activated carbon to gypsum ratio of 20:1, with 2 wt. % zinc oxide catalyst. By using CaO from waste gypsum as a catalyst it was identified that transesterification of waste cooking oil with methanol gave highest ester content of 97.72% for 3 wt. % and 5h variation. The ICP-MS results revealed that sonication and filtration using activated carbon are needed twice to reduce the cation leachate (especially Ca^{2+}) in order to fit EN 14214's "Group II metals" content and sulphur content. The engine testing using B10 blend biodiesel blend made using produced FAME showed that threatening carbon monoxide reduced by 0.03% volume to 0.05% volume compared to petro-diesel.

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