

Optimisation of embedded CaO catalyst for biodiesel production

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Abstract:

In recent years, there has been much attention on climate change, boosted by the Paris agreement, and energy has become a topical issue across the globe. Many are looking for alternatives to conventional petroleum which leaves a high carbon footprint. Biodiesel is a green and renewable source of energy which can be produced from waste oil. Due to such environmental advantages, biodiesel could be one of the future substitutes of conventional petroleum as an environmentally friendly source of energy, therefore it is important to research its production. This research aims to synthesize biodiesel using novel embedded CaO catalyst for biodiesel production through experimentation of different independent variables to achieve optimal yields of biodiesel. Certain tests were carried out to find out more about the biodiesel such as finding the calorific values, density and kinematic viscosity and comparing them to ASTM Biodiesel Standards. From changing the ratios of methanol to oil, it was found that 12:1 molar ratio of methanol to oil is the most optimal as it produced 64% yield and corresponds with other similar research papers. This suggests that a molar ratio of 12:1 with CaO catalyst could be a viable option for biodiesel production, although future research has to be carried out to confirm this.

1. Introduction

1.1. Literature review

According to Huang, Zhou, Lin (2012), due to the increasing awareness of the depletion of fossil fuel resources and environmental issues, biodiesel has become more attractive in recent years. Biodiesel production is a promising and important field of research because of the relevance it gains from the rising petroleum price and its environmental advantages. Relative to the fossil fuels they displace, greenhouse gas emissions are reduced 12% by the production and combustion of ethanol and 41% by biodiesel. Biodiesel consists of mono alkyl esters of long chain fatty acids. It can be produced from vegetable oils or fats by chemical transesterification.

Several processes for biodiesel fuel production have been developed, among which transesterification using alkali-catalysis gives high levels of conversion of triglycerides to their corresponding methyl esters in short reaction times. This process has therefore been widely utilized for biodiesel fuel production in a number of countries (Fukuda, Kondo and Noda 2001). Biodiesel production by recycling WCO and methanol in the presence of calcium oxide (CaO) nano-catalysts offers several benefits such as economic, environmental and waste management (Degfie, Mamo, Mekonnen 2019). From experiments done by Degfie, Mamo, Mekonnen (2019), the highest conversion of WCO to biodiesel was estimated to be 96%, at optimized experimental conditions i.e., 50 °C, 1:8 WCO oil to methanol ratio, 1% by weight of catalyst loading rate and 90 minutes reaction time, which is among few highest conversions reported so far. Calcium Oxide has become more popular as a catalyst for biodiesel production through transesterification because according to Liu, He, Wang, Zhu, Piao (2006), producing biodiesel using CaO as a solid base catalyst has many advantages, such as higher activity, mild reaction conditions, long catalyst lifetimes, low catalyst cost and so on. Furthermore, due to the low production cost and high basicity of CaO, it has been used as a catalyst by many researchers (Granados *et al.*, 2007). Liu et al. (2012) studied the transesterification of oil using CaO as a heterogeneous catalyst and achieved 95% conversion of the oil to the ester using a methanol to oil molar ratio of 12:1, 8% of CaO in relation to the oil mass and a reaction time of 3 h. Viola et al. (2012) performed the same reaction and reached 93% of conversion with 80 min of reaction time using CaO as the catalyst at a reaction temperature of 65 °C using 5% of catalyst in relation to oil (m/m). The authors used a methanol to oil molar ratio of 6:1. The results of these studies indicate that the conversion of vegetable oil to biodiesel through transesterification using CaO as a heterogeneous catalyst provides a maximum conversion of 95% in the production of biodiesel when all of the parameters are optimized (Colombo, Ender, Barros 2016). Among the researched heterogeneous catalysts, CaO shows potential in the transesterification reaction. The catalyst is able to be reused (cost effective), has a tolerance of moisture and FFA (which allows the use of lower-quality used/waste oils), is inexpensive (obtainable from waste shells), has low methanol solubility, is non-corrosive and is environmental friendly (Boey, Maniam, Hamid 2011). This shows that Calcium oxide is an efficient catalyst which would have representative value when its conditions are optimised because of its potential.

However, a major challenge of calcium oxide as a catalyst is its unsuitability towards the very high FFA content feedstock (Boey, Maniam, Hamid 2011). Apart from that, according to Ivana, Miodrag, Ljiljana, Dejan (2016), the rate of transesterification process realized using heterogeneous catalysts like calcium oxide is generally lower than that of the homogeneous catalysts because of diffusion problems of reactants to the active sites of solid catalyst, which is associated with the three-phase system (oil-methanol-solid catalyst). Also, active species of almost all the heterogeneous base catalysts leach out into the reaction mixture and produce soap if vegetable oil contains free fatty acids (FFAs). Dissolving of solid-base catalysts decreases the possibility of their reuse in subsequent batches or for longer use in continuous processes. It is also important to be pointed out that solid-base catalysts are mainly active in the transesterification at the temperatures around the boiling point of methanol. Also, the process of making biofuels currently does not provide a net energy gain, and does not offer clear environmental and economic benefits, and reduce food supplies and increases costs, and hence falls short of economic requirements and is staying as a niche market, without wide-scale public use (Clemente, 2015).

1.2. Objectives

Our first objective is to produce purified biodiesel that is usable in based on ASTM International standards utilising novel embedded calcium oxide catalysts. Our second objective is to optimise the efficiency of transesterification of biodiesel using embedded calcium oxide catalyst and increase the yield by finding the optimal mass to volume of catalyst to oil to be used for transesterification; finding the optimal temperature of reaction for transesterification; finding the optimal molar ratio of alcohol to oil to be used for transesterification.

1.3. Hypothesis:

It is hypothesised that firstly, embedded CaO is able to catalyse the transesterification reaction. Secondly, the optimal temperature of reaction is at 60°C. Thirdly, the optimal molar ratio of alcohol to oil for the biodiesel of best yield and quality is 9:1. To test our hypotheses, different independent variables were intended to be experimented with. The first independent variable is the molar ratio of alcohol to oil and the different ratios are 6:1, 9:1 and 12:1. The dependent variable is the yield of the reaction. The second independent variable is the mass to mass ratio of

catalyst and oil and the different ratios are as follows 1:100, 2:100 and 3:100. The dependent variable is the yield of reaction. The temperature of transesterification is the third independent variable and the different temperatures required for the reaction is 50°C, 60°C, 70°C. The dependent variable is the yield of reaction.

2. Methods and Materials

2.1. Apparatus/equipment and chemicals required

2.1.1. Chemical Materials

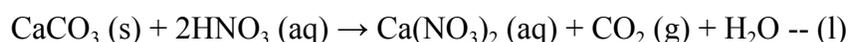
Calcium carbonate was obtained in the form of egg shells, collected from the households of group members and school canteen stalls. Calcium oxide was a result of calcining calcium nitrate from the reaction between calcium carbonate from egg shells and nitric acid. Leftover waste coffee powder was obtained from the households of group members. Methanol, nitric acid, fresh sunflower oil, Dodecanol-1 was obtained from the SRC lab.

2.1.2. Apparatus

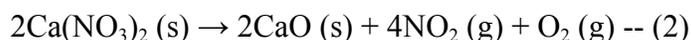
Hot water baths, 500ml 2 neck round bottom flasks, thermometers, weighing scales, magnetic stir bars, laboratory hot air oven reflux condensers weighing scales, centrifuge machine, centrifuge tubes were all obtained from the chemistry laboratory. The burette and metal ball bearing were obtained from the physics laboratory.

2.2. Obtaining and embedding calcium oxide catalyst on waste coffee powder

9.15g of pulverized eggshells was dissolved in 200ml of 2 molar nitric acid to form calcium nitrate solution. The formation of calcium nitrate solution is seen in equation (1)



Vacuum filtration was carried out to remove undissolved impurities resulting from eggshells. 50g of coffee powder was then soaked in the calcium nitrate solution for 6 hours. Coffee powder was dried for 12 hours in the laboratory oven at 60°C. Coffee powder which was soaked in calcium nitrate solution was calcined in the laboratory oven at 800°C for 2 hours to obtain an embedded calcium oxide catalyst. The formation of calcium oxide is seen in equation (2).



2.3. Production of biodiesel

In transesterification, a glyceride in the vegetable oil reacts with an alcohol, which we will use methanol in this case, in the presence of a catalyst to form fatty acid methyl esters (FAME) and glycerol .

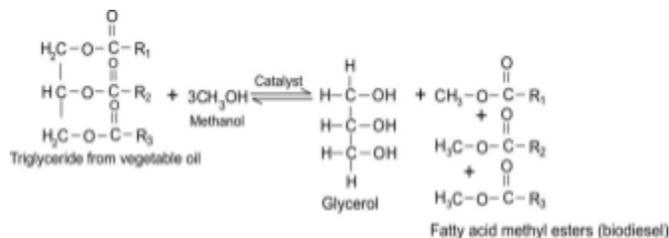


Fig. 1: Transesterification reaction

2.3.1 Independent variable: Methanol to oil ratio

200ml of sunflower oil was added into a 2 neck round bottom flask with a magnetic stirrer and a thermometer to keep track of the temperature. A 1:100 mass to mass ratio of catalyst to sunflower oil was used. Methanol is added to a beaker of catalyst at a molar ratio of 6:1 alcohol to oil under a fume hood. A 2 neck round bottom flask filled with 200ml of sunflower oil was placed on a heating mantle and heated to 60°C. A reflux condenser was used to prevent the evaporation of methanol into the surroundings. Embedded calcium oxide was added to methanol in a beaker under a fume hood. Then, the methanol and calcium oxide mixture was added to the pre-heated sunflower oil. The reaction was allowed to take place for 2.5 hours. After 2.5 hours, the impure biodiesel is left to cool to room temperature then filtered into a separating funnel. The previous procedures are repeated with a 9:1 and 12:1 molar ratio of alcohol to oil. The reaction parameters were determined by preliminary research from related literature.

2.3.2 Independent variable: Mass to mass ratio of catalyst to oil

200 ml of waste cooking oil into a 2 neck round bottom flask with a magnetic stirrer and thermometer. Then, the CaO catalyst is added at a mass to volume ratio of catalyst to oil at 1:100. The 2 neck round bottom flask with the oil is preheated on a heating mantle to 60°C. Methanol is added to a beaker of catalyst at a molar ratio of 9:1 alcohol to oil under a fume hood. After the methanol-catalyst mixture is added to the 2 neck round bottom flask, a reflux condenser is placed on one of the necks to prevent methanol from evaporating from heating. The reaction will then take place for 2.5h. After being left to cool, the impure biodiesel is filtered into a separating funnel. The previous procedures are repeated with 2:100 and 3:100 mass to mass ratio of catalyst to oil. The reaction parameters were determined by preliminary research from related literature. **However, this could not be materialized due to time constraints.**

2.3.3 Independent variable: Temperature of reaction

200ml of sunflower oil was added into a 2 neck round bottom flask with a magnetic stirrer and a thermometer to keep track of the temperature. A 1:100 mass to mass ratio of catalyst to sunflower oil is used. Methanol is added to a beaker of catalyst at a molar ratio of 9:1 alcohol to oil under a fume hood. A 2 neck round bottom flask filled with 200ml of sunflower oil was placed on a heating mantle and heated to 50°C. A reflux condenser was used to prevent the evaporation of methanol into the surroundings. Embedded calcium oxide was added to methanol in a beaker under a fume hood. Then, the methanol and calcium oxide mixture was added to the pre-heated sunflower oil. The reaction was allowed to take place for 2.5 hours. After 2.5 hours, the impure biodiesel is left to cool to room temperature then filtered into a separating funnel. The previous procedures are repeated with a temperature of 60°C and 70°C. The reaction parameters were determined by preliminary research from related literature. **However, this could not be materialized due to time constraints.**

2.4. Purification of biodiesel

Once the biodiesel had cooled to room temperature, it was filtered into a separating funnel to retrieve the Calcium Oxide catalyst. Water was sprayed into the separating funnel containing the impure biodiesel for washing. Due to soap formation, the biodiesel was placed in centrifuge tubes and centrifuged at 8000rpm for 5 minutes at a time to separate the impurities from the biodiesel. A dropper was used to remove the biodiesel from the centrifuge tubes to be transferred into containers for storage and for analysis. These steps were repeated twice to ensure that there was the least amount of impurities in the biodiesel samples.

2.5. Yield of reaction

Due to a lack of time, we were unable to send our biodiesel for H-NMR Spectrometer testing, hence, for purposes of testing, we will assume that our samples are 100% biodiesel. To calculate the yield of biodiesel, we will take the mass of biodiesel obtained over the original mass of oil to get a percentage yield.

$$\text{Biodiesel yield} = \text{FAME\% from H - NMR analysis} \times \frac{\text{Mass of biodiesel}}{\text{Mass of oil used}}$$

2.6. Kinematic Viscosity

A sample of about 50cm^3 of biodiesel was placed in a vertical burette held by a retort stand. A metre ruler was held vertically by another retort stand beside the burette. An iPhone camera was used to record the drop of the metal ball bearing at 240fps in slow motion. The video was framcounted to find the time of drop and the terminal velocity of the ball was recorded.

$$v = \frac{gd^2(pp-pm)}{18\mu}$$

$$KV = \frac{v}{pm}$$

Where v is dynamic viscosity; g is acceleration due to gravity; d is diameter of ball bearing; pp is density of metal ball bearing; pm is density of biodiesel sample; μ is terminal viscosity; KV is kinematic viscosity

2.7. Calorific Value

A simple calorimeter was constructed with an alcohol burner below a test tube supported by a board, with 10ml of deionized water in the test tube and a thermometer to measure the change in water temperature. Alcohol burners were filled halfway with samples of biodiesel obtained and set alight when the water was at thermal equilibrium with the surroundings and extinguished when the temperature of water reached 60°C . Its mass was measured before and after it was burnt to record change in mass. This was repeated thrice. The amount of heat energy in the water immediately after the flame was extinguished can be calculated using the equation below.

$$Q_{\text{water}} = m \cdot c \cdot \Delta T$$

Where Q is heat energy (J), m is mass of water (g), c is the specific heat capacity of water ($4.18\text{ J/g }^\circ\text{C}$) and ΔT is the change in temperature ($^\circ\text{C}$). When the heat energy is divided by the change in fuel mass, calorific value is obtained.

$$\text{Obtained Calorific Value} = \frac{Q_{\text{water}}}{\text{Change in fuel mass}}$$

However, not all of the heat energy produced by the combustion of biodiesel was transferred to the water as heat energy was lost to the surroundings. Hence, an approximate value of efficiency of heat transfer to the test tube of water was deduced by burning an alcohol of known heating value, Dodecanol-1. An average calorific value was obtained over three repetitions.

$$\text{Efficiency} = \frac{\text{Obtained Calorific Value (J/g)}}{42664 \text{ (J/g)}}$$

The enthalpy change of combustion of Dodecanol-1 is -7950 kJ/mol (Freeman & Bagby, 1989), which gives a calorific value of 42664 J/g as the molar mass of Dodecanol is 186.34 g/mol. Hence, by dividing the obtained calorific value of Dodecanol-1 by the actual calorific value of Dodecanol-1 we obtain the efficiency. Thus, we can find out the biodiesel's true calorific value by dividing its obtained value by the efficiency of heat transfer to the test tube of water.

$$\text{Calorific value} = \frac{\text{Obtained Calorific Value}}{\text{Efficiency}}$$

2.8. Density

The mass of biodiesel was measured using a weighing scale accurate to 2dp. The volume of biodiesel was measured using a measuring cylinder.

3. Results and Discussion

3.1. Yield of reaction

For the biodiesel sample with a 6:1 methanol to oil ratio, our yield is 46.0%. For the biodiesel sample with a 9:1 methanol to oil ratio, our yield is 50.2%. For the biodiesel sample with a 12:1, our yield is 64.0%. Our yield was greatly diminished due to the saponification reaction.

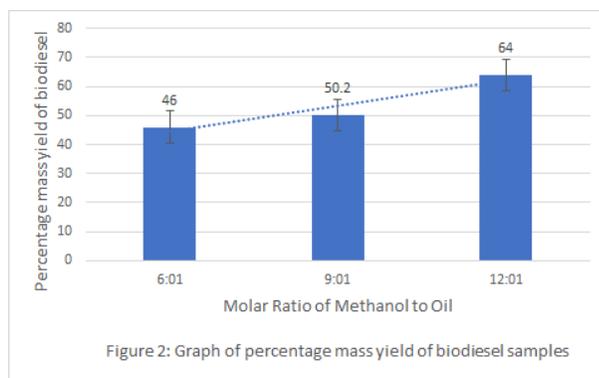
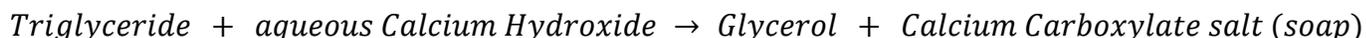


Figure 2: Graph of percentage mass yield of biodiesel samples

Calcium oxide dissolved in water to form Calcium cations and hydroxide anions. Aqueous Calcium hydroxide reacts with a triglyceride to form soap and glycerol.



If biodiesel has a high soap level, then water washing can lead to emulsions that can cause significant yield loss. (J. Wall et al. 2010) Nonetheless our values correspond with the paper, Prediction of biodiesel yield during transesterification process using response surface methodology (M. Mohamad et al., 2017), that also used a CaO catalyst. Due to a lack of time, the experiments for the independent variable of mass to mass ratio of catalyst to oil and the temperature of reaction were not carried out.

3.2. Calorific Value

Tested values of calorific values of dodecanol were expressed as a percentage of

| | Calorific values (6:1) / (MJ/kg) | Calorific values (9:1) / (MJ/kg) | Calorific values (12:1) / (MJ/kg) |
|-----------------|----------------------------------|----------------------------------|-----------------------------------|
| Trial 1 | 7.279 | 6.715 | 6.543 |
| Trial 2 | 7.155 | 6.935 | 7.088 |
| Trial 3 | 7.401 | 6.825 | 6.816 |
| Adjusted values | 39.99 | 37.50 | 37.45 |

Table 2: Comparison of calorific values of biodiesel samples made using different molar ratios of methanol to oil

| | Dodecanol 1 |
|---------|-------------|
| Trial 1 | 7630.7 J/g |
| Trial 2 | 7700.7 J/g |
| Trial 3 | 7986.7 J/g |

Table 1: Trial values of calorific values of dodecanol used to determine efficiency of system

actual energy values of dodecanol to find the system's efficiency of 18%. All 3 calorific values fulfill EU standards of minimum 35MJ/kg. Biodiesel of 9:1 and 12:1 molar ratio of methanol to oil correspond with other research papers. One reason 6:1 molar ratio of methanol to oil produced biodiesel of anomalous calorific value could be that the reaction was incomplete and unreacted sunflower oil was mixed with biodiesel as they have similar densities and could not be removed by water washing as it is non-polar, giving the biodiesel a higher calorific value. However, this test is not ideal in terms of accuracy, as heat is not fully transferred to the test tube of water and the efficiency of heat transfer calculated does not account for time taken to reach 60°C which affects heat loss hence the results obtained are only approximate values.

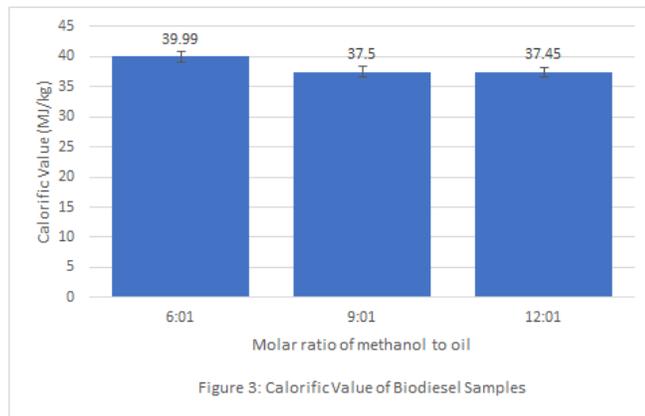


Figure 3: Calorific Value of Biodiesel Samples

3.3. Density

To calculate the density of the biodiesel, we used the equation m/v . We will be referring to the ASTM Standards EN ISO 3675, EN ISO 12185, the lowest density acceptable is 860kg/m³ and the highest density acceptable is 900kg/m³. All densities obtained are acceptable as they correspond with the ASTM standards.

Table 3: Comparison of densities of biodiesel made from different molar ratios of methanol to oil

| Identity of sample | Sample with 6:1 molar ratio of methanol to oil | Sample with 9:1 molar ratio of methanol to oil | Sample with 12:1 molar ratio of methanol to oil |
|--------------------|------------------------------------------------|------------------------------------------------|-------------------------------------------------|
| Density | 900kg/m ³ | 890kg/m ³ | 890kg/m ³ |

3.4. Kinematic Viscosity

In the American standard ASTM D6751, the requirements for the kinematic viscosity of the biodiesel is 1.9–6.0 cSt (Knothe & Steidley, 2007). The kinematic viscosity showed a slight downward trend as the molar ratio of methanol to oil increases. However, the kinematic viscosity of neither samples fulfilled the ASTM standards for kinematic viscosity. Given that all other standards

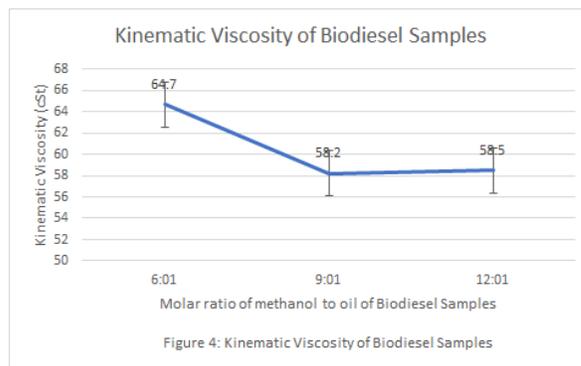


Figure 4: Kinematic Viscosity of Biodiesel Samples

indicate that the sample displayed properties of biodiesel, and the fact that the values obtained were significantly different from expected results, it is likely that these results are due to

experimental errors and not because the samples were not biodiesel. Firstly, the samples were not preheated to 40°C before testing, so the viscosity of the samples at the lower room temperature was likely much higher than its viscosity at 40°C. Secondly, the burette used might not have been sufficiently long for the metal bearing to reach terminal velocity, also resulting in the values being higher than expected. Thirdly, buoyant force of the metal ball was not taken into account as it could contribute to the decrease in terminal velocity, leading to a higher kinematic viscosity. Lastly, a ball with a smaller diameter should have been used to decrease the Reynolds number and reduce turbulence which leads to inaccuracy of results (Ballereau et al., 2016)

3.5. Identification of FAME

A sample of biodiesel from transesterification reactions of 6:1, 9:1, 12:1 molar ratios of methanol to oil was planned to be sent to NUS for H-NMR spectrometry analysis. However, this could not be materialized due to time constraints.

4. Conclusion and Recommendations for future work

Embedded CaO catalyst can be used to synthesize biodiesel from sunflower oil as the density and calorific values were all within acceptable ranges by ASTM standards, which suggests that the biodiesel is of good quality. However, it cannot be certain that the biodiesel produced is fit for industrial and commercial use due to limitations when measuring the kinematic viscosity of biodiesel as other potential factors like turbulence etc may cause discrepancies in the results. Other factors determining the quality of biodiesel are calculating the acid number, flash, fire point etc. but were not measured due to a lack of equipment and time and they can be researched in the future. After measuring the yield of biodiesel, it can be concluded that the molar ratio of methanol to oil of 12:1 is optimal, followed by 9:1 and 6:1. Higher methanol to oil molar ratios should be explored to find out if the upward trend in yield continues, so as to find the true optimal molar ratio of methanol to oil. During future research, biodiesel samples should be sent to labs for H-NMR testing to ensure the successful synthesis of FAME. Other independent variables such as mass to mass ratio of catalyst to oil and temperature of reaction can be considered to conclude the most optimal conditions for the highest yield of biodiesel. Furthermore, other types of oil could be explored, mainly waste cooking oil to reap the full

environmental benefits of biodiesel, exploration could be done on whether its more affordable price can offset costs required to clean the oil before reaction.

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