

Biodiesel Production from Palm Oil using Heterogeneous Base Catalyst

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ABSTRACT

In this study, the transesterification of Soya bean oil with methanol for biodiesel production was studied by using CaO–MgO as a heterogeneous base catalyst prepared by incipient wetness impregnation (IWI) and co-precipitation (CP) methods. The biodiesel produced was also characterized, the result was compared using American Society for Testing and Materials (ASTM).

Keywords : Biodiesel, Catalyst, Transesterification, Kinematic viscosity, Flash point, Smoke point, Fire point, Acid value, Density, Specific gravity

I. INTRODUCTION

In the recent years, the world's energy demand, derived from petroleum, mineral coal, natural gas, is increasing at an exponential rate. The main cause of the fast diminishing of energy resources is due to the rapidly increasing of population and a growth of industrialization. In order to solve this problem, researchers are seeking new alternative energy sources to replace the fossil fuel. Therefore, biomass is consequently gaining international attention as a source of renewable energy. The scarcity of conventional fossil fuels, growing emissions of combustion-generated pollutants and their increasing costs will make biomass sources more attractive. Biodiesel is becoming more attractive recently because of its environmental advantages and the fact that it is made from renewable resources. The finite nature of fossil fuels necessitates consideration of alternative fuels from renewable sources. Biodiesel is a notable alternative to the widely used petroleum-derived diesel fuel since it can be generated by domestic natural sources such as soybeans palm, coconuts, and

even recycled cooking oil and thus reduces dependence on diminishing petroleum fuel. Engine performance and fuel consumption were favorable making it a better substitute diesel.

1.1 What is biodiesel?

Biodiesel, a mixture of fatty acid methyl esters (FAMES), is a renewable clean bio-fuel has been developed as one of the most promising alternative fuel for fossil fuel regarding to the limited resources of fossil fuels and the environmental concerns. Biodiesel is a renewable and biodegradable source of energy derived from various biological sources which can be used in unmodified diesel engines. Biodiesel releases less harmful emission into the atmosphere than petrodiesel. Biodiesel when used as pure fuel is known as B100. Biodiesel is a fuel that can be blended easily with fossil diesel fuel and offer several advantages. The blend is designated "BXX" where XX is the percentage of biodiesel in the blend. It has higher biodegradability, low CO, SO_x, NO_x, and particulate matter (PM) contents, renewability, and lack of

aromatic compounds as compared to the conventional diesel fuels. On the other hand, biodiesel can be used in diesel engines without engine modification because its characteristics are similar to those of petroleum-based diesel fuels. Biodiesel is generally more expensive than petro-diesel. However, the cost of biodiesel is expected to drop due to technological advancement, economies of scale, rising cost of crude oil, etc.

In 1895, Dr. Rudolf Diesel invented the diesel engine with the intention of running it on a variety of fuels, including vegetable oil. In fact, when he demonstrated his engine at the World Exhibition in Paris in 1900, he used peanut oil as fuel. However, biodiesel and vegetable oil are very different. Raw Soya bean oil or recycled greases that have not been processed into esters are not biodiesel. Neat Soya bean oil has high viscosity and tendency to polymerize during storage and combustion. All these lead to fuel filter clogging, ring sticking, gum formation, accumulation of oil in the lubricating system, sludge formation, poor atomization in spray system, incomplete combustion and injector choking. Technical aspects of biodiesel are approached; such as the physical and chemical characteristics of methyl ester related to its performance in compression ignition engines. Vegetable oils are triglycerides and highly viscous. Soya bean oils consist of triglyceride molecules of three long chain fatty acids that are bonded to a single glycerol molecule. The fatty acids differ by the length of carbon chains, the number, and orientation of double bonds in the chains. In principle, vegetable oils can replace petro-diesel; however, vegetable oils have high viscosities and variable acid composition which create problems in diesel engines. There are at least four ways in which oils and fats can be converted to biodiesel namely: pyrolysis, thermal cracking, dilution and transesterification.

The production of biodiesel is performed by transesterification reaction of triglycerides using alcohol in the presence of a catalyst. However,

transesterification is the best method for producing higher quality biodiesel.

Transesterification or alcoholysis, is a reaction when a fat or oil reacts with an alcohol by using a catalyst to form esters and glycerol. Many types of alcohol can be used for such as methanol, ethanol, propanol, and butanol. The most common used is methanol because it gives a proper viscosity and boiling point and a high cetane number. In the transesterification of vegetable oils, triglyceride reacts with methanol producing glycerol and a mixture of fatty acid methyl esters (biodiesel).

1.2 Types of catalyst used in biodiesel production

The main catalysts used can be classified, according to their chemical presence in the transesterification reaction, as homogeneous or heterogeneous catalysts. Homogeneous catalysts act in the same liquid phase as the reaction mixture, whereas heterogeneous catalysts act in a different phase from the reaction mixture, usually as a solid. Heterogeneous catalysts are noncorrosive, a green process and environmentally friendly. They can be recycled and used several times, thus offering a more economic pathway for biodiesel production. The preferred alcohol for the production of biodiesel due to its low cost and industrial availability (Leung et al., 2010).

In the transesterification process, biodiesel is usually prepared in the presence of homogeneous base or acid catalysts. The acid-catalyzed process often uses hydrochloric acid or sulfuric acid as a catalyst; however, a high molar ratio of methanol to oil is needed, and the reaction time is very long. So the base catalysts are preferred to be used instead of acid catalyst because the catalytic activity of a base is higher than that of an acid and acid catalysts are more corrosive.

The homogeneous process leads to higher yields, especially if the content of free fatty acids (FFAs) in the used oil is less than 1%, when compared to the heterogeneous catalytic transesterification (Karmakar

et al., 2010). Nevertheless, the homogeneous process presents some disadvantages in its application, such as expensive separation of the homogeneous catalyst from the reaction mixture; generation of large amounts of wastewater during the separation and purification of the product and formation of soap due to the reaction of the alcohol with the FFA's present in the reaction medium (Sharma and Singh, 2010). Therefore, advantages like product separation, the most favorable reuse of the catalyst and reaction conditions, inherent in heterogeneous catalytic transesterification process, have led to the development of different heterogeneous solid catalyst (Borges and Díaz, 2012).

Metal oxides represent a group of heterogeneous basic catalysts most widely studied in the literature. Among them there are the calcium oxide, magnesium oxide, strontium oxide, mixed oxides and hydrotalcite (Borges and Díaz, 2012). Heterogeneous catalysts can be separated from final product by filtration and reused. This causes less consumption of both chemicals and time of course. Heterogeneous catalysts are noncorrosive. They have a high selectivity and can be easily separated from the products. Both basic solids such as metal oxides (CaO) and zeolites as well as acid solid such as sulphated tin oxide are used. The use of catalyst supports such as alumina, silica, zirconia and zinc oxide in order to improve the mass transfer limitation of the three phase reaction will be included. Examples of heterogeneous catalyst are; La₂O₃, SrO, BaO, KNO₃, KF, CaO, CaCO₃.

Many features of acidic heterogeneous catalyst are suitable for biodiesel production, for instance insensitivity for water content and free fatty acid content. Heterogeneous catalyst can be used in batch or continuous system. Acid catalysts are more expensive than alkali heterogeneous catalysts. They have also less active site, therefore, they are more affected by adsorption reactants rate, surface reaction rate, desorption product rate resulting in limiting biodiesel yield. Currently, among the alkaline earth

metal oxides, CaO is the most widely used for transesterification with high yield (98% during the first cycle of reaction).

Homogeneous catalysed transesterifications of vegetable oils and animal fats cause major drawbacks such as instrument corrosiveness, difficulty in catalyst separation, and health hazards to the operators due to the generation of massive waste stream from washing steps³. In contrast the transesterification reaction in the presence of heterogeneous catalyst generate products easier to separate and to purify, making the production process more economical and causing less environmental damage. Calcium compounds, although of lower basic strength than Sr and Ba are less toxic, widely available, cheaper, ease to handle, making them promising catalyst and/or catalyst support in the biodiesel industry. The catalytic activity of CaO can be accelerated with an initial pretreatment with methanol at room temperature activation at high temperature⁴, and/or Sites.

1.3 Why heterogeneous catalyst?

However, in this conventional homogeneous method, the removal of these catalysts is very difficult, and a large amount of wastewater is produced to separate and clean the catalyst and the products. Therefore, conventional homogeneous catalysts are expected to be replaced by environmental friendly heterogeneous catalysts. The replacement of the homogeneous catalyst by the heterogeneous catalysts would have various advantages such as the easy catalyst separation from the reaction mixture, product purification, and the reduction of environmental pollutants.

In this work, transesterification was carried out using CaO-MgO as heterogeneous basic catalyst. The effect of reaction parameters, such as molar ratio of methanol to oil, reaction time, amount of catalyst, and reaction temperature, were optimized for the production of biodiesel.

II. METHODS AND MATERIAL

2.1 Materials

Soy bean oil was bought from shoprite mall in Akure, analytical grade methanol was purchased from Pascal chemical store, and the set of beakers, conical flasks, measuring cylinders, standard flask, PH meter, thermometer, burette, oven, tripod, and magnetic stirrer and separating funnel was gotten from the chemistry laboratory while the muffle furnace was gotten from the food science and technology laboratory of the Federal University of Technology, Akure.

2.2 Methodology

The production of the heterogeneous catalyst (CaO-MgO) and production and characterization of biodiesel was carried out in the chemistry department of the Federal University of Technology, Akure.

2.2.1 Equipment used

1. Beakers
2. Conical flask
3. PH meter
4. Measuring cylinder
5. Pipette
6. Tripod stand
7. Magnetic stirrer
8. Standard flask
9. Thermometer
10. Separating funnel
11. Three necked flask
12. Condenser
13. Petri dish

2.2.2 Reagent used

1. Methanol
2. Calcium Nitrate Tetrahydrate
3. Magnesium Nitrate hexahydrate
4. Phenolphthalein

2.3 Acid value determination

The acid value was determined by directly titrating the oil in an alcoholic medium with aqueous NaOH solution. Free fatty acid was calculated as oleic acid.

Reagents: Ethyl alcohol (95%) by volume and neutral solvent. Phenolphthalein indicator, 100ml of di-ethyl ether, 100ml methanol, standard aqueous 0.1M NaOH solution.

Procedure: About 1g of sample oil was weighed in a conical flask. 25ml of ethyl alcohol and indicator, 100ml of di-ethyl ether, 100ml methanol, standard aqueous 0.1M NaOH solution.

Calculations:

Acid Value = $\text{Titre Value} \times \text{Molarity of KOH} \times 56.1 / \text{Weight of the sample}$

2.4 Determination of free fatty acid (FFA)

1. 0.65g of KOH was dissolved in 100ml of distilled water.
2. The mixture was poured into a 25ml burette.
3. 0.65g of Soya bean oil was measured into a conical flask.
4. 25ml each of di-ethyl ether and ethanol were measured and poured into the beaker of oil.
5. Two drops of phenolphthalein was placed into the mixture.
6. KOH was titrated against it.
7. The titre value was noted to be 0.8 on the burette.
8. The acid value was calculated to give 1.2.
9. The free fatty acid value was calculated to give 0.6%

Calculation:

Acid Value = $\text{Titre value on Burette} \times \text{Molarity of KOH} \times \text{Molar weight of KOH} / \text{Weight of sample}$

Free Fatty Acid = $\text{Acid value} / 2$

2.5 Preparation of CaO-MgO Catalyst by Incipient-Wetness Impregnation (IWI) method

1. 20g of Magnesium oxide is dried in an oven at 110 C for 2h to remove the absorbed water on^o the surface.
2. This unmodified MgO was stored in a silica gel desiccator prior to use.
3. To prepare modified MgO with different Ca loadings, the MgO is impregnated with anaqueous solution of 20g of Ca(NO₃)₂.4H₂O.
4. Samples with various Ca loadings, given in weight percentage, were impregnated for 24h to ensure that the Ca diffused and dispersed thoroughly on the surface of ZnO.
5. The loading amounts of Ca were calculated on the basis of the amounts of the starting materials.
6. The pretreated samples are dried in an oven at 110 C for overnight and then calcined at^o different temperatures before being used for the reaction.

2.6 Preparation of CaO-MgO catalyst by Co-Precipitation(CP) method

1. 40g of Calcium nitrate tetrahydrate (Ca(NO₃)₂.4H₂O) was weighed
2. 40g of Magnesium nitrate hexahydrate (Mg(NO₃)₂.6H₂O) was weighed
3. Both compounds were mixed and then dissolved in 100cl distilled water in a beaker
4. The mixture was placed on a magnetic stirrer and under continuous stirring and heating at 80°C.
5. 0.1M Na₂CO₃ was prepared by using this method first the moles of sodium carbonate in the given solution.

$$\text{Moles} = \text{concentration} \times \text{volume} \quad (n = cv)$$

$$= 0.1 \text{ mol/dm}^3 \times (250/1000) \text{ dm}^3$$

$$(0.1\text{M} = 0.1\text{mol/dm}^3 \text{ and converting } 250\text{ml} \text{ into } \text{dm}^3 \text{ by dividing by } 1000) = 0.025$$

$$\text{Next find out the molecular mass of Na}_2\text{CO}_3 = (23 \times 2) + 12 + (16 \times 3) = 106(\text{g/mol})$$

Then find out the mass of Na₂CO₃ needed to give 0.025 mol (the amount of moles in the solution)

$$\text{Moles} = \text{mass} / \text{molar mass}$$

$$\text{Therefore, mass} = \text{moles} \times \text{molar mass}$$

$$= 0.025 \times 106$$

$$= 2.65\text{g}$$

6. Pour the 0.1M Na₂CO₃ into the mixture of the Calcium nitrate tetrahydrate and Magnesium nitrate hexahydrate to adjust the PH to 8. The PH meter will be inserted into the mixture to measure the PH
7. The mixture was then aged for 1hr (12:20pm – 1:20pm).
8. The mixture was then washed with warm distilled water.
9. The precipitate was dried in the oven at 110°C overnight.
10. The precipitate was then calcined in the muffled furnace for about 3hrs at temperature ranging from 560°C to 600 °C

Calcination is defined as heating to high temperature (550°C-1150°C, or 1000 – 2100 F) in air or oxygen to bring about a thermal decomposition.

11. After calcination, the sample was stored in silica gel desiccator prior to use



Figure 1 : The mixture (Calcium nitrate tetrahydrate and Magnesium nitrate hexahydrate) was placed on a magnetic stirrer and under continuous stirring and heating at 80 C^o



Figure 2: standard flask to prepare 0.1M of sodium carbonate (Na_2CO_3)



Figure 3: mixture of calcium nitrate tetrahydrate and magnesium nitrate hexahydrate



Figure 4: PH meter for adjusting the PH of the mixture to 8



Figure 5: The precipitate stored in the oven at temperature 110 C overnight



Figure 6: The precipitate calcined in the muffle furnace at temperature at temperature ranging from 550-600 C°



Figure 7: Storing the catalyst in the desiccator prior to use.

2.7 Transesterification of Soya bean oil

1. 40ml of the Soya bean oil and 100ml of the Methanol were measured
2. 0.9grams of the Heterogeneous catalyst was weighed
3. Then, the catalyst, Soya bean oil and methanol were added to a three-necked flask
4. A magnetic stirrer was used for mixing oil, methanol, and catalyst.
5. The mixture was heated to 60 C in a 500ml three-necked flask°
6. The reaction was carried out for 2 hours.
7. After that the reaction was stopped by cooling down the reactor to room temperature.
8. The catalyst was separated out from the product by using a suction flask. the products were placed in a separating funnel overnight to ensure the phase of glycerol are separated completely.

9. The 25wt% of sodium sulphate based on weight of methyl ester product was added into methyl esters to remove water.



Figure 8 : Transesterification of soya bean oil



Figure 9 : glycerol (lower region) and biodiesel (upper region)



Figure 10: water (lower region) and biodiesel (upper region)

2.7.1 Determination of smoke point

A petri dish was filled with 20ml of the sample oil and this was heated continuously on a hot plate until the sample gave off a thin fume with continuous streams of bluish flame. A thermometer was clamped on the retort-stand, the sensitive part of thermometer was dipped into the petri dish to record the temperature at this point.

2.7.2 Determination of flash point

The same oil sample was used and with the aid of a clamp holder, a thermometer was hung and dipped inside the oil, ensuring that the thermometer does not touch the bottom of the petri dish which is placed on a hot plate. The sample was heated until a sufficient vapor is produced when flame was applied, it causes burning for more than one minute. The temperature is then recorded.

2.7.3 Determination of fire point

The sample that was used for the flash point was used with the heating continued until a sufficient vapor was produced and the flame was applied, it caused burning for a period of more than one minute. The temperature was then recorded.



Figure 11: Set-up for determining smoke, flash, and fire points

2.7.4 Determination of Specific Gravity

Calculation:

Specific gravity = Weight of oil/Weight of equal volume water



Figure 12: Specific Gravity Bottle with Oil



Figure 13: Measurement of SGB weight

2.7.5 Determination of viscosity

A clean and dry viscometer with a flow time above 200seconds was selected for the oil sample. The viscometer was charged with the sample by inverting the tube's thinner arm into the liquid sample and suction force was drawn up to the time mark of the viscometer, after which the instrument was turned to its normal vertical position. The viscometer was placed into holder and inserted to a constant temperature water bath set at 25°C. The suction force was then applied to the thinner arm to draw the sample slightly above the upper timing mark. The efflux time was recorded by timing the flow of the sample as it flowed freely from the upper timing mark to the timing mark to the lower timing mark.

Calculation:

$$\text{Viscosity} = \text{Flow time} \times \text{specific gravity} \times 1.002 / \text{Flowrate of water}$$

$$v = \eta / \rho$$

Where, v is the kinematic viscosity [g/cm, s]



Figure 14: On-going viscosity test

III.RESULTS AND DISCUSSION

3.1 Characterization of Soya Bean Oil

The properties, such as the specific gravity, kinematic viscosity, free fatty acid content, moisture content, saponification value, ester value, iodine value, odour, colour was measured by the methods was mentioned below and presented in Table 4.1.

TABLE 1. RAW MATERIAL (SOYA BEAN OIL) SPECIFICATION

Properties	Unit	Soya bean oil	Biodiesel using heterogeneous catalyst
Density	g/ml ³	0.917	0.87
Specific gravity		0.9064	0.85
Viscosity	mm ² /s	52	5.4
Fire point	°C	360	144
Flash point	°C	330	135
Acid value	°C	2.6	1.0
Free fatty acid	%FFA	1.2	0.5
Smoke point	°C	230	102

The results for properties of soya bean was obtained from <https://en.wikipedia.org/wiki/Soybean>

TABLE 2. PROPERTIES OF BIODIESEL PRODUCED

Properties	Unit	Biodiesel using homogeneous catalyst NaOH	Biodiesel using heterogeneous catalyst	ASTM Standard
Density	g/ml ₃	0.877	0.87	
Specific gravity	At 25°C	0.877	0.85	0.88(at 15.5°C)
Viscosity	At 40°C	5.30	5.4	1.9-6.0
Fire point	°C	142	144	--
Smoke Point	°C			
Flash point	°C	132	153	100-170
Acid value		0.3		(0.8max)
Free fatty acid	%FFA	0.15	0.5	---

The results for Biodiesel using NaOH as catalyst are gotten from Nkem Ebo et al (2017). Preparation and characterization of biodiesel (Fatty Acid Methyl Ester) from a commercial (Soya bean) oil. International Journal of Research and Review. 2017; 4(5):65-71
Table 4.2: Properties of biodiesel produced from soya bean oil in comparison with the standard value.

3.2.1 Density

It can be defined as mass of an object divided by its volume. it is an important physical property of liquid fuel and biodiesel. A slight change in density can affect engine output power. the result from this work shows a density of 0.87g/ml.

3.2.1 Specific gravity

Specific gravity is defined as the ratio of weight of a substance and weight of equal volume of water. Specific gravity of fuel is very important in diesel

engine because fuel injection operates on a volume metering basis. Specific gravity of Biodiesel varies with the fatty acid composition and the quality of the diesel fuel. A denser biodiesel has higher energy content and results in better mileage and increased power. Specific gravity is used as a precursor for a number of fuel properties such as heating value, viscosity and cetane number the result from this work shows a specific gravity of 0.85 of which is in quite in agreement with the ASTM norms

3.2.2 Kinematic viscosity

Kinematic Viscosity is a measure of resistance of fluid flow under the influence of gravity. Viscosity is a measure of the internal friction or resistance of oil to flow. As the temperature of oil is increased, its viscosity decreases and it is therefore able to flow more readily. Viscosity is the most important of biodiesel properties since it affects the operation of fuel injection equipment, particularly at low temperatures when the increase in viscosity affects the fluidity of the fuel. The result from this work shows a kinematic viscosity at 40 °C of 5.4mm²/sec which is in quite in agreement with the ASTM norms. viscosity of a fuel is related to fuel lubricity. Low viscosity fuels are more likely to provide unsatisfactory lubrication in fuel injection pump: these often lead to increase in wear. High viscosity of fuel are responsible of atomization of fuel, incomplete combustion and increased exhaust emissions, choking of injectors, thereby forming larger droplets on injector, ring carbonization and accumulations of fuel in the engine.

3.2.4 Flash point

Flash point is the minimum temperature at which a fuel must be heated for it to ignite air -vapour mixture. The U. S Department of Transportation specified 90 0C as the flashpoint for nonhazardous fuel. The flash point for this work is 135 0C. This result shows appreciable consistency with both ASTM, standard for biodiesel and works of other researchers. The high value obtained in this study clearly signifies that the

biodiesel produced is basically free from methanol; this is because even small quantity of methanol can reduce the flash point reasonably and also negatively affects diesel engine parts such as fuel pumps, seals and elastomers.

3.2.5 Smoke point

Smoke point which is also known as burning point is the temperature at which, under specific and defined conditions, it begins to produce a continuous bluish smoke that becomes clearly visible. The more the Free Fatty Acid (FFA) content the quicker it will break down and start smoking. A high smoke point indicates a fuel of low smoke producing tendency.

4.2.6 Fire point

The fire point of a biodiesel is the lowest temperature at which the vapour will continue to burn for at least five seconds after ignition by an open flame. Although in general Fire point can be assumed to be 10°C higher than the flash points. A fire point happens, when an ignition source is applied and the heat produced is self-sustaining, as it supplies enough vapors to combine with air and burn even after removal of ignition source. The flash point for this work is 144 OC. This result shows appreciable consistency with both ASTM, standard for biodiesel and works of other researchers.

4.2.7 Acid value

Acid value is the mass of potassium hydroxide (KOH) in milligrams required to neutralize one gram of chemical substance. The acid number is used to quantify the acidity of a substance. The acid value obtained for this work is relatively high compare to the EN standard and reported work of Acid value measures directly the free fatty acids content of the methyl ester. It clearly helps to state the corrosive nature of the fuel, its filter clogging tendency and the amount of water that may be likely present in the biodiesel. This parameter can also be used to measure the freshness of the biodiesel. The higher the acid

value the lower the quality of the fuel. The result from this work shows Acid value of 1.0 which is in quite in agreement with the ASTM norms.

IV.CONCLUSION

In the present study, CaO-MgO was identified as an effective heterogeneous catalyst for the transesterification of soybean oil and methanol. The biodiesel was produced at the optimum condition and the physical properties of the produced biodiesel were evaluated and compared with biodiesel ASTM.

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