

**DEVELOPMENT OF HETEROGENEOUS BASE CATALYST FOR  
TRANSESTERIFICATION REACTION**

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## ABSTRACT

Among the many possible clean fuel sources, biodiesel derived from vegetable oil (VO) attracts attention as a promising one for substitution or blending with conventional diesel-based fuels. The current process utilizes alkali hydroxide dissolved in methanol for catalyzing the transesterification. However, the homogeneous catalyst gives a problem to environmental which would be leading the environmentally toxic. Therefore, the heterogeneous base catalyst should be developing to replace the homogeneous base catalyst as prevention. Here, the Calcium Oxide (CaO) loaded on Alumina ( $\text{CaO}/\text{Al}_2\text{O}_3$ ) is used as the heterogeneous base catalyst for transesterification of Palm Oil and Methanol. The  $\text{CaO}/\text{Al}_2\text{O}_3$  was prepared by using impregnation method and calcined at  $650^\circ\text{C}$  for 5 hours and characterized by Fourier Transfer Infrared Spectroscopy (FT-IR) and Scanning Electron Microscope (SEM). Both of equipment showed that Calcium Oxide is successfully supported by Alumina which 50% Calcium Oxide loading on Alumina is the best result. Then the catalyst was tested for the transesterification between palm oil and methanol. The transesterification reaction between palm oil and methanol is carried out at different parameter such as reaction temperature, mass ratio of catalyst to oil and molar ratio of methanol to oil. Then, the sample is analyzed using Gas Chromatography (GC). The result showed that the higher conversion of biodiesel is obtained by adding CaO catalyst of 3.5%, reaction temperature of  $65^\circ\text{C}$  and molar ratio of methanol to oil of 14:1. The higher total conversion of biodiesel is 48.82%. As a conclusion, the biodiesel conversion increased when there are increase in reaction temperature, amount of catalyst loading and molar ratio of methanol to oil.

## ABSTRAK

Antara sumber bahan bakar bersih, biodiesel yang diperbuat daripada minyak sayuran (VO) telah menjadi pilihan utama sebagai sumber bahan bakar bersih yang dicampurkan dengan diesel secara konvensional. Proses ini menggunakan alkali hidroksida yang dilarutkan di dalam metanol untuk pengtransesteran pemangkin tersebut. Namun begitu, pengguna pemangkin sekata akan memberi masalah kepada pencemaran alam sekitar. Oleh kerana itu, pemangkin tidak sekata alkali telah dicipta untuk menggantikan pemangkin sekata alkali sebagai langkah mengatasi masalah pencemaran alam sekitar. Di sini, Kalsium Oksida (CaO) yang dilarutkan di dalam Alumina ( $\text{CaO}/\text{Al}_2\text{O}_3$ ) telah digunakan sebagai pemangkin tidak sekata untuk pengtransesteran Minyak Sawit dan Metanol.  $\text{CaO}/\text{Al}_2\text{O}_3$  ini dihasilkan dengan menggunakan kaedah impregnasi dan dikalsinasi pada suhu  $650\text{ }^\circ\text{C}$  selama 5 jam. Penyifatan pemangkin tidak sekata alkali telah dikenal pasti dengan menggunakan Fourier Transfer Infrared Spectroscopy (FT-IR) dan Scanning Elektron Microscope (SEM). Kedua-dua peralatan ini telah membuktikan bahawa Kalsium Oksida berjaya dilarutkan di dalam Alumina dan dengan melarutkan sebanyak 50% Kalsium Oksida ke dalam Alumina merupakan hasil yang terbaik. Seterusnya pemangkin diuji untuk pengtransesteran antara minyak sawit dan metanol. Reaksi pengtransesteran antara minyak sawit dan metanol dilakukan pada parameter yang berbeza seperti suhu reaksi, nisbah masa pemangkin terhadap minyak dan nisbah molar metanol terhadap minyak. Kemudian, sampel dianalisis dengan menggunakan Gas Chromatography (GC). Keputusan kajian menunjukkan bahawa penghasilan biodiesel adalah lebih tinggi dengan menambah pemangkin CaO terhadap minyak sebanyak 3.5%, reaksi suhu  $65\text{ }^\circ\text{C}$  dan nisbah molar metanol terhadap minyak 14:1. Penghasilan keseluruhan biodiesel yang paling tinggi adalah 48.82%. Kesimpulannya, penghasilan biodiesel akan meningkat apabila suhu reaksi, jumlah nisbah larutan pemangkin terhadap minyak dan nisbah molar metanol terhadap minyak meningkat.

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**LIST OF SYMBOLS**

T	-	Temperature
°C	-	Degree Celsius
mg	-	Miligram
ml	-	Milimeter
wt/wt	-	Weight per weight
vol/wt	-	Volume per weight
%	-	Percentage
min	-	Minute
kJ	-	Kilo Joule

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## **CHAPTER 1.0**

### **INTRODUCTION**

#### **1.1 Background of Study**

In today's economy, fuel is a main concern. Today's society is totally dependent on fuel for transportation, heating, and other petroleum products, petrochemicals and plastics. The world economy also is dependent on petroleum in so many ways. Petroleum is non-renewable because it is taking millions of years for the earth to produce. At the rate the world is going now, it will only take a few more years for us to deplete it as well. This is actually a very serious issue. Many efforts have been underway to develop clean fuel in many countries like wind and solar energy. In order to resolve these problems, biodiesel is increasingly gaining international attention as a source of renewable energy.

Biodiesel fuel is beneficial to the environment because of having cleaner emissions compared to coal oil and conventional petroleum based fuel. The vegetable oil (VO) is renewable, nontoxic, and widely available from a variety of sources and has a lower sulphur contents close to zero. The

vegetable oils like palm oil (Malaysia), soybean oil (United States), sunflower oil (Europe), peanut oil, and olive oils are used as alternative fuel for diesel engines. Through a refinery process called transesterification, the reaction will remove the glycerine in the biodiesel fuel. Glycerine is a by-product that can be harmful to the engines. So as a result, biodiesel can be used in any normal petrodiesel engine, in pure form or in different blends.

There are more than 350 oil-bearing crops identified, researchers have been led in using different sources of vegetable oil such as peanut, rapeseed, corn, soybean or sunflower seed because they are considered as potential alternative fuels for diesel engines. The use of vegetable oils as alternative renewable fuel also competing with petroleum was proposed in the beginning of 1980s. The advantages of vegetable oils as diesel fuel are:

1. Liquid nature – portability
2. Ready availability
3. Renewability
4. Higher heat content ( about 88% of no. 2 diesel fuel)
5. Lower sulphur content
6. Lower aromatic content
7. Biodegradability

## **1.2 History of Biodiesel**

Biodiesel is a renewable substitute fuel for petroleum diesel made from vegetable or animal fats via transesterification of alcohols. Transesterification of triglycerides in oils is not a new process. Scientists E. Duffy and J. Patrick conducted it as early as 1853. Life for the diesel engine began in 1893 when the famous German inventor Rudolph Diesel published a paper entitled ‘The theory and

construction of a rational heat engine'. What the paper described was a revolutionary engine in which air would be compressed by a piston to a very high pressure thereby causing a high temperature. Dr. Rudolph Diesel designed the original diesel engine to run on vegetable oil. Dr. Rudolph Diesel used peanut oils to fuel on of this his engines at the Paris Exposition of 1900 (Nitschke, 1965). Because of high temperature created, the engine was able to run a variety of vegetable oils including hemp and peanut oil. At the 1911 World's Fair in Paris, Dr. Rudolph Diesel ran his engine on peanut oils and declared 'the diesel engine can be fed with vegetable oils and will help considerably in the development of the agriculture of the countries which use it. One of the first uses of transesterified vegetable oil was powering heavy-duty vehicles in South Africa before World War II. The name 'biodiesel' has been given to transesterified vegetable oil to describe its use as a diesel fuel (Demirbas, 2002).

### **1.3 Technical Properties of Biodiesel**

Biodiesel is a fuel comprised of monoalkyl esters of long chain fatty acids derived from vegetable oils or animal fats, designated B100, and meeting the requirements of the American Society for Testing and Materials (ASTM) D 6751. It is a clean burning fuel, which is nontoxic, biodegradable, and considered as the fuel of the future. It can be used neat or mixed with petroleum diesel to produce a biodiesel blend that can be used in compression ignition engines under a variety of operating conditions. Pure biodiesel fuel contains no petroleum fuels and emits virtually no sulphur, aromatics, particulates, or carcinogenic compounds and is thus a safer alternative to petroleum diesel (Singh et al., 2007). Table 1.1 shows the fuel ASTM standards of biodiesel (Demirbas, 2009).

**Table 1.1:** ASTM standard of biodiesel

Properties	Test Method	Biodiesel
Standard number	-	ASTM D6751
Specific gravity (g/ml)	-	0.88
Flash point (K)	D 93	403 K
Water and sediment	D 2709	0.05 max % volume
Kinematic viscosity (at 313 K)	D 445	1.9 – 6.0 mm <sup>2</sup> /s
Sulfated ash	D 874	0.02 max % wt
Sulfur	D 5453	0.05 max % wt
Copper strip corrosion	D 130	No. 3 max
Cetane number	D 613	47 min
Carbon residue	D 4530	0.05 max % mass
Cloud point (K)	D 2500	Report
Acid number	D 664	0.80 max wt%
Free glycerine	D 6584	0.02 wt%
Total glycerine	D 6584	0.24 wt%
Phosphorus	D 4951	0.001 wt%
Vacuum distillation end point	D 1160	360°C max, at 90% distilled



#### **1.4 Raw Material Use in Biodiesel.**

Biodiesel can be produced from any types of vegetable oil or animal fats. Since edible oils are readily available feedstock in huge quantities, they can be the immediate feedstock for biodiesel production. Thus edible oils such as rapeseed, soybean and palm oil are potential feedstock for biodiesel production.

Vegetable oils have become more attractive than animal fats recently because of its environmental benefits and the fact that it is made from renewable resources. Vegetable oils are a renewable and potentially inexhaustible source of energy with an energetic content close to diesel fuel.

Several types of vegetable oils can be used for the preparation of biodiesel. For examples soybean, rapeseed, sunflower and palm oils are the most studied. Table 1.2 shows the oil species that can be used in biodiesel production (Demirbas, 2009).

**Table 1.2:** Oil species for biodiesel production

Group	Source of Oils
Major Oils	Coconut (copra), corn (maize), cottonseed, canola (a variety of rapeseed), olive, peanut (groundnut), safflower, sesame, soybean, and sunflower.
Nut Oils	Almond, cashew, hazelnut, macadamia, pecan, pistachio and walnut.
Other Edible Oils	Amaranth, apricot, argan, artichoke, avocado, babassu, baylaurel, beechnut, ben, Borneotallownut, carobpod(algaroba), cohune, coriander seed, false flax, grape seed, hemp, kapok seed, lallemantia, lemon seed, macauba fruit( <i>Acrocomia sclerocarpa</i> ), meadow foam seed, mustard, okra seed(hibiscus seed), perilla seed, pequi ( <i>Caryocar brasiliensis</i> seed), pinenut, poppy seed, prune kernel, quinoa, ramtil( <i>Guizotia abyssinica</i> seed or Niger pea), rice bran, tallow, tea(camellia), thistle( <i>Silybum marianum</i> seed), and wheat germ.
Inedible Oils	Algae, babassu tree, copaiba, honge, jatropha or ratanjyote, jojoba, karanja or honge, mahua, milk bush, nagchampa, neem, petroleum nut, rubber seed tree, silk cotton tree, and tall.
Other oils	Castor and radish

Natural vegetable oils and animal fats are solvent extracted or mechanically pressed to obtain crude oil or fat. These usually contain free fatty acids, phospholipids, sterols, water, odorants and other impurities. The free fatty acid and water contents have significant effects on the transesterification of glycerides with alcohols using alkaline or acid catalysts. They also interfere with the separation of fatty acid alkyl esters and glycerol because of salt formation in the product. The fatty

acid compositions of vegetable oil samples are given in the Table 1.3 (Demirbas, 2005).

**Table 1.3:** Fatty acid compositions of vegetable oil samples.

Sample	16:0	16:1	18:0	18:1	18:2	18:3	Others
Cottonseed	28.7	0	0.9	13.0	57.4	0	0
Poppyseed	12.6	0.1	4.0	22.3	60.2	0.5	0
Rapeseed	3.5	0	0.9	64.1	22.3	8.2	0
Safflowerseed	7.3	0	1.9	13.6	77.2	0	0
Sunflowerseed	6.4	0.1	2.9	17.7	72.9	0	0
Sesameseed	13.1	0	3.9	52.8	30.2	0	0
Linseed	5.1	0.3	2.5	18.9	18.1	55.1	0
Wheat grain	20.6	1.0	1.1	16.6	56.0	2.9	1.8
Palm	42.6	0.3	4.4	40.5	10.1	0.2	1.1
Corn marrow	11.8	0	2.0	24.8	61.3	0	0.3
Castor	1.1	0	3.1	4.9	1.3	0	89.6
Tallow	23.3	0.1	19.3	42.4	2.9	0.9	2.9
Soybean	13.9	0.3	2.1	23.2	56.2	4.3	0
Bay laurel leaf	25.9	0.3	3.1	10.8	11.3	17.6	31.0
Peanut kernel	11.4	0	2.4	48.3	32.0	0.9	4.0
Hazelnut kernel	4.9	0.2	2.6	83.6	8.5	0.2	0
Walnut kernel	7.2	0.2	1.9	18.5	56.0	16.2	0
Almond kernel	6.5	0.5	1.4	70.7	20.0	0	0.9
Olive kernel	5.0	0.3	1.6	74.7	17.6	0	0.8
Coconut	7.8	0.1	3.0	4.4	0.8	0	65.7

## 1.5 Process of Biodiesel

The direct use of vegetable oils in fuel engine is problematic such as carbon deposits, coking and trumpet formation on the injectors, oil ring sticking and others (Hanna, 1999). Due to their high viscosity (about 11 – 17 times higher than diesel fuel) and low volatility, they do not burn completely and would be form deposits in the fuel injector of diesel engines (Demirbas, 2003). So that, there are four different ways have been considered to reduce the high viscosity of vegetable oils. For example:

1. Dilution of 25 parts of vegetable oil with 75 parts of diesel fuel.
2. Microemulsifications with short chain alcohols such as methanol or ethanol.
3. Catalytic cracking, which produces alkanes, cycloalkanes and alkylbenzenes
4. Transesterificaation with methanol or ethanol.

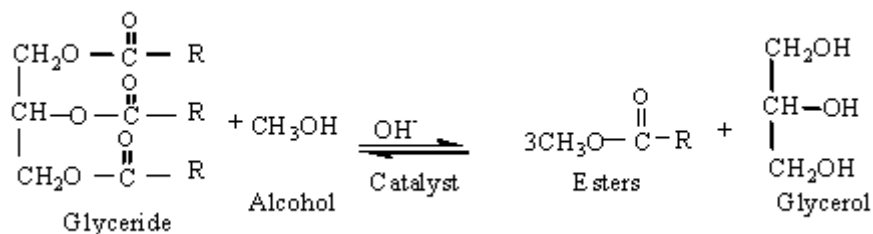
Dilution of oils with solvents and microemulsions of vegetable oils will produce the lowers viscosity. However, some engine performance problems such as injector coking and more carbon deposits still exist. Therefore, among these entire alternative, the transesterification seems to be the best choice, as the physical characteristics of biodiesel are much closed to those of diesel fuel and the process is relatively simple.

### 1.5.1 Transesterification of Biodiesel

Transesterification is the process of using an alcohol such as methanol, ethanol propanol and butanol in the presence of catalyst to chemically break the molecule of raw renewable oil into methyl or ethyl esters of the renewable oil with glycerol as by product (Demirbas, 2003). Methanol is commonly used alcohol in this process due in part to its low cost. However, ethanol is a preffered alcohol in the transesterification

process compared to the methanol because it is derived from agricultural products and is renewable and biologically less objectionable in the environment. Alkali or base catalyzed transesterification has been most frequently used industry, mainly due to its fast reaction rate and economic.

Besides that, the process to produce the biodiesel fuel is involves using a catalyst and an alcohol as a solvent. The current method of producing biodiesel is the base catalyzed transesterification of vegetable oil. The transesterification is an equilibrium reaction, and the transformation occurs essentially by mixing the reactants. However, the presence of a catalyst (acid or base) could accelerate and control the equilibrium, to achieve the high yield of the ester. However, the alcohol has to be used in excess. The overall process is a sequence of three consecutive and reversible reactions, in which mono-, di- and triglycerides are formed as intermediates. The stoichiometric reaction requires 1 mol of triglyceride reacts with 3 mol of alcohol (primarily methanol) in the presence of a strong catalyst (acid, base, or enzymatic), producing a mixture of fatty acid alkyl esters (biodiesel) and glycerol like in figure 1.1 (Singh et al., 2008). Because the transesterification is a reversible reaction, a stoichiometric ratio of 6:1 is practically used to increase the concentration of the product (biodiesel) and to precede the equilibrium toward the product side (Schuchardta et al., 1998).



**Figure 1.1:** Transesterification of triglyceride and oil.

By this method, alcohol and the catalyst are mixed together where the catalyst will dissolved in the alcohol. The vegetable oil or fat is then will add into the mixture of alcohol and catalyst. After that just wait for them components to react with each other. There are times that the mix is kept just above the boiling point of the alcohol to help speed up the reaction.

Nowadays, most industrial applications are performed in batch or continuous stirred tank reactors at temperatures ranging from 60°C to 200°C using homogeneous basic catalyst. Basic catalysts are usually preferred than acid catalyst in industry because of the higher activity and the lower process temperatures required. The most commonly used base catalyst for biodiesel production is sodium hydroxide or potassium hydroxide. However, the use of homogeneous base catalysts requires neutralization and difficult separation process to separate from the final reaction mixture. So that, it will leading to a series of environmental problems related to the use of high amounts of solvent and energy.

In the other hand, heterogeneous base catalysts are in solid condition. Therefore, they could be easily separated from the reaction mixture by simple filtration without using the solvent. Besides that, they are easily generated and have less corrosive character. It is also leading to safer, cheaper and more environmental friendly operation. Consequences, it is one of interest to investigate the possibility of replacing the homogeneous base catalysts by solid base catalysts in transesterification reaction.

## **1.6 Catalysis in Transesterification**

The transesterification reaction can be catalyzed by acids, bases or enzymes. Various studies have been carried out using different vegetable oils as raw material, different alcohol, as well as different catalysts including homogeneous and heterogeneous. Table 1.4 show the advantages and disadvantages of different types of catalyst (Lam et al., 2010).

**Table 1.4:** Advantages and disadvantages of different types of catalyst.

Type of catalyst	Advantages	Disadvantages
Homogeneous base catalyst	<ul style="list-style-type: none"> <li>• Very fast reaction rate – 4000 times faster than acid catalyzed transesterification</li> <li>• Reaction can occur at mild reaction condition and less energy intensive</li> <li>• Catalysts such as NaOH and KOH are relatively cheap and widely available</li> </ul>	<ul style="list-style-type: none"> <li>• Sensitive to Free Fatty Acid (FFA) content in the oil</li> <li>• Soap will formed if the FFA content in the oil is more than 2 wt%</li> <li>• Too much soap formation will decrease the biodiesel yield and cause problem during product purification especially generating huge amount of wastewater</li> <li>• Separation of catalyst from product is problematic</li> <li>• Cannot regenerated</li> </ul>



<p>Heterogeneous base catalyst</p>	<ul style="list-style-type: none"> <li>• Relatively faster reaction rate than acid catalyzed transesterification</li> <li>• Reaction can occur at mild reaction condition and less energy intensive</li> <li>• Easy separation of catalyst from product</li> <li>• High possibility to reuse and regenerate the catalyst</li> <li>• Environmental friendly process</li> <li>• Less corrosion occur on reactor and pipelines</li> </ul>	<ul style="list-style-type: none"> <li>• Poisoning of the catalyst when exposed to ambient air</li> <li>• Sensitive to Free fatty Acid (FFA) content in the oil due to its basicity properties</li> <li>• Soap will formed if the FFA content in the oil is more than 2 wt%</li> <li>• Too much soap formation will decrease the biodiesel yield and cause problem during product purification</li> <li>• Leaching of catalyst active sites may result to product contamination</li> </ul>
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<p>Homogeneous acid catalyst</p>	<ul style="list-style-type: none"><li>• Intensive to Free Fatty Acid (FFA) and water content in the oil</li><li>• Preferred method if low grade oil is used</li><li>• Esterification and transesterification occur simultaneously</li><li>• Reaction can occur at mild reaction condition and less energy intensive</li></ul>	<ul style="list-style-type: none"><li>• Corrosive catalyst such as <math>H_2SO_4</math> used can lead to corrosion on reactor and pipelines</li><li>• Separation of catalyst from product is problematic</li><li>• Cannot be regenerated</li><li>• Complicated catalyst synthesis procedures lead to higher cost</li><li>• Normally, high reaction temperature, high alcohol to oil molar ratio and long reaction time are required</li></ul>
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