PREPARATION, CHARACTERIZATION AND MECHANISTIC STUDY OF ALUMINA SUPPORTED CALCIUM OXIDE BASED CATALYSTS IN TRANSESTERIFICATION OF REFINED COOKING OIL

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A Special Dedication to my beloved family especially to:

My sweetheart Mohd Norhelmi,
My cute little flower Siti Aisyah Humaira,
My sweet mums Habsah and Sepiah
My handsome dads Mohd Kamal and Samsudin
My supportive sisters Ashikin, Aishah and Aini
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For all the love, supports and continues doa to complete this work

"I am the person I am today because of all the people who have shaped me in every way"

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ABSTRACT

Biodiesel synthesized from the transesterification reaction using heterogeneous base catalyst has been extensively studied over the past decades. In this research, a series of alumina-supported catalysts were synthesized utilizing CaO and MgO via impregnation with transition metal oxides of Cu, Zn and Ni. Through the catalyst screening process, $Cu/Ni/Ca/Al_2O_3$ and $Cu/Zn/Ca/Al_2O_3$ were selected, and further studies were carried out for optimization of several reaction parameters. The optimum calcination temperature of $Cu/Ni/Ca/Al_2O_3$ was at 700ºC, while that of $Cu/Zn/Ca/Al_2O_3$ was at 800ºC. The ratio of co-catalyst to based for both the catalysts was 3:7:90 wt%, with two times of alumina coatings performed on the samples. Study of the surface morphology by FESEM and TEM revealed the existence of agglomerated platelet-shape particles on the catalysts surface. XRD analysis showed the crystallinity of synthesized catalysts were generally poor, with the particle sizes of less than 50 nm. The $Cu/Ni/Ca(3:7:90)/Al_2O_3$ catalyst with BET surface area of 140 m^2/g, exhibited a higher amount of weak and moderate basic sites (4.02 mmol/g) when compared to the $Cu/Zn/Ca(3:7:90)/Al_2O_3$ catalyst, as obtained from the CO_2-TPD data. The optimum conditions were found as reaction temperature of 65ºC, catalyst loading of 4 wt% and oil to methanol molar ratio of 1:16, to achieve 90.12% of biodiesel production for 90 minutes of reaction time, in the transesterification of refined cooking oil over the $Cu/Ni/Ca(3:7:90)/Al_2O_3$ catalyst. Meanwhile, the $Cu/Zn/Ca(3:7:90)/Al_2O_3$ with 10 wt% catalyst loading underwent 180 minutes of reaction time and subsequent achievement of 82.34% of biodiesel production. The validated data from RSM analysis indicated that the selected model was adequate with a percentage error less than 5%. Mechanistic study of the catalyst surface using FTIR and the GC-FID analysis of the transesterification product showed that both the catalysts obeyed the Langmuir mechanism rule and capable to produce the *cis* and *trans* isomers of oleic acid methyl ester. The biodiesel produced complied with the quality standard and specification recommended by the American Society for Testing Materials D6751.

ABSTRAK

Biodiesel yang disintesis daripada tindak balas transesterifikasi menggunakan mangkin bes heterogen telah dikaji secara meluas sejak berdekad yang lalu. Dalam penyelidikan ini, satu siri mangkin berpenyokong alumina telah disintesis menggunakan CaO dan MgO secara pengisitepuan oksida logam peralihan daripada Cu, Zn dan Ni. Melalui proses penyaringan mangkin, $Cu/Ni/Ca/Al_2O_3$ dan $Cu/Zn/Ca/Al_2O_3$ telah terpilih dan kajian lanjut telah dilakukan untuk pengoptimuman beberapa parameter tindak balas. Suhu pengkalsinan optimum bagi $Cu/Ni/Ca/Al_2O_3$ ialah pada 700ºC, manakala bagi $Cu/Zn/Ca/Al_2O_3$ pada 800ºC. Nisbah ko-mangkin kepada asas untuk kedua-dua mangkin adalah 3:7:90 wt% dengan dua kali salutan alumina. Kajian morfologi permukaan menggunakan FESEM dan TEM mendedahkan kewujudan zarah berbentuk platelet yang teraglomerat pada permukaan mangkin. Analisis XRD menunjukkan kehabluran mangkin yang disintesis pada umumnya adalah rendah, dengan saiz zarah di bawah 50 nm. Mangkin $Cu/Ni/Ca(3:7:90)/Al_2O_3$ dengan luas permukaan BET 140 m^2/g, mempamerkan jumlah tapak bes lemah dan sederhana (4.02 mmol/g) yang lebih tinggi apabila dibandingkan dengan mangkin $Cu/Zn/Ca(3:7:90)/Al_2O_3$, seperti yang diperoleh dari data CO_2-TPD. Keadaan optimum adalah suhu tindak balas 65ºC, muatan mangkin 4 wt% dan nisbah molar minyak kepada metanol 1:16, untuk mencapai 90.12% pengeluaran biodiesel bagi masa tindak balas 90 minit, dalam transesterifikasi minyak masak bertapis dengan menggunakan mangkin $Cu/Ni/Ca(3:7:90)/Al_2O_3$. Sementara itu, $Cu/Zn/Ca(3:7:90)/Al_2O_3$ dengan muatan mangkin 10 wt% menjalani 180 minit masa tindak balas, diikuti dengan pencapaian 82.34% pengeluaran biodiesel. Data yang disahkan daripada analisis RSM menunjukkan bahawa model yang dipilih adalah mencukupi dengan peratus ralat kurang daripada 5%. Kajian mekanistik ke atas permukaan mangkin menggunakan FTIR dan analisis produk menggunakan GC-FID menunjukkan bahawa kedua-dua mangkin mematuhi peraturan mekanisme Langmuir dan boleh menghasilkan isomer *cis* dan *trans* metil ester asid oleik. Biodiesel yang dihasilkan telah mematuhi spesifikasi dan piawaian yang diperakukan oleh Persatuan Amerika untuk Bahan Ujian D6751.

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LIST OF ABBREVIATIONS

ANOVA	Analysis of Variance
2D	Two Dimensional
3D	Three Dimensional
BBD	Box-Behnken Design
BET	Brunauer-Emmet-Teller
c	cubic
FESEM-EDX	Field Emission Scanning Electron Microscope - Energy Dispersive X-Ray
FID	Flame Ionization Detector
FTIR	Fourier Transform Infrared Spectroscopy
GC	Gas Chromatography
h	Hexagonal
JCPDS	Joint Committee on Powder Diffraction Standard
RSM	Response Surface Methodology
TAN	Total Acid Number
TGA-DTA	Thermogravimetry Analysis-Differential Thermal Analysis
TPD	Temperature Programmed Desorption
XPS	X-Ray Photoelectron Spectroscopy
XRD	X-Ray Diffraction
NA	Nitrogen Adsorption
RSM	Response Surface Methodology
TEM	Transmission Electron Microcopy

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CHAPTER 1

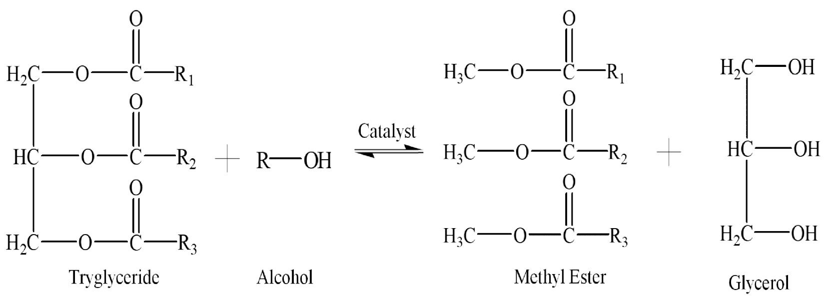
INTRODUCTION

1.1 Biodiesel as Alternative Energy Resources

Fossil fuel was naturally produced from organic matter that decays deep in the earth over the course of millions years (Droege *et al.,* 2002). Since the industrial development was started, a large quantity of fossil fuel was utilized to drive the economy to huge number of people (Shafiee *et al.,* 2009). From the data analysis in global energy statistical 2017, petroleum oil is one of the most fossil fuel resources that are widely used in Malaysia especially in transportation. Thus, it is estimated that world crude oil reserves will vanish at the rate of 4 billion tons a year and it has become increasingly evident that humanity faces a number of unprecedented challenges in terms of future energy resources and consumption (Atabani *et al.,* 2012).

On the other hand, the usage of petroleum based fossil fuel (petro diesel) may leads to the several disadvantageous. The combustion of petro diesel may releases the greenhouse gases to the environmental surrounding and contributes to the global warming. The emission from the combustion can produce nitrogen oxide (NO_x), sulphur oxide (SO_x) and aromatic particulates which harmed the human health (Zhong *et al.,* 2017). Therefore, several researches were conducted to solve this critical issue by searching new alternative energy resources which were technically feasible, economically competitive, environmentally acceptable, and readily available. Above all, biodiesel is one of the best renewable energy resources to substitute the petro diesel and contribute more advantageous due to environmentally benign.

Biodiesel which are derived from vegetable oil and animals fats through transesterification reaction has become one of the renewable energy resources that have similar properties as petro diesel. The reaction takes place when one mole of triglycerides (contain in vegetable oil and animal fats) reacted with 3 mole of methanol in the presence of catalyst (heterogeneous or homogeneous acid, basic or enzymatic) to produced three mole of methyl ester (biodiesel) and 1 mole of glycerol. The overall reaction is presented as in Equation 1.1.



(1.1)

Biodiesel is a liquid fuel usually stated as B100 was in un-homogenised form. Like petroleum diesel, biodiesel was used as fuel in compression ignition engines. It has frequently used as a blend with regular diesel fuel and can be used in several diesel vehicles without any engine modification. The most common biodiesel blend is B20, which was the combination of 6% to 20% biodiesel blended with petroleum diesel. Meanwhile, B5 (5% biodiesel, 95% diesel) was commonly used in fleets.

There were several advantageous of using biodiesel as petro diesel substitution. For example, it can improve the fuel lubricity and raises the cetane number of the fuel. The diesel engines depend upon the lubricity of the fuel to stay moving components from wearing untimely. Besides that, the emissions from the biodiesel were better than regular diesel and produced less NOx gases. In addition, it is also easy to use and less vehicle modification or any fuelling equipment was needed. Moreover, from environmental side of view, biodiesel is helping in reducing pollution and improve health by lowering the emission of CO_2 thus may reduces the effect of global warming. Moreover, it is also safer to handle due to less toxic and easy to be stored compared to petro diesel.

In producing biodiesel, the feedstock was the major reactant that has to be taken into account before the transesterification reaction takes place. There were several feedstocks that have been used in order to produce high quality of biodiesel. Previous study proved that the biodiesel can be synthesize from palm oil, canola oil, sunflower oil, corn oil, rice bran oil and rapeseed oil (Canakci e*t al.,* 2008). However, the production of biodiesel from first generation vegetable oil has a limitation issues. The crisis was raised up due to the competing with arable land culture and human food supply. Due to this problem, the idea of using non edible feedstock (second generation of biodiesel) was generated since it may not give any negative impact especially for human and more environmental friendly.

Synthesized of biodiesel from non edible feedstock or known as second generation biodiesel was becoming a new idea to solve this crucial crisis (Mardhiah *et al.,* 2017). The biodiesel was synthesis from the whole plant matter, agricultural residue and processing waste has become the main attention among researchers. For example, there were studies that use microalgae plants as the feedstock to produce biodiesel (Chiaramonti *et al*., 2017). Besides that, the use of rise husk, oil palm leave, palm kernel and *Jatropha Curcas* was also reported to have a potential in producing a biodiesel (Karmakar *et al.,* 2010). However, the production was not reached the industrial scale especially for commercialization due to the source limitation.

Along with that, feedstock from waste cooking oil has been generating a new idea to produce biodiesel. It was due to the large quantities of waste cooking oils and animal fats generated throughout the country (Sudhir *et al.,* 2007). Management of oils and fats was a significant challenge due to the disposal problems which leads to the contamination of the water and land resources (Chhetry *et al.,* 2008). Even though some of this waste cooking oil is used for soap production, a major part of it was being discharged into the environment. Therefore in this study, refined cooking oil was introduced as the feedstock to produce biodiesel and offers significant advantages due to the reduction in environmental pollution and lowers in production cost.

1.2 Catalysts in Biodiesel Production

In transesterification reaction, the process involved both endothermic and reversible reaction. It can be carried out either in catalytic or non catalytic approach. It has been recognised that the catalyst acts by reducing the activation energy along the reaction pathway. The lower the activation barrier, the faster the reaction will takes place. Thus, the usage of catalyst was necessary in order to increase the production rate and saving the production time. In normal reaction, the catalyst was required to shift the equilibrium to the right and produce high production of biodiesel. Various catalysts were used in transesterification reaction such as heterogeneous, homogeneous and enzymatic catalyst. In addition, the catalyst can also be basic or acidic depends on the catalytic reaction and the mechanism pathway might be different (Atadashi *et al.,* 2013)

Homogeneous catalyst was widely used especially in industrial development due to the better performance with lower FFA content in the feedstock. Large amount of FFA (>1%) in the system may neutralized the basic catalyst and leads to the formation of soap and water (Ramachandran *et al.,* 2013). In addition, homogeneous base catalysts mainly dissolved in glycerol and alcohol after the reaction was completed. It cannot be recycled for the following batches and the purification process with water was needed. Thus, it may leads to the waste water problem which was harmful to the environmental (Thanh *et al.,* 2012).

Since the homogeneous catalyst leads to the several crises, thus heterogeneous catalyst was synthesis to substitute the using of homogeneous catalyst. Heterogeneous catalyst can be categorized as basic and acidic depends on the nature of the catalyst itself. In previous studies, heterogeneous catalyst such as metal oxide, zeolite and hydrotalcites catalyst was extensively used due to the ability to be separated from the final product and can be reused back in the next transesterification reaction (Kiakalaieh *et al.,* 2013). Most of these catalysts were alkaline metal oxides supported on materials with a large surface area and have high basicity (Wittoon *et al.,* 2014). Similar properties to homogeneous catalyst, solid base-catalysts are more active as compared to solid acid-catalysts.

In this study, the performance of bimetallic and trimetallic alumina supported basic catalyst was evaluated in transesterification of refined cooking oil to produce biodiesel. The catalyst was prepared via wetness impregnation method by incorporating the transition metal elements like copper (Cu), Nickel (Ni) and zinc (Zn) as dopant and co dopant. Meanwhile, calcium (Ca) and magnesium (Mg) was selected as the based and supported on gamma alumina. The development of this catalyst was rarely explored especially in transesterification reaction. Thus, it may become the advantage for this research to synthesis new catalyst which was lower in production cost and more environmental friendly.

1.3 Response Surface Methodology (RSM)

In this study, the experimental was done to determine the optimum condition of the catalyst prepared and transesterification reaction condition to produce high biodiesel production. Thus, in order to validate this optimization study, another tools was used which is called response surface methodology (RSM) analysis. RSM is defined as a collection of mathematical and statistical techniques for empirical model building. The objective is to optimize a response (output variable) which was influenced by several independent variables (input variables). An experiment is a series of tests, called runs, in which changes are made in the input variables in order to identify the reasons for changes in the output response. There are two popular experimental designs that generally applied which are the central composite design (CCD) and the Box-Behnken design (BBD). Both designs are built up from simple factorial or fractional factorial designs.

RSM had been extensively used by many researchers in a wide variety of fields for process optimization. However, there still lack of research in the study of RSM on the trimetallic catalyst in the transesterification reaction of biodiesel. Thus, in this study, RSM was applied in order to check the suitability of the technique to optimize the catalytic performance of trimetallic alumina supported catalyst reaction and the transesterification reaction condition was evaluated using BBD. The BBD was selected since it is an economical design with high efficiency and requires only three levels for each factor (the optimum conditions for each variable). It was used in RSM and validates the optimal conditions obtained from laboratory experiments.

1.4 Problem Statement

Biodiesel was one of the renewable energy resources that gave a better substitution from the usage of petro diesel. However, there were several challenges that have been noticed in the production of biodiesel like type of feedstock, catalyst used in the biodiesel synthesis and quality of biodiesel due to the leaching effect of the metal. Thus, this limitation leads to the research to be conducted in order to find the solution for the crisis that has been faced.

As mention earlier, previous research has synthesized the biodiesel using edible oil from vegetable and animal fats which leads to the competition with food consumption for human supply. Therefore, employing refined waste feedstock or non edible oil was become the main idea to overcome this crisis. Thus, in this study refined cooking oil (refined from the used cooking oil) was selected as feedstock to produce biodiesel in the presence of heterogeneous basic catalyst. Besides it can solve the competition with food supply, it also may lower down the production cost as compared from using edible oil.

Currently, many type of heterogeneous catalysts such as monometallic alkaline earth metal oxides supported on alumina have been reported to be catalyzing in transesterification reactions. However, the monometallic based metal oxides may cause leaching effect of metal into the reaction systems and reduce the quality of the biodiesel. Besides that, the basicity of the single oxide was lower and leads to the low biodiesel production. Thus, in order to solve these issues, the bimetallic and trimetallic supported catalyst was synthesize by using calcium (Ca) and magnesium (Mg) as based and incorporated with nickel (Ni), copper (Cu) and zinc (Zn). The modification of the catalyst may reduce the leaching effect due to the strong interaction between metal oxides and increase the degree of basicity for the catalyst that leads to the increasing of biodiesel production.

On the other hand, the usage of support in synthesizing the heterogeneous catalyst was an important for the catalyst to perform at the highest potential. In previous, zeolites present severe limitations when engage with the large molecules of reactant. It has a narrow and uniform micropore size distribution due to their crystallographically defined pore system (Taguchi and Schuth, 2005). In order to overcome the existing problem, the pursuit of solid base catalyst has been recently focused on mesoporous gamma alumina supported catalyst due to very high surface area, uniformity in pore size and high thermal stability which promise great opportunity for application as catalysts and catalytic supports. Thus in this study, series of alumina supported mixed metal oxide was synthesis and used in transesterification of refined cooking oil to produce high quality of biodiesel.

1.5 Objectives of the Study

The main goal of this research was to develop a new heterogeneous basic catalyst that can be used in transesterification reaction of refined cooking oil effectively at optimum conditions. The objectives of this research were:-

1. To synthesize the alumina supported calcium and magnesium oxides based catalysts for transesterification of refined cooking oil.
2. To screening and optimize the performance of prepared catalysts in transesterification reaction under normal reflux condition.
3. To characterize the catalysts in order to understand the chemical and physical properties of the catalysts.
4. To study the mechanistic reaction involve over potential catalysts and verified the biodiesel obtained according to American Standard Testing Material (ASTM) using potential catalyst

1.6 Scope of the Research

This research was focused on synthesis higher biodiesel production from refined cooking oil using alumina supported calcium (Ca) and magnesium (Mg) oxides based catalysts while, copper (Cu), nickel (Ni) and zinc (Zn) as dopants and co-dopants. The catalyst was prepared using nitrate salt via wetness impregnation method. After that, all the catalysts were screening in the transesterification reaction using refined cooking oil and methanol as reactant to produce methyl ester and glycerol and measured by gas chromatography-flame ionization detector (GC-FID) analysis. From the screening process, the highest potential catalyst and less potential catalyst was selected and were optimized according to the number of alumina coating, co-catalyst loading and calcination temperatures, reliability, reusability and regeneration testing. The optimum conditions over the two catalysts were validated by response surface methodology (RSM) via Box-Behnken design (BBD). It were then characterized by using various techniques in order to understand the physical properties of the catalysts such as X-ray diffraction (XRD) analysis for bulk structure, field emission scanning electron microscope-energy dispersive X-ray (FESEM-EDX) for morphology and elemental composition study, transmission electron microcopy (TEM) for particle size, nitrogen adsorption (NA) for pore texture and surface area of the catalyst. Meanwhile, thermal gravimetric analysis (TGA) was also performed to study the mass loss of the catalyst during temperature change while, the active surface components and reducibility of the catalysts were investigated using X-ray photoelectron spectroscopy

(XPS) and CO_2-temperature programmed desorption (CO_2-TPD). Furthermore, the mechanistic study occurred in product and on the surface of the catalyst was evaluated using GC-FID and FTIR-ATR. Lastly, the biodiesel produce using the best catalyst was selected and verified to study the properties of the biodiesel according to the American Standard Testing Material (ASTM).

1.7 Significance of Study

Biodiesel can be synthesized from edible and non edible oil of vegetable and animal fats. However, in order to solve the competition with human food supply this research was conducted using refined cooking oil as feedstock which were more easily obtained and more feasible to be employed in large scale production. Besides that, the catalyst prepared for this study was categorized as heterogeneous basic catalysts which were more stable, low in production cost and can be recycled. Thus this study was significant to be conducted and the novelties of this research study could be listed as follows:

1. The development of highly basic metal oxide catalysts from transition metal (Cu, Ni and Zn) and alkaline earth metal (Ca and Mg) as based catalysts.
2. The usage of refined cooking oil as feedstock in producing biodiesel
3. The postulated mechanism deduced from the most potential catalyst.

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