SURFACE MODIFIED MAGNESIUM OXIDES FOR PRODUCTION OF BIODIESEL VIA HETEROGENEOUS BASE CATALYST TRANSESTERIFICATION OF PALM OIL

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Dedicated with much love and affection
to my beloved father ***Samadi Kasmuri*** *and my amazing mother* ***Zainab Ibrahim***
and to my helpful brothers, ***Mohd Syazmil, Mohd Syazli, Mohd Syazaruddin***
and someone that never been forgotten ***Muhammad Syazmi Amir***
and a special dedication to my fiancé ***Megat Husin Yunus***
with deep gratitude for all their love in me
Your love would be so much worth to me…

To my supervisor
Prof. Dr. Abdul Rahim Yacob
Thanks for the kindness in giving me an opportunity and encouragement
By trusting me in everything I do…

To my beloved ***friends***
who always support me and always there for me
when I need them
thanks for understand me well…

To all ***lab assistants, lecturers*** *and my* ***research team,***
Special thanks for all your kindness in helping me…

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PREFACE

This thesis is the result of my work carried out in Department of Chemistry, Universiti Teknologi Malaysia between December 2008 until December 2010 under the supervision of Prof. Dr. Abd. Rahim Yacob. Part of my work described in this thesis has been sent for exhibition participations and reported in the following publications:

1. Abd. Rahim Yacob, Mohd Khairul Asyraf Amat Mustajab and Nur Syazeila Samadi. (2008). Surface Modified MgO for Base Catalytic Conversion of Palm Oil to Biodiesel. Exhibition participation for the Industrial Art and Technology Exhibition (INATEX) 2008. Bronze medal award. Universiti Teknologi Malaysia, Skudai, Johor

2. Abd. Rahim Yacob, Mohd Khairul Asyraf Amat Mustajab and Nur Syazeila Samadi. (2008). Surface Modified MgO for Base Catalytic Conversion of Palm Oil to Biodiesel. Exhibition participation for the Malaysia Technology Expo (MTE) 2008. Bronze medal award. Putra World Trade Centre. Kuala Lumpur. 19 – 21 February 2009

3. Abd. Rahim Yacob, Mohd Khairul Asyraf Amat Mustajab and Nur Syazeila Samadi. (2009). Palm Oil Biodiesel: Transesterification of Nano CaO. Exhibition participation for the Industrial Art and Technology Exhibition (INATEX) 2009. Silver medal award. Universiti Teknologi Malaysia, Skudai, Johor

4. Abd. Rahim Yacob, Mohd Khairul Asyraf Amat Mustajab and Nur Syazeila Samadi. (2010). Palm Oil Biodiesel: Transesterification of Nano CaO. Exhibition participation for the Malaysia Technology Expo (MTE) 2010. Silver medal award. Putra World Trade Centre. Kuala Lumpur. 4 – 6 February 2009

5. Abd. Rahim Yacob, Mohd Khairul Asyraf Amat Mustajab and Nur Syazeila Samadi. (2009). Calcination Temperature of Nano MgO on Base Transesterification of Palm Oil. Submitted full paper for International Conference on Energy and Environment 2009. Singapore. 26 – 28 August 2009

6. Abd. Rahim Yacob, Nur Syazeila Samadi and Mohd Khairul Asyraf Amat Mustajab. (2010). Activation Temperature Effect on the Basic Strength of Prepared Aerogel MgO (AP-MgO). Submitted full paperwork for International Journal of Basic and Applied Sciences IJBAS-IJENS Vol. 10 Issues 02. Pages 118-121.

7. Abd. Rahim Yacob, Mohd Khairul Asyraf Amat Mustajab and Nur Syazeila Samadi. (2010). Physical and Basic Strength of Prepared Nano Structured MgO. Submitted full paper for 2010 International Conference on Mechanical and Electrical Technology. Singapore. 10 – 11 September 2010.

8. Abdul Rahim Yacob and Nur Syazeila Samadi. Photosensitization Study of Triethylamine and Acetophenone in NaY Zeolite Supercages Using ESR. Poster presentation of Regional Annual Fundamental Science Symposium 2010. Kuala Lumpur. 8-9 June 2010.

9. Abd. Rahim Yacob, Nur Syazeila Samadi and Mohd Khairul Asyraf Amat Mustajab. (2009). Synthesized Surface Modified Metal Oxide in Base-Catalytic Transesterification for Biodiesel Production. Poster presentation of 2nd Junior Chemist Colloqioum. Sarawak. 1-2 July 2009.

ABSTRACT

Surface morphology of prepared alkaline earth metal oxide MgO contributes to the effect of basicity and reactivity in heterogeneous catalysis reactions. In this study, two methods to prepare surface modified MgO were employed for comparison. The first method is by conventional (CP-MgO) and the second method by aerogel (AP-MgO). The methods of preparation will differentiate the effect of size and morphology towards basicity and reactivity. For conventional method, commercial magnesium oxide (CM-MgO) was first transformed into its hydroxide, CP-$Mg(OH)_2$ followed by heat under vacuum at 10^{-3} mbar. For aerogel method, magnesium ribbon was transformed into its magnesium hydroxide AP-$Mg(OH)_2$ followed by heat and vacuum as in conventional method. In both methods, magnesium hydroxides were heated under vacuum at temperatures 100, 200, 300, 400, 500, 600 and 700°C respectively. The surface modified magnesium oxides were then characterized. Detailed characterization involving Fourier Transform Infra-Red (FTIR), Thermogravimetry Analysis (TGA), X-Ray Powder Diffraction (XRD), Nitrogen Gas Adsorption, Field Emission Scanning Electron Microscope (FESEM), Energy Dispersive X-Ray (EDX) and basicity titration has allowed a rationale explanation for the high chemical reactivities. In this study, the prepared aerogel AP-MgO, had a high surface area compared to the conventional CP-MgO. This is however due to the smaller nano particle size of AP-MgO as compared to CP-MgO. The main factors of AP-MgO which contributes to the high reactivity are due to the pore volume and size distribution, unusual surface morphologies, and trace residual surface of –OH and –OCH_3. This will then effect the percentage conversion of transesterification reaction when compared to CP-MgO. To study the reactivity both of the best prepared CP-MgO and AP-MgO were used in base heterogeneous catalyst for transesterification of palm oil to fatty acid methyl ester or also known as biodiesel. The resulting transesterification reaction of palm oil to biodiesel was then studied using Gas Chromatography equipped with Flame Ionization Detector (GC-FID) and the highest percentage conversion of biodiesel of the best catalyst used was AP-MgO at 700 ^{o}C is 94.3%. Further analysis of the biodiesel products was then characterised using FTIR and Gas Chromatography equipped with Mass Spectrometry (GC-MS) to determine the components of complex organic mixtures.

ABSTRAK

Morfologi permukaan daripada penyediaan logam oksida alkali bumi MgO memberi kesan terhadap kebesan dan kereaktifan dalam tindak balas sebagai pemangkin heterogen. Dalam kajian ini, dua kaedah telah digunakan dalam penyediaan modifikasi permukaan MgO sebagai perbezaan. Kaedah pertama adalah menggunakan kaedah konvensional (CP-MgO) dan kaedah kedua menggunakan aerogel (AP-MgO). Kaedah penyediaan pemangkin ini akan memberi kesan perbezaan saiz dan morfologi terhadap kebesan dan kereaktifan. Melalui kaedah konvensional, magnesium oksida komersial (CM-MgO) telah ditindak balaskan untuk ditukarkan kepada bentuk hidroksida, CP-$Mg(OH)_2$ dan diikuti dengan pemanasan menggunakan vakum pada 10^{-3} mbar. Melalui kaedah aerogel pula, pita magnesium telah diubah kepada magnesium hidroksida, AP-$Mg(OH)_2$ dan kemudian dipanaskan dan divakumkan seperti kaedah konvensional. Kedua-dua magnesium hidroksida telah dipanaskan menggunakan vakum pada suhu 100, 200, 300, 400, 500, 600 dan 700°C. Magnesium oksida yang telah diubahsuai ini dilabel dan dicirikan menggunakan kaedah kimia setiap satunya. Pencirian terperinci melibatkan Transformasi Fourier Infra Merah (FTIR), Analisis Thermogravimetrik (TGA), Analisis Pembelauan Sinar-X (XRD), Penjerapan gas Nitrogen, Mikroskop Imbasan Elektron Emisi Medan (FESEM), Sinar-X Penyebaran Tenaga (EDX) dan kaedah penitratan bes untuk memberikan penjelasan rasional terhadap kereaktifan kimia pemangkin. Dalam kajian ini, penyediaan melalui kaedah aerogel AP-MgO mempunyai luas permukaan yang tinggi berbanding kaedah konvensional CP-MgO. Ini adalah kerana saiz nano partikel AP-MgO yang kecil berbanding CP-MgO. Faktor utama AP-MgO yang menyumbang kepada tindak balas yang tinggi adalah disebabkan isipadu liang, pengagihan saiz, morfologi permukaan yang berubah dan peninggalan sisa –OH dan –OCH_3 ini lantas permukaan yang mempunyai kesan dan menyebabkan peratusan perolehan pertukaran dalam tindakbalas transesterifikasi berbanding CP-MgO. Untuk mempelajari kedua-dua reaktiviti sebagai pemangkin terbaik, CP-MgO dan AP-MgO telah digunakan sebagai pemangkin bes heterogen untuk tindak balas transesterifikasi minyak kelapa sawit kepada biodiesel. Keputusan dalam tindak balas transesterifikasi minyak kelapa sawit kepada biodiesel dengan memilih pemangkin terbaik, kemudian dikaji dengan menggunakan kromatografi gas yang dilengkapkan dengan Flame Ionization Detector (GC-FID) dan peratusan pertukaran biodiesel paling tinggi adalah dengan menggunakan AP-MgO yang dikalsinkan pada suhu 700°C iaitu sebanyak 94.3 %. Analisis mendalam terhadap produk biodiesel telah dicirikan lagi dengan menggunakan FTIR dan kromatografi gas yang dilengkapkan dengan Spektrometri Jisim (GC-MS) untuk menentukan komponen dalam campuran kompleks organik.

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LIST OF SYMBOLS / ABBREVIATIONS

$^{\circ}C$	-	degree Celsius
μm	-	micrometer
BET	-	Brunauer-Emmett-Teller
cm	-	centimetre
DTA	-	Differential thermal analysis
EDX	-	Energy Dispersive X-ray analysis
Eq.	-	equation
FTIR	-	Fourier Transformation Infra red
g	-	gram
K	-	Kelvin
NA	-	nitrogen adsorption
mL	-	millilitre
nm	-	nanometer
rpm	-	rotation per minute
TG	-	Thermogravimetry
XRD	-	X-ray Diffraction
θ	-	Half angle of diffraction beam

λ	-	wavelength
MgO	-	Magnesium Oxide
CM-MgO	-	Commercial Magnesium Oxide
CP-MgO	-	Commercial Prepared Magnesium Oxide
AP-MgO	-	Aerogel Prepared Magnesium Oxide
$Mg(OH)_2$	-	Magnesium Hydroxide
CP- $Mg(OH)_2$	-	Commercial Prepared Magnesium Hydroxide
AP- $Mg(OH)_2$	-	Aerogel Prepared Magnesium Hydroxide
BaO	-	Barium Oxide
SrO	-	Strontium Oxide
CaO	-	Calcium Oxide
%	-	percentage
FESEM	-	Field Emission Scanning Electron Microscope
NA	-	Nitrogen gas Adsorption
GC-FID	-	Gas Chromatography equipped with Flame Ionization Detector
GC-MS	-	Gas Chromatography equipped with Mass Spectrometry
KBr	-	Potassium Bromide
FAME	-	Fatty acid methyl ester
Ni	-	Nickel
CO_2	-	Carbon Dioxide
ASTM	-	American Standard Testing Material
SO_2	-	Sulfur dioxide
m^2/g	-	milli square per gram

OH	-	Hydroxide
IV	-	Iodine Value
CPO	-	Crude Palm Oil
CO	-	Carbon Monoxide
NO_x	-	Nitrogen Oxides
KOH	-	Potassium Hydroxide
NaOH	-	Sodium Hydroxide
FFA	-	Free fatty acid
BDDT	-	Brunauer, Demming, Demming and Teller
UV	-	Ultraviolet Radiation
FWHM	-	full width half maximum
SEM	-	Scanning Electron Microscope

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CHAPTER 1

INTRODUCTION

1.1 Background of Research

The reactivity of catalyst in some cases are based on the porosity and cavity. This is true for zeolite. This idea is used to produce catalyst by using other compound than zeolite. Surface modified metal oxide such as magnesium oxide on the other hand can be a good catalyst since it has high porosity, simple structure and high cavity. It can also be used as an adsorbent.

The preparation process of surface modified with higher surface area metal oxide is easy and economical in cost compared with zeolite and noble catalyst. In this research, the surface modified magnesium oxide was produced via dehydration process of commercial magnesium oxide (CP-MgO) and aerogel magnesium oxide (AP-MgO) method. The aim of this research is to study on the preparation and characterization of high surface area metal oxide and the reaction as a catalyst.

In this study, the commercial magnesium oxide and magnesium ribbon were used as the precursor to produce surface modified commercial prepared magnesium oxide (CP-MgO) and aerogel prepared magnesium oxide (AP-MgO) respectively. Both were activated at different temperature from 100^{o}C till 700^{o}C respectively. The research was emphasized on the preparation and characterization of surface modified MgO and the reaction as a heterogeneous base catalyst for the transesterification reaction of palm oil to biodiesel.

1.2 Problem of Statement

The reserves of nonrenewable resource such as oil can be considered economically depleted when 80% of the supply resources have been used and the remaining 20% is too expensive to extract. According to Puppan (2001), the oil extinction that its reserves may be 80% depleted within 35-84 years, depending on how rapidly the oil used. At the current rate of consumption, the oil reserves predicted will be last for another 44 years. The oil that have not been covered yet is thought to exist might last around another 20-40 years ahead. Instead of remaining the amount of oil at the same current level, the global oil consumption is projected to increase by about 25% by year 2010. This somehow will hasten the gradual depletion of oil reserve.

The gradual depletion of petroleum reserves gives big influence interest in finding alternative source of energy. Furthermore, world oil demand driven by economic development, posted the highest growth rate in recent years and the high price of oil also created desire to find renewable and sustainable alternative energy to decrease dependences in petroleum.

The last few decades, a substantial amount of research have been conducted in order to find a new renewable and sustainable energy source to substitute the usage of petrol fuel. A researchers Marchetti *et* al. (2007) concluded that there is one promosing renewable source of energy that can substitute the nonrenewable energy that is biodiesel.

Currently, biodiesel which also known as fatty acid methyl ester (FAME) is commonly produced by performing a transesterification reaction of renewable lipid feedstock such as animal fat or vegetable oil with homogeneous base catalysts such as KOH or NaOH dissolved in methanol. However, the produced FAME needs to undergo purification process to remove impurities such as base catalyst and glycerine by using a water washing process and this somehow will requires the disposal of a large amount of basic water.

1.3 Objectives of Research

The objectives of this research are to prepare Surface Modified Commercial Prepared MgO (CP-MgO) and Surface Modified Aerogel Prepared MgO (AP-MgO) which varies in temperature from 100°C-700°C respectively and characterize all the prepared magnesium oxide. Secondly, to investigate the prepared magnesium oxide catalytic capabilities as a solid base catalyst in the transesterification of palm oil for biodiesel production.

1.4 Significance of Research

This research has several importances to the as the substitution of the petroleum based oil such as:

1) To substitute petrol fuel which is nonrenewable source of energy with renewable resources such as vegetable oil
2) To enhances the usage of magnesium oxide base catalyst for other applications.
3) To reduced the green house gases effect of production of waste water and reduce global warming phenomenon.
4) To develop a new alternative energy sources other than petroleum resources from fuel.

1.5 Scope of Research

The scope of the research is to prepare and characterize seven types of surface modified MgO (CP-MgO and AP-MgO) which differs in temperature of activation. The prepared surface modified MgO was prepared at various temperatures; 100°C, 200°C, 300°C, 400°C, 500°C, 600°C and 700°C respectively. Resulting CP-MgO and AP-MgO samples were characterized using spectroscopy instrument which include Fourier Transform Infra-Red (FTIR). To have better understanding on the surface and morphological properties, Nitrogen Gas Adsorption (NA) and Field Emission Scanning Electron Microscopes (FESEM), Energy Dispersive X-Ray (EDX) X-Ray Diffractometer (XRD) and Thermogravimetry Analysis were used. For determination of bacisity of magnesium oxide, basicity titration was used. In reaction as a catalyst for transesterification of palm oil to biodiesel, samples were analyzed using Fourier Transform Infrared (FTIR), Gas Chromatography equipped with Flame Ionization Detector (GC-FID) and Gas Chromatography – Mass Spectrometry (GC-MS). Figure 1.1 shows the schematic layout of the research scope.

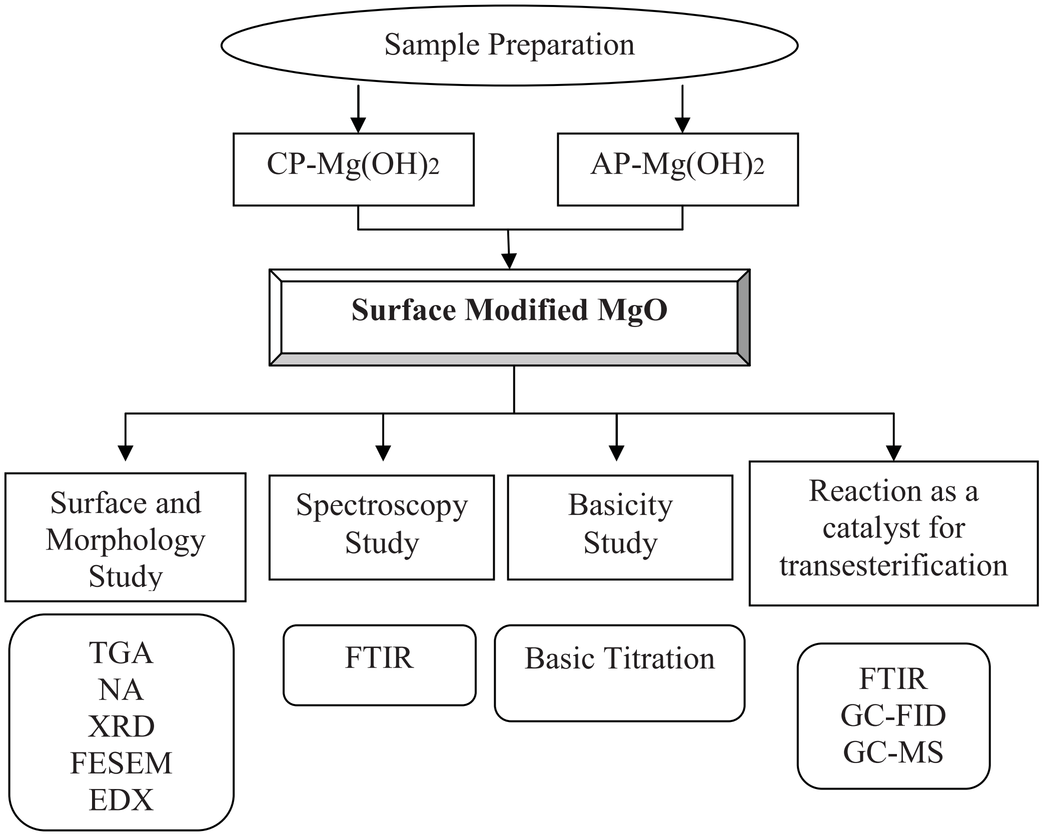

Figure 1.1 Schematic Layout of Research Scope

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