UNIVERSITI PUTRA MALAYSIA

SYNTHESIS AND CHARACTERISATION OF CaO DERIVED FROM COCKLESHELL AS A SUPPORT FOR MIXED-MgO AND Fe2O3 CATALYSTS FOR FAME PRODUCTION USING WASTE COOKING OIL

EZZAH MAHMUDAH BINTI SALIM

FS 2018 62

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By

EZZAH MAHMUDAH BINTI SALIM

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfillment of the Requirements for the Degree of Master of Science

August 2018

Special Dedication to:
My Lovely Husband:
Mohamad Zaihan bin Zailan
&
My Beloved Daughter:
Eiz Khaira Hadani binti Mohamad Zaihan
and My Family

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the Master of Science

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EZZAH MAHMUDAH BINTI SALIM

July 2018

Chairman : Prof. Taufiq Yap Yun Hin, PhD

Faculty : Science

As an alternative for fossil diesel fuels, biodiesel has gained interest of most researchers recently in promoting environmentally sustainable fuels. With the presence of metal oxide modified cockleshell catalysts, biodiesel can be easily produced via transesterification of triglyceride with alcohol under reaction conditions. In this study, the magnesium oxide (MgO) and iron (III) oxide (Fe_2O_3) doped on natural CaO catalyst, which derived from cockleshell. MgO/CaO and Fe_2O_3/CaO were prepared and utilized by a single-step reaction process. The CaO were crushed and milled to obtain fine powder and calcined at 900 ºC for 6 h. Then, MgO/CaO and Fe_2O_3/CaO catalysts were synthesized using wet impregnation method; followed by calcination at 500 ºC for 4 h to produce heterogeneous catalyst with high activity and better selectivity which relatively giving a better performance in transesterification reaction. The catalysts were characterized in detail by both qualitative and quantitative methods such as X-ray fluorescence (XRF), X-ray diffraction (XRD), scanning electron microscope (SEM), scanning electron microscope-energy dispersive X-ray (SEM-EDX), thermal gravimetric analysis (TGA), temperature programmed desorption of ammonia and carbon dioxide (TPD-NH_3 and TPD-CO_2), and Brunauer-Emmett-Teller (BET) analyses, while the synthesized biodiesel was characterized using gas chromatography-flame ionization detector (GC-FID). The operating parameters such as methanol-to-oil molar ratio, catalyst amount and reaction time were investigated in order to optimize for the reaction condition for the biodiesel production. As a result, the optimum reaction parameters for MgO/CaO were 10:1 methanol-to-oil molar ratio, 4 h of reaction time, 2 wt. % of the catalyst loading and reaction temperature of 65 ºC shows 74 % FAME yield, meanwhile the optimum reaction parameters for Fe_2O_3/CaO were found to be 15:1 methanol-to-oil molar ratio, 3 h of reaction time and 1 wt. % of catalyst loading with reaction temperature of 65 ºC which produced 92 % FAME yield. The results revealed suggestively high potential of the heterogeneous MgO/CaO catalyst can be reusable at least 3 reaction cycles only while Fe_2O_3/CaO catalyst for direct conversion of waste cooking oil to biodiesel with the possibility to be reusable at least 5 reaction cycles without any reactivation process. Several physicochemical properties of waste cooking-based biodiesel produced was tested and agreed to ASTM D4052, ASTM D445, ASTM D464, ASTM D974 and EN 14214 standard.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Master Sains

SINTESIS DAN PENCIRIAN CaO TERBITAN DARIPADA CENGKERANG KERANG SEBAGAI PENYOKONG UNTUK PEMANGKIN CAMPURAN-MgO DAN Fe_2O_3 UNTUK PENGELUARAN FAME MENGGUNAKAN SISA MINYAK MASAK

Oleh

EZZAH MAHMUDAH BINTI SALIM

Julai 2018

Pengerusi : Prof Taufiq Yap Yun Hin

Fakulti : Sains

Sebagai alternatif untuk bahan api diesel fosil, biodiesel telah mendapat minat kebanyakan penyelidik baru-baru ini dalam mempromosikan bahan bakar lestari alam sekitar. Dengan kehadiran oksida logam yang diubahsuai pemangkin cengkerang kerang, biodiesel dapat dihasilkan dengan mudah melalui transesterifikasi trigliserida dengan alkohol dalam keadaan reaksi. Dalam kajian ini, magnesium oksida (MgO) dan besi (III) oksida (Fe_2O_3) dihidupkan ke dalam pemangkin CaO semulajadi, yang diperolehi daripada cengkerang kerang. MgO/CaO dan Fe_2O_3/CaO disediakan dan digunakan oleh proses reaksi satu langkah. CaO telah ditumbuk dan dihancurkan untuk mendapatkan serbuk halus dan dikalsin pada 900 ^oC selama 6 jam. Kemudian, pemangkin MgO/CaO dan Fe_2O_3/CaO telah disintesis menggunakan kaedah pengisitepuan basah; diikuti dengan pengkalsinan pada 500 °C selama 4 jam untuk menghasilkan pemangkin heterogen dengan aktiviti yang tinggi dan selektiviti yang lebih baik yang secara relatifnya memberikan prestasi yang lebih baik dalam reaksi transesterifikasi. Pemangkin telah dicirikan dengan terperinci oleh kedua-dua kaedah kualitatif dan kuantitatif seperti pendarfluor sinaran-X (XRF), pembelauan sinaran-X (XRD), mikroskop imbasan elektron (SEM), mikroskop imbasan electron-tenaga penyerakan sinaran-X (SEM-EDX), analisis terma gravimetri (TGA), aturcara suhu nyahjerapan ammonia dan karbon dioksida (TPD-NH_3 dan TPD-CO_2), dan analisis kaedah Brunauer-Emmett-Teller (BET), manakala biodiesel yang disintesis dicirikan menggunakan gas kromatografi-pengesan ionisasi api (GC-FID). Parameter operasi seperti nisbah molar metanol-kepada-minyak, jumlah pemangkin dan masa reaksi diselidiki untuk mengoptimumkan keadaan reaksi untuk pengeluaran biodiesel. Hasilnya, parameter tindak balas optimum untuk MgO/CaO ialah nisbah molar 10:1 metanol-kepada-minyak, 4 jam masa reaksi, 2 wt. % berat pemangkin dan suhu tindak balas 65 °C menunjukkan 74 % hasil FAME, sementara parameter tindak balas optimum untuk Fe_2O_3/CaO didapati nisbah molar 15:1 metanol-kepada-minyak, 3 jam masa reaksi dan 1 wt. % berat pemangkin dengan suhu tindak balas 65 °C, dimana menghasilkan 92 % hasil FAME. Hasilnya menunjukkan potensi tinggi pemangkin MgO/CaO yang berkemungkinan tinggi boleh digunakan sekurang-kurangnya 3 kitaran tindak balas sahaja sementara pemangkin Fe_2O_3/CaO untuk penukaran langsung sisa minyak masak kepada biodiesel dengan kemungkinan

untuk menggunakan semula sekurang-kurangnya 5 kitaran reaksi tanpa sebarang pengaktifan semula proses. Beberapa sifat fizikokimia biodiesel berasaskan-masakan sisa yang dihasilkan telah diuji dan dipersetujui untuk standard ASTM D4052, ASTM D445, ASTM D464, ASTM D974 dan EN 14214.

ACKNOWLEDGEMNTS

"In the name of Allah, the Most Gracious and the Most Merciful"

I wish to express my outmost gratitude to Allah SWT for the strength and patience He bestowed on me to complete this study.

I would like to express my greatest gratitude to my main supervisor, Professor Dr. Taufiq Yap Yun Hin, for his guidance, suggestion, patience, support and encouragement throughout my study in Universiti Putra Malaysia. His valuable advices and humanitarian values have giving significant impact towards my morale and skills in conducting my research and my life. I am also grateful to my co-supervisor, Dr. Izham Mohd Saiman for his guidance and help during the study.

I am also thankful and grateful to my mother and father for being a supportive parent who has always been there for me during my study. Deepest gratitude also extended to my beloved husband, Mohamad Zaihan Zailan, thank you very much for being very supportive, encouraging and spending this wonderful journey in our life. Thanks also for your non-stop advices and Doa' for my success and not to be forgotten both my family members.

In addition, I express my special thanks to all the SusCAT members for their support, expertise and valuable friendship during the period. I sincerely thanks to all staffs in Chemistry Department, Faculty of Science, Institute of Advance Technology (ITMA) and Institute of Bioscience, UPM. Special gratitude also goes to Mohd Sufri Mastuli and Dr. Mohd Lokman Ibrahim, lecturers from Faculty of Applied Science, Universiti Teknologi Mara (UiTM), for their big help, guidance and assistance during my catalysis research study.

Last but not least, I am also grateful and appreciate the financial support of the Graduate Research Fund (UPM) and Ministry of Higher Education (MOHE) Malaysia via the scholarships of MyMaster.

~Alhamdulillah~

I certify that a Thesis Examination Committee has met on 31 July 2018 to conduct the final examination of Ezzah Mahmudah binti Salim on her thesis entitled "Synthesis and Characterisation of CaO Derived from Cockleshell as Support for Mixed-MgO and Fe_2O_3 Catalysts for Fame Production Using Waste Cooking Oil " in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Master of Science.

Members of the Thesis Examination Committee were as follows:

Janet Lim Hong Ngee, PhD
Associate Professor
Faculty of Science
Universiti Putra Malaysia
(Chairman)

Tan Kar Ban, PhD
Associate Professor
Faculty of Science
Universiti Putra Malaysia
(Internal Examiner)

Aishah Abdul Jalil, PhD
Associate Professor
Universiti Teknologi Malaysia
Malaysia
(External Examiner)

RUSLI HAJI ABDULLAH, PhD
Professor and Deputy Dean
School of Graduate Studies
Universiti Putra Malaysia

Date: 27 September 2018

This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfillment of the requirement for the degree of Master of Science. The members of the Supervisory Committee were as follows:

Taufiq Yap Yun Hin, PhD
Professor
Faculty of Science
Universiti Putra Malaysia
(Chairman)

Mohd Izham Saiman, PhD
Senior Lecturer
Faculty of Science
Universiti Putra Malaysia
(Member)

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ROBIAH YUNUS, PhD
Professor and Dean
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Signature : ______________________
Name of Chairman of
Supervisory Committee : Taufiq Yap Yun Hin, PhD

Signature : ______________________
Name of Member of
Supervisory Committee : Mohd Izham Saiman, PhD

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LIST OF ABRREVIATIONS

AAS	Atomic Absorption Spectroscopy
ASTM	American Society for Testing and Materials
BET	Brunauer-Emmett-Teller
$CaCO_3$	Calcium carbonate
CaO	Calcium oxide
CO_2	Carbon dioxide
CS	Cockleshell
CS700	CaO derived from cockleshell calcined at 700 °C
CS800	CaO derived from cockleshell calcined at 800 °C
CS900	CaO derived from cockleshell calcined at 900 °C
DGs	Diglycerides
EDX	Energy Dispersive X-ray
EN	European Standard
FAME	Fatty acid methyl ester
Fe_2O_3	Iron (II) oxide
FFA	Free fatty acid
GC-FID	Gas Chromatography-Flame Ionization Detector
JCPDS	Joint Committee on Powder Diffraction Standards
MgO	Magnesium oxide
SEM	Scanning Electron Microscopy
TGA	Thermal Gravimetry Analysis
TGs	Triglycerides
TPD-CO_2	Temperature Programmed Desorption-Carbon Dioxide
TPD-NH_3	Temperature Programmed Desorption-Ammonia
UCS	Uncalcined Cockleshell

WCO	Waste Cooking Oil
XRD	X-Ray Diffraction
XRF	X-Ray Fluorescence

CHAPTER 1

INTRODUCTION

1.1 Fossil Fuel Energy

The world energy supply had relied to non-renewable fossil crude oil for more than two centuries. The world current limitations of fossil crude oil due to the depletion of fossil fuel (Foster et al., 2017). The exhaustion of the fossil fuel source has reduced enormous problem of energy dependency in most countries. (Alba-Rubio et al., 2010). The world's oil assets are located in certain location. The specific areas which have suitable geological features and allowed the creation along with accumulation of the oil. Figure 1.1 and 1.2 show the current demograhic of the oil production around the world. It is found that majority about more than 10 million barrels per day produced by US, Europe and Euroasia (Murray and King, 2012).

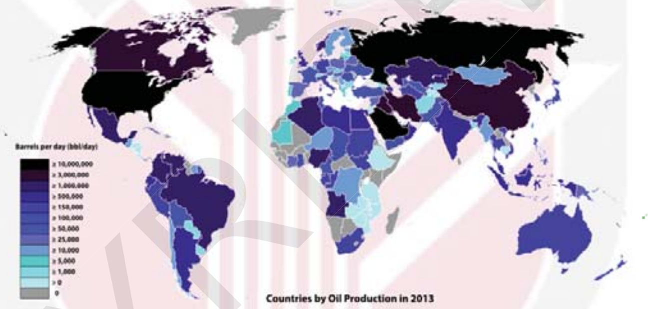

Figure 1.1: Distribution Of The Oil Produces In The World (Murray And King, 2012)

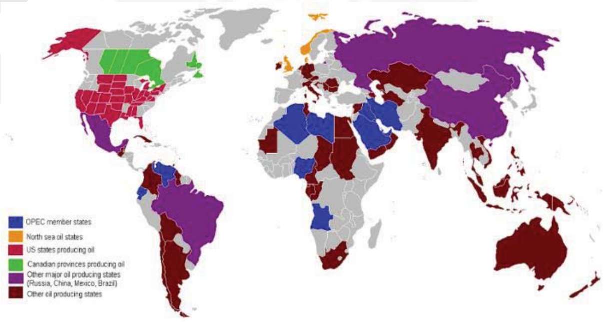

Figure 1.2: Oil Production Among The Countries (Murray and King, 2012)

Furthermore, Figure 1.3 shows projection of the energy consumption for near future. The approaching reduction of fossil fuel production along with the rapid growth of population and urbanization contributed to the increasing demand for the petroleum derived fuels for daily necessities mainly in transportation sector. Rahman and Mashud (2010) stated that the growing consumption of energy resulted in the country's increasing dependent on fossil fuels such as coal, oil and gas in 2010. However, energy sources are limited and lessening gradually. Therefore, limitability and unrenewability energy for fossil resources are the main purpose to the rising of cost of petroleum-derived fuels. Henceforth, new energy sources which can meet the demands should be explored.

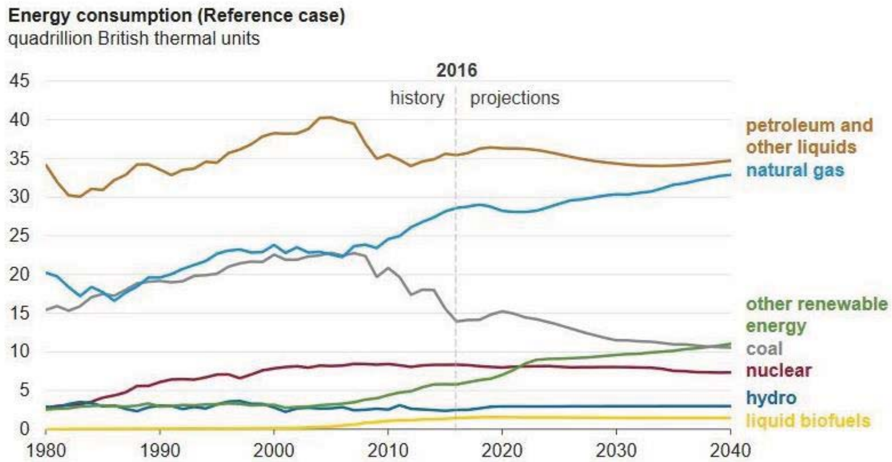

Figure 1.3: Projection Of The Energy Consumption From 1980-2040 (EIA, 2017)

The application of the petroleum-derived fuels is one of the reasons to the increasing emission of polluted gasses which lead to the global warming effect such as carbon monoxides, CO_x and nitrogen oxides, NO_x. It is recognized that global warming will lead to a serious climatic change and threatening human nature. Global warming is mainly produced by greenhouse gases, particularly CO_2 that are formed during burning of fossil fuels which results in significant alterations in the ecosystem. The atmospheric concentration of CO_2 has reached its utmost level over the past century as shown in Figure 1.4.

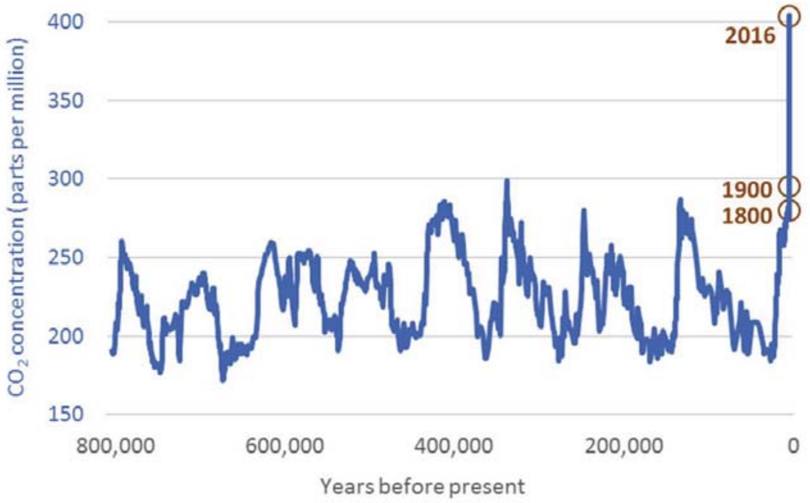

Figure 1.4: The Variation Of The Concentration Level Of CO_2 In The Atmosphere (Webb et al., 1993)

Kouzu and Hidaka reported that the constant ascend of the earth's average temperature, threatens millions of people with growing risk of hunger, floods, water shortage and diseases such as malaria. The diminishing resources of fossil fuels energy and the accumulation of carbon dioxide and other greenhouse gases in the atmosphere are the core explanation for the change in climates, and other living organisms. (Escobar et al., 2009). Consequently, to solve this problems, it is necessary by making great effort in order to reduce the climate change and also reduce the level of greenhouse gas emission. In this day and age, all the developed countries are opposing among themselves to create new alternative low cost fuel known as biodiesel which should be sustainable and also friendly to environment.

1.2 Problem Statement

The increasing crude oil petroleum prices (Genc, 2017), limited resources of fossil fuels (Foster et al., 2017) and environmental concern have led to the search for new alternatives fuels which are more sustainable, energy conservation, efficiency and environmental friendly (Board, 2015). In this current study, biodiesel production from transesterification reaction is dedicated by application of heterogeneous catalyst using waste cooking oil.

Up till now, there is no researcher who studies MgO/CaO and Fe_2O_3/CaO using waste cooking oil. Among various catalysts available, metal oxides, MgO and Fe_2O_3 have attracted substantial attention due to their promising potential in biodiesel production. Due to its certain properties, including stoichiometry and composition, cation valence, redox properties, acid-base character, and crystal and electronic structure, researchers normally consider MgO as a promising high surface area heterogeneous catalyst support, additive, and promoter for many kinds of chemical reactions. Moreover, the existence of

MgO as a promoter catalyst also transforms the electronic state of the overall catalytic performance by electron transfer between the native catalyst and MgO. In the meantime, the method, chemical composition, and condition in the preparation of MgO are the important aspects affecting its surface and catalytic properties. Consequently, MgO with a high surface area and nanocrystalline structure has promising applications for some reactions, including transesterification process. Meanwhile, iron (Fe) is a catalyst for some imperative industrial chemical reactions and has some advantage which is plentiful and inexpensive, therefore making the use of it on the metric ton scale viable. In the present work, for the first time, a series of MgO/CaO and Fe_2O_3/CaO were prepared.

1.3 Scope of Research

This research involved the synthesis of MgO/CaO and Fe_2O_3/CaO derived from waste shells of cockleshell as heterogeneous mixed metal oxide catalysts for biodiesel production using waste cooking oil (WCO). The best calcination temperature for CaO supported on Fe_2O_3 and MgO synthesis was investigated and characterized by using XRF, XRD, TGA, BET, TPD-NH_3, TPD-CO_2, SEM and SEM-EDX. Then, the catalyst was continued for optimization study. The condition of the transesterification reactions of WCO was also studied by investigating the effect of variable parameters such as catalyst loading, methanol-to-oil molar ratio and reaction time. The reusability of the MgO/CaO and Fe_2O_3/CaO catalysts were determined and the leaching of calcium, iron and magnesium species into the reaction product were confirmed by using atomic absorption spectroscopy (AAS) elemental analysis. The entire biodiesel product in the reaction was analyzed by using gas chromatography (GC-FID). Lastly, the biodiesel fuel standard quality properties were determined by using ASTM D6751 and European 14212 standard specifications.

1.3 Objectives

This dissertation aims to synthesize and modify calcium oxide catalysts derived from cockleshell. This study also discussed the physical and chemical properties of synthesized catalysts and feasibility of biodiesel production from waste cooking oil via transesterification reaction with methanol. In order to achieve the main aim, there are six research objectives have been addressed as follows:

1. To synthesize, screen and characterize calcium oxide derive from cockleshell.
2. To synthesize, screen and characterize MgO/CaO and Fe_2O_3/CaO catalysts using several methods such as X-ray Flourescence Spectroscopy (XRF), X-ray Diffraction (XRD) analysis, Thermogravimetric Analysis (TGA), Temperature Programmed Desorption of Ammonia (TPD-NH_3), Temperature Programmed Desorption of Carbon Dioxide (TPD-CO_2), Brunaueur-Emmett-Teller (BET), Scanning Electron Microscopy-Energy Dispersive X-ray (SEM-EDX) and Scanning Electron Microscopy (SEM).
3. To optimize and investigate the biodiesel production by manipulating its parameters (catalyst loading, methanol-to-oil molar ratio and reaction time) and the reusability also leaching of MgO/CaO and Fe_2O_3/CaO catalysts.
4. To determine and evaluate the properties of WCO biodiesel.

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