UNIVERSITI PUTRA MALAYSIA

SYNTHESIS AND CHARACTERIZATION OF HETEROGENEOUS MIXED OXIDE CATALYSTS BASED ON EGG SHELL FOR BIODIESEL PRODUCTION FROM WASTE COOKING OIL

NASAR MANSIR

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By

NASAR MANSIR

Thesis submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirement for the Degree of Doctor of Philosophy

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DEDICATION

The thesis is wholly dedicated to my loving parents Alh Mansir Lawal and Hajiya Safiya Muhammad for their support and encouragement throughout my life.

Abstract of thesis submitted to the Senate of Universiti Putra Malaysia in fulfilment of the requirements for the degree of Doctor of Philosophy

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Chairman : Professor Taufiq-Yap Yun Hin, PhD
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Biodiesel is one of the most promising biofuel alternatives to conventional fossil fuel, considering its number of advantages particularly environmental benign and availability of feedstock. Conventionally, homogeneous catalytic system is used for the production of biodiesel in commercial scale using high grade feedstock such as soybean oil, palm oil and sunflower oil. However, the current biodiesel production process is no longer sustainable considering the high cost of feedstock and other environmental related issues associated to homogeneous catalyst system. Newly developed calcium oxide based catalysts doped with mixed bimetallic oxides were synthesized using simple wet impregnation method. The synthesized mixed oxide catalysts were used for the transesterification of waste cooking oil using normal reflux method to produce fatty acid methyl ester (FAME). The catalysts (Molybdenum Zirconia Calcium oxide (Mo-Zr/CaO), Manganese Zirconia Calcium oxide (Mn-Zr/CaO), Tungsten Zirconia Calcium oxide (W-Zr/CaO), and Tungsten Molybdenum Calcium oxide (W-Mo/CaO) were characterized to investigate their physico-chemical properties using various characterization techniques such as XRD, TPD, BET, SEM, EDX, TGA and FTIR. The basicity and acidity of the catalysts determined their activity towards transesterification reaction. The Mo-Zr/CaO and Mn-Zr/CaO catalysts achieved 90.1% and 92.1% FAME yield under the reaction temperature of 80 ^{o}C and reaction time of 3 h in both cases. W-Zr/CaO catalyst recorded biodiesel yield of 94.1% at 80 ^{o}C reaction temperature, & 1 h reaction time. W-Mo/CaO catalyst achieved the biodiesel yield of 96.2% at reaction temperature of 70 ^{o}C, & 2 h reaction time. The order of activity of the synthesized catalyst for FAME production is W-Mo/CaO > W-Zr/CaO > Mn-Zr/CaO > Mo-Zr/CaO. The Ca^{2+} leaching has reduced significantly with the increase in transition metal mixed oxide loading over the CaO surface. Additionally, all the synthesized catalysts could convert high FFA waste cooking oil to FAME at mild reaction conditions and be

reused and regenerated for subsequent biodiesel production cycle. The most stable catalyst (W-Mo/CaO) achieved 90 % FAME yield at 70 ^{o}C temperature and 2 h reaction time in the 5th reusability cycle. The synthesized biodiesel was tested and met the biodiesel standard quality parameters according to ASTM D67751 and EN 14214.

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

SINTESIS DAN PENCIRIAN MANGKIN HETEROGEN OKSIDA CAMPURAN BERASASKAN KULIT TELUR UNTUK PENGHASILAN BIODIESEL DARIPADA SISA MINYAK MASAK

Oleh

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Biodiesel merupakan salah satu bahan api bio yang menjanjikan alternatif kepada bahan api fosil, disebabkan oleh beberapa kelebihan terutamanya mersa alam dan sumber yang boleh diperbaharui. Sistem pemangkinan homogenus digunakan secara konvensional untuk penghasilan biodiesel bagi skala komersial menggunakan sumber bahan mentah bergred tinggi seperti minyak kacang soya, minyak sawit dan minyak bunga matahari. Walaubagaimanapun, penghasilan biodiesel semasa tidak lagi mampan berikutan peningkatan kos sumber bahan mentah dan isu-isu berkaitan alam sekitar yang melibatkan sistem pemangkinan homogenus. Mangkin berasaskan kalsium oksida yang baru dibangunkan didopkan bersama campuran oksida dwilogam yang disintesis menggunakan kaedah pengisitepuan basah. Mangkin oksida campuran yang disintesis digunakan untuk transesterifikasi sisa minyak masak menggunakan kaedah refluks normal untuk menghasilkan asid lemak metil ester (FAME). Mangkin (Molibdenum Zirkonia Kalsium oksida (Mo-Zr/CaO), Mangan Zirkonia Kalsium oksida (Mn-Zr/CaO), Tungsten Zirkonia Kalsium oksida (W-Zr/CaO), and Tungsten Molibdenum Kalsium oksida (W-Mo/CaO) dicirikan untuk mengenalpasti sifat fiziko-kimia menggunakan pelbagai teknik pencirian seperti XRD, TPD, BET, SEM, EDX, TGA dan FTIR. Sifat kebesan dan keasidan menentukan aktiviti mangkin terhadap tindak balas transesterifikasi. Mangkin Mo-Zr/CaO dan Mn-Zr/CaO menghasilkan 90.1% dan 92.1% FAME pada suhu tindak balas 80 ^{o}C dan 3 jam masa tindak balas untuk kedua-duanya. Mangkin W-Zr/CaO mencatatkan hasil biodiesel sebanyak 94.1 % pada suhu tindak balas 80 ^{o}C dan 1 jam masa tindak balas. Mangkin W-Mo/CaO menghasilkan biodiesel 96.2% pada suhu tindak balas 70 ^{o}C dan 2 jam tindak balas. Susunan aktiviti mangkin yang disintesis untuk penghasilan FAME adalah W-Mo/CaO > W-Zr/CaO > Mn-Zr/CaO > Mo-Zr/CaO. Perlunturan ion Ca^{2+} menunjukkan pengurangan yang ketara dengan peningkatan kandungan logam peralihan campuran oksida pada permukaan CaO.

Tambahan pula, semua mangkin yang disintesis mampu menukarkan FFA yang tinggi dalam sisa minyak masak kepada FAME pada keadaan sederhana dan boleh digunasemula untuk beberapa kitaran penghasilan biodiesel. Mangkin yang paling stabil (W-Mo/CaO) menghasilkan 90 % FAME pada suhu 70 ^{o}C dan 2 jam masa tindak balas dalam 5 kitaran. Biosiesel yang disintesis diuji dan mencapai parameter kualiti piawai berdasarkan kepada ASTM D67751 dan EN 14214

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This thesis was submitted to the Senate of the Universiti Putra Malaysia and has been accepted as a fulfilment of the requirement for the degree of Doctor of Philosophy. The members of the supervisory committee were as follows:

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This is to confirm:

- that the research conducted and the thesis writing was under our supervision;
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LIST OF ABBREVIATIONS

AAS	Atomic Absorption Spectroscopy
AOAC	Association of Official Analytical Chemists
ASTM	American Society for Testing Materials
AV	Acid Value
P	Actual gas pressure
υ	Adsorbed gas quality
BET	Brunauer Emmett Teller
R_1, R_2, R_3	Carbon chain of fatty acid
C_{IS}	Concentration of Internal Standard
CO_2 –TPD	Temperature Programmed Desorption of Carbon dioxide
CP	Cloud Point
DG	Diglycerides
EDX	Energy Dispersive X-ray
FAME	Fatty Acid Methyl Ester
FFA	Free Fatty Acid
FID	Flame Ionization Detector
FP	Flash Point
FTIR	Fourier Transform Infrared
FWHM	Full Width of Half Maximum
GC-MS	Gas chromatogram – mass spectroscopy
ICDD	International Centre for Diffraction Data
A_{IS}	Internal standard peak area
JCPDS	Joint Committee on Powder Diffraction Standard
MPOB	Malaysian Palm Oil Board
υm	Monolayer adsorbed gas quality
$MW_{fatty\ acid}$	Average molecular weight of fatty acid

$MW_{glycerol}$	Average molecular weight glycerol
MW_{water}	Average molecular weight of water
MW_{oil}	Average molecular weight of waste cooking oil
PME	Palm oil Methyl Ester
PP	Pour Point
P/P_o	Relative Pressure
ΣA	Total peak area of fatty acid methyl esters
S_{BET}	Total surface area
SEM	Scanning Electron Microscopy
SFA	Saturated Fatty Acid
SV	Saponification Value
TCD	Thermal Conductivity Detector
TG/DTA	Thermogravimetric and Differential Thermal Analysis
V_{IS}	Volume of the internal standard
WCO	Waste Cooking Oil
WFO	Waste Frying Oil
WES	Waste Egg Shell
WCPO	Waste Cooking Palm Oil
XRD	X-Ray Diffraction

CHAPTER 1

INTRODUCTION

1.1 Background

Conventional fossil fuel will continue to be a relevant key player as an energy source for human activities such as production of goods and transport services for decades to come (Galadima & Muraza, 2014). However, the global energy crisis and the possible future depletion of conventional fossil fuel, environmental problems as a result of poisonous gases especially carbon monoxide (CO) from automobile exhausts and industries are the greatest challenges of fossil fuel today. The aforementioned are the major drivers prompting the search for alternative renewable source of energy that could replace the conventional fossil fuel (Chouhan & Sarma, 2011; Muhammad *et al.*, 2015).

The current global economy depends solely on the production of goods and services, including transportation, which depends solely on petroleum based sources of energy. Apart from coal, nuclear power, natural gas and hydroelectricity that serve at different capacity in the provision of energy, transportation of goods and services sector alone is more than 90% dependence on fossil fuels (petroleum) with more than 60% annual global fuel consumption (Borges & Díaz 2012; Avhad & Marchetti 2015). Renewable sources of energy such as bio-fuels are generally considered as replaceable options alternative to traditional fossil fuel due to their environmental friendly and feedstock availability (Rabiah Nizah *et al.,* 2014; Shajaratun Nur *et al.*, 2014).

Biofuel is generally referred to as fuel either in liquid or gaseous form produced from biomass and used as fuel for internal combustion engines (cars, generators, ships and airplanes). Biofuels has proved to be economically viable considering the availability of feedstocks and eco-friendly as a result of biodegradability and less toxicity (Nigam & Singh, 2011). Other advantages of biodiesel over Petro-diesel include; the provision of independent economy that would employ rural people and reduce over dependence on fossil fuel economy. Biodiesel has improved combustion due to high oxygen content and could easily be blended with fossil based diesel in different proportions.

Biodiesel is one of the promising alternative source of energy considering its biodegradability and low emission of carbon dioxide, free sulphur and non-toxic nature (Fauzi & Amin., 2013; Mardhiah *et al.*, 2017). Another interesting thing about biodiesel is that it possesses all the chemical properties of traditional fossil fuel such as improved cetane number, high flash point low cloud point and good pour point (Galadima & Muraza, 2014).

As a result of rapid growth in human population, scientific and technological advancements in developing countries, the biodiesel demand is estimated to either doubled or tripled by the year 2020 and beyond (Galadima & Muraza, 2014). However, relevant studies have fully verified number of issues regarding biodiesel that is not convincingly addressed yet. Conversion of triglycerides into fatty acid methyl ester requires a reaction of the former with monohydric alcohol (methanol). Most of the researchers recommended that the short chain monohydric alcohol, typically methanol, ethanol and propanol with no distinct justification of which provides the best viscosity requirements in line with specifications by American Society of Testing and Materials (ASTM) and European standard (EN) (Galadima & Muraza, 2014).

Technically, biodiesel is not yet considered as a popular alternative source of energy to conventional fossil fuel worldwide considering issues related to feedstock and catalyst system. High cost of food grade oil, which covers more than 80% of total biodiesel production, and conventional homogeneous catalyst system that require large amounts of water and energy during separation after the reaction, makes biodiesel production difficult and expensive. Inadequate of raw material and effective catalytic system, are the major problems facing commercialization of biodiesel to meet the global demand and the united nation millennium environmental policy. Hence the fundamental policy of biodiesel is not yet achieved.

Biodiesel production in commercial scale at low cost and environmentally benign condition is the only way to make biodiesel globally accepted fuel for industrial and transport services. This can only be achieved through diversifying and utilization of different low cost feedstock for biodiesel synthesis. The low cost feedstock for biodiesel include palm fatty acid distillate (PFAD), waste cooking oil, *jatropha curas oil* etc (Bhuiya *et al.*, 2014). Considering the free fatty acid (FFA) content of these feedstock, separation difficulty and environmental issues of homogeneous catalyst system after reaction, heterogeneous catalyst is regarded as the best option for sustainable commercial production of biodiesel with less difficulty.

Generally a catalyst is defined as any material or substance that speeds up the rate of chemical reaction by lowering its activation energy (Peter & Julio, 2010). It is usually added in a minute amount in comparison to the quantity of the reactants, which is not consumed during the chemical reaction; it is also known as an initiator. However, in some cases the catalyst inhibits the reaction by being consumed and regenerated, while in other cases it seems not to include in the process and functions by high calibre of surface characteristics (Ertl *et al.*, 2008). Catalysis generally represents the vital technology for accelerating the essential chemical conversion, which is a key to recognize environmentally friendly and commercially feasible reactions for transforming energy carriers to direct usable energy. However, the use of heterogeneous catalysts for chemical conversions not only decreases the total energy input needed for production processes, but also improves two considerable catalyst aspects, i.e. selectivity and thermal stability, thus leading to ecologically benign green technology.

1.2 Problem statement

The extensively available feedstocks make biodiesel production a striking field to invest in and enlarge. Nevertheless, current employed manufacturing processes produced an expensive renewable-based fuel in contrast to conventional fossil-based fuel, which is attributed to feedstock and manufacturing costs.

A conventional catalytic system (homogeneous catalyst) adopted for the production of biodiesel in commercial scale is only fitting for the higher grade vegetable oils. Continual use of such oil as feedstock for fuel may pose hunger threat due to food versus fuel competition in the near future. Besides, continual usage of liquid catalysts will make biodiesel not only difficult, but pollute the environment through the generated waste water during washing of catalyst.

Sustainable biodiesel production should employ the usage of low grade feedstock such as waste cooking oil, palm fatty acid distillate (PFAD), which would maintain a new route being cheap and accessible to overcome the associated problems with food grade oil. Heterogeneous catalyst has revealed less marked operational difficulty when compared to homogeneous catalysts, as they have the advantages of easy separation after use, reused severally, environmentally benign, low cost and easy to dispose after use.

However, most of heterogeneous catalysts for biodiesel production are associated to severe reaction conditions during the reaction process, which are considered as one of the main obstacles to this category of catalyst. This project will focus more on developing new heterogeneous catalyst system that could convert the low grade feedstock specifically waste cooking oil with high free fatty acid (FFA) and some impurities such as water content at mild conditions (low reaction temperature ≤ 80 ^{o}C, short reaction time ≤3 h, low catalyst loading and moderate methanol to oil molar ratio). The catalytic activity of these catalysts would be improved to achieve high biodiesel yield at reduced severe reaction conditions by providing the appropriate basic and acid site densities in the catalysts samples. Leaching of catalyst active sites of most heterogeneous catalysts is another problem that greatly affects the final biodiesel yield. The newly synthesized catalysts should also made to be be highly stable and reduced in leaching of active sites and therefore record high yield and reused severally before losing the catalytic activity.

Mixed metal oxides catalysts from CaO, a highly basic material and transition metals oxides were developed (Mo-Zr/CaO, Mn-Zr/CaO, W-Zr/CaO, and W-Mo/CaO) via wet impregnation method. The CaO was synthesised from egg shell. Physico-chemical properties of the mixed oxide catalysts were determined through different characterization techniques. The catalyst synthesis was optimized by varying transition metal oxide composition over the CaO surface. The catalytic activity of the synthesized catalysts was determined via transesterification reaction of waste cooking oil to biodiesel. The prepared catalysts could have reduced leaching of Ca^{2+} metal ions in the produced biodiesel. The catalysts reusability was evaluated, which further ascertained the stability of the newly developed catalysts.

1.3 The scope of the research

The scope of this study includes the synthesis and characterizations of mixed metal oxides supported CaO from the egg shell for the transesterification of high FFA waste cooking oil to biodiesel. The catalyst was synthesized by simple wet impregnation method. The transition metal oxides such as WO_3, ZrO_2, MnO_3 and MoO_3 were made to bimetallic oxide and then impregnated on to the egg shell powder (CaO). The prepared catalysts were characterized to investigate their physico-chemical properties using XRD, TPD, SEM, EDX, TGA BET and FTIR techniques. The bimetallic oxide supported CaO displayed excellent physico-chemical properties that enhanced their catalytic performance on the high FFA waste cooking oil for biodiesel production. Catalysts in different stoichiometric composition were synthesized and tested for transesterification reaction of waste cooking oil. The best catalyst system was optimized for biodiesel production under four independent reaction parameters, which includes reaction temperature (60-100^oC), methanol to oil molar ratio (5-25 wt%) reaction time (1-5h), and catalyst loading (1-5wt%). The catalyst reusability, stability and yield were investigated. Leaching of catalytic active phase is the major drawback of CaO based catalysts. The leaching of catalyst active sites was investigated by studying the effect of bimetallic mixed oxide loading on CaO.

1.4 Objectives of the research

The main objectives of this research are itemized as follows;

1. To synthesize and characterized the mixed metal oxides modified eggshell derived catalysts.
2. To investigate the catalytic activity of the synthesized catalysts for biodiesel production from waste cooking oil.
3. To optimize the reaction conditions for biodiesel production.
4. To evaluate the reusability and leaching of the synthesized catalysts.
5. To compare the physico-chemical properties of the produced biodiesel with the standard biodiesel fuel properties according to the ASTM and EN standards

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