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ORIGINAL RESEARCH PAPER

Production of Biodiesel from Palm Oil Using Ethanol and Sodium Hydroxide Catalyst

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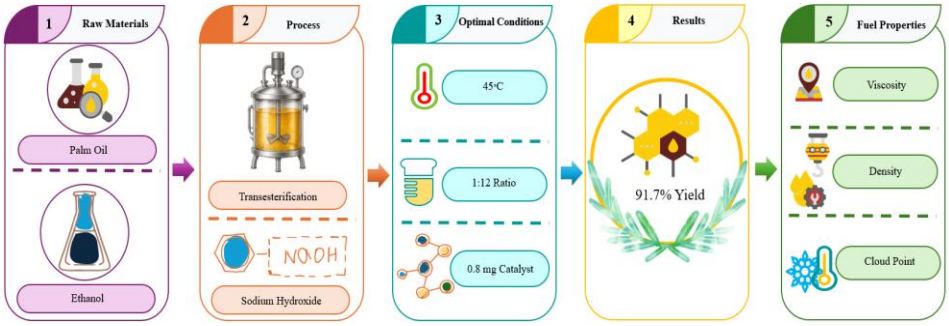
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A B S T R A C T

Biodiesel is a renewable, clean-burning alternative to conventional diesel that enhances lubricity and operates without requiring engine modifications. This study investigates the transesterification of palm oil using ethanol and sodium hydroxide (NaOH) to determine optimal production parameters and evaluate the fuel's physical properties. The transesterification process was conducted by reacting 500 mL of preheated palm oil with ethanol under varying conditions. The free fatty acid content yielded an average acid value of 1.2 mg KOH/g. Process variables, including reactant proportions, catalyst loading, temperature, and agitation speed, significantly influenced the final yield. Experimental results identified the optimum operating conditions as a reaction temperature of 45°C, moderate stirring, an oil-to-ethanol molar ratio of 1:12, and a catalyst loading of 0.8 mg catalyst/g oil, achieving a peak biodiesel yield of 91.7%. Fuel characterization including kinematic viscosity, density, refractive index, cloud point, and pour point demonstrated close conformance to commercial diesel standards. These findings confirm the technical viability of ethanol as an effective acyl acceptor for palm oil transesterification, while future research should address the broader sustainability and food-versus-fuel trade-offs associated with edible feedstocks.



1. Introduction

Biodiesel is defined as mono-alkyl esters of fatty acids mainly derived from vegetable oils or animal fats. Due to the increasing cost of fuel, there is great interest in biodiesel production as it is an alternative fuel for diesel engines. It can be used pure or in blends with diesel fuel in unmodified diesel engines. Biodiesel is biodegradable, nontoxic and reduces exhaust pollutants. The fuel is attractive because it can be produced easily from renewable feedstock's such as waste cooking oil and various plant seeds (Valle et al., 2010). According to the International Energy Agency, the largest biodiesel producer is Germany, which accounts for 50% of global production. Biodiesel is currently most often used in 5% (B5) to 20% (B20) blends with conventional diesel, or even in pure B100 form. Biodiesel production is based on trans-esterification of vegetable oils and fats through the addition of an alcohol and a catalyst, which results in glycerol as a by-product. The various feedstock includes rapeseeds, sunflower seeds, soy seeds and palm oil seeds from which the oil is extracted chemically or mechanically. Biodiesel is commercially produced using homogeneous base catalysts such as sodium hydroxide or potassium hydroxide, because alkali-catalyzed transesterification occurs easily under mild reaction conditions (Demirbas, 2007). The aim is to investigate the production of biodiesel from palm oil. The palm oil will be reacted with ethanol in the presence of sodium hydroxide as a catalyst. The objectives for this study are as follows:

- Perform an acid test to determine the amount of free fatty acids in the palm oil.
- To determine the optimum reaction conditions (temperature and stirrer speed, etc.) for the experiment.
- To determine the oil to alcohol molar ratio and the amount of catalyst that yields the largest production of biodiesel.
- To conduct property testing on the biodiesel product.
- Compare experimental results to commercial diesel.

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1.1. Literature Review

The biodiesel refers to methyl or ethyl esters obtained by transesterification of animal fats or vegetable oils. Biodiesel can be blended with commercial diesel. In the case of mixtures, the proportion of biodiesel mixed in petroleum diesel is indicated as B20. This means the mixture consists of 20% biodiesel and 80% petroleum diesel and B100 indicates pure biodiesel.

1.1.1. Biodiesel Production

The commercial method used for the biodiesel production is the transesterification. The transesterification consists of the reaction of oils alkaline catalyst (NaOHusually ethanol or methanol) with the presence of an alkaline catalyst (NaOH or KOH) to produce esters and glycerin.

The reaction takes place at atmospheric pressure and at a temperature 40-60°C. The process uses constant agitation, for a duration of 1 to 2 hours. The triglyceride molecule consists of three fatty acids attached to a glycerol backbone. When a triglyceride from the palm oil reacts with ethanol, three fatty acid methyl esters and a glycerin molecule forms (Madras, Kolluru and Kumar, 2010). Since transesterification is a reversible reaction, an excess amount of alcohol is used to drive the reaction forward. Saka and Kusdiana (2004) reports that 60–100% excess alcohol is generally used to make sure that the reaction reaches completion.

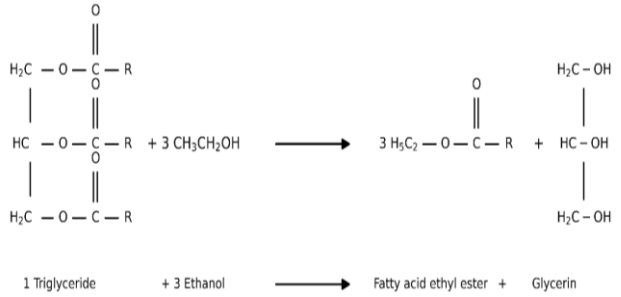


Figure 1. Reaction mechanism for biodiesel production (Nas and Berktray, 2011)

Source: Research Findings

The use of ethanol produces fatty acid ethyl esters (FAEE), and similarly methanol produces methyl esters (FAME). The biodiesel production process results in the formation of glycerol as a byproduct from the reaction. Once the reaction is completed, the biodiesel is washed with water to dissolve impurities such as salts, residual ethanol, and residual glycerin. Thereafter, it is purified using a rotary evaporator. The water and glycerol can contain small amounts of ethanol. Crude glycerol is used in soap production and as a compost accelerator. A second transesterification reaction could be performed to maximize the biodiesel yield.

When a triglyceride reacts with a basic catalyst, it results in the formation of soap (saponification), which is an undesirable side reaction as the yield of biodiesel decreases (Alejandro, 2011). The main factors affecting to the cost of biodiesel are raw material, purification, and storage (World bank, 2008).

Factors Affecting Biodiesel Reaction (Adapted from Environmental Agency, 2012)

1. Oil Purity: The purer the oil, the better the quality and yield of biodiesel.
2. Temperature: The reaction is temperature sensitive. At a high temperaturethe reaction does not reach completion.
3. Stirrer speed: A low to medium speed results in a favorable yield.
4. Type of oil used Restaurant oil: This oil tends to be high in free fatty acids which is unfavorable as this results in lower biodiesel yields due to soap formation.
5. Grease type of oil: Solid and bacon greases are not suitable reactants as they raise the cloud point and not suitable for cold weather problems.
6. Water content in oil: A high water content results in only partial conversion of the oil and promotes formation of soap.

1.1.2. Biodiesel Features and Properties

The biodiesel properties depend on the used feedstock (new vegetable oil, waste cooking oils, animal fats, etc.). The advantages and disadvantages of different feedstocks (Environmental Agency, 2012) can be seen in the following table below.

Table 1. Advantages and disadvantages of different feedstock sources (Environmental Agency, 2012)

Feedstock	Advantages	Disadvantages
1. Fresh Oil (e.g. palm oil)	No water No free fatty acid Constant composition	Highest cost
2. Restaurant Oil	Low cost Constant composition	Has water and food particles High free fatty acid Contains animal fats
3. Home Cooking Oil	Lowest cost Low in free fatty acids	Contains water Mixed composition Has food particles Contains animal fat

Source: Research Findings

1.1.3. Important Properties of Biodiesel (Daniel,2010)

- It can be used pure or blended with diesel in engines and used in the diesel engine without any modifications.
- It can be stored in the same containers as diesel
- It lubricates moving engines and can prolong engine life due to the higher lubrication capacity.
- It improves combustion process. Biodiesel burns better than diesel and Less smoke is produced.
- It is biodegradable, nontoxic and is non-irritating to the skin.
- It does not contain sulfur. No sulfur emissions are emitted during the combustion
- During the combustion biodiesel emits less harmful gases into the environment compared with commercial diesel with reduced CO_2 emissions.
- The biodiesel transport and its storage are less dangerous due to it having a lower flashpoint temperature than diesel.

1.1.4. Advantages and Disadvantages of Biodiesel (adapted from Wenner, 2018).

- Biodiesel Advantages
  a) Biodiesel provides an alternative in terms of energy sources. It is an important as it allows countries without fossil fuel sources to flourish.
  b) The biodiesel contributes to agricultural and rural development.
  c) It is better for the engine and the environment due to it producing lesser harmful emissions.
  d) It is made from renewable materials thus ensuring fuel security.
- Biodiesel Disadvantages
  a) There is competition for the limited resources such as water and land as a growing population requires more food and fuel.
  b) The cost as biodiesel production is expensive compared with petro-diesel.
  c) The biodiesel needs more additives, mainly in cold countries, due to its high cloud point.
  d) Lower long-term storage stability. The biodiesel becomes rancid due to oxidation and bacterial air. This rancidity process produces aldehydes, ketones, and acids, which have strong and unpleasant odors.
  e) It is required 1.1 liters of biodiesel to replace one liter of petroleum diesel, because of their lower calorific power.

Biodiesel may dilute the lubricating oil of engines.

1.1.5. Biodiesel Quality Tests

According to the American Society for testing and methods(ASTM), there are numerous tests one can perform on a biodiesel sample such as acid number, cetane number, cloud point, flash point, oxidation stability, pour point, viscosity, and sulfur content tests etc. Testing can cost an average of $325 per sample (ASTM, 2018). If a sample passes the tests, it will be deemed a satisfactory biodiesel product that is acceptable to use. The results of the property testing will be compared to the values shown in Appendix C by crimson renewable energy.

2. Method

2.1. Methodology

2.1.1. Calculations and Definitions for biodiesel production

The yield of biodiesel indicates the percentage of biodiesel produced, in relation to the theoretical volume calculated.

Yield Calculation: Percentage Yield= (Mass of oil used / Mass of biodiesel obtained)×100

Density Calculation: Density (kg/m^3)= Specific Gravity × Reference Density

The density is defined as "the mass per unit volume of any liquid at a given temperature". Biodiesel has a slightly higher density compared to diesel.

API Gravity: The American Petroleum Institute gravity (API gravity) is a measure of how heavy or light a petroleum liquid is compared to water. It is used to compare densities of petroleum liquids.

API Gravity Calculation: API= (141.5/ SG) −131.5

Viscosity: It is an indicator of "the measure of resistance to flow of a liquid due to internal friction of one part of a fluid moving over another". Biodiesel has a similar viscosity to the diesel. High viscosity values can be a result of a not efficient washing and due to any remaining mono-glyceride.

Refractive index: It is the relation between light speed in the vacuum and the light speed through the substance (Claire MacLeod, 2008).

2.1.2. Material, Equipment and Experimental Procedure

Materials for biodiesel production.

Palm Oil

Ethanol

Sodium Hydroxide Pellets

Phenolphthalein Indicator

Ice

Deionized Water

Apparatus Required

Suitable beakers (500ml, 1000ml and 2000ml) and measuring cylinders.

Spatula

Stirrer rod

Thermometer

Stopwatch

Para-film

Burette

2.1.3. Experimental Equipment

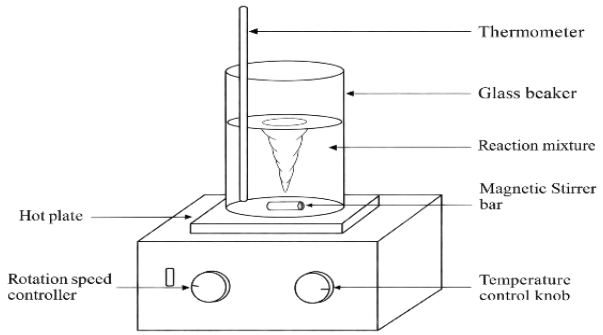


Figure 2. Hot plate with magnetic stirrer bar (adapted from Naiker,2018).

Source: Naiker,2018

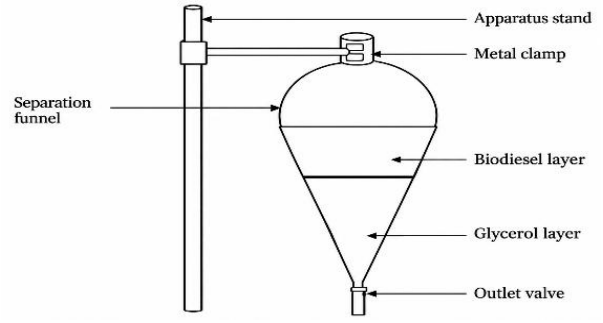


Figure 3. Separating funnel to separate the biodiesel from glycerol (adapted from Naiker, 2018).

Source: Naiker,2018

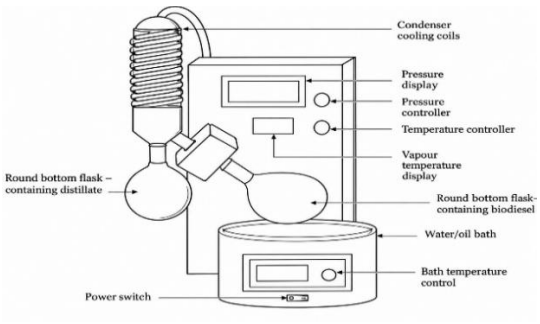


Figure 4. Rotary Evaporator to purify biodiesel sample (adapted from Naiker,2018).

Source: Naiker,2018

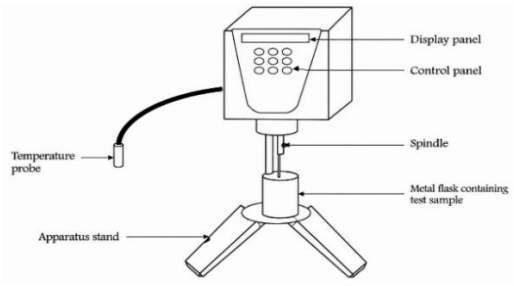


Figure 5. Viscosity Meter (adapted from Naiker,2018).

Source: Naiker,2018

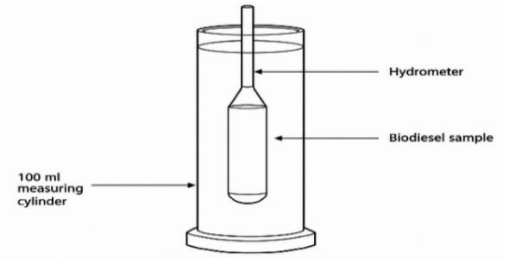


Figure 6. Hydrometer to determine the density of sample (adapted from Naiker,2018).

Source: Naiker,2018

2.1.4. Experimental Procedure (adapted from Naiker,2018)

Initially a blank and actual titration needs to be conducted to determine the amount of free fatty acids contained in the palm oil.

- **Blank titration**

A standard solution was prepared using 1 gram of sodium hydroxide pellets that was dissolved in 1 liter of water. Approximately 10 ml of ethanol was measured and was added to a beaker. Three drops of phenolphthalein indicator were added to the alcohol and was stirred. The standard solution was placed in a burette and was titrated against the alcohol-phenolphthalein mixture. The mixture was continuously swirled to ensure proper mixing took place. The solution reached the endpoint when the colour turned a light pink.

- **Actual titration**

10 ml of ethanol was mixed with 1 ml of palm oil and three drops of phenolphthalein indicator. The standard solution was titrated against the alcohol-oil mixture until the mixture turned a light pink. The amount of standard solution used was taken as the acid number of the oil.

- **Biodiesel production by transesterification**

- 500ml of palm oil was heated to 40°C on the heating plate.
- The number of moles of oil was calculated and was multiplied by three ,due to stoichiometry, and was then multiplied by 12 in accordance with the selected molar ratio of oil to alcohol of 1:12. This calculation allows us to calculate the amount of alcohol and catalyst is needed.
- The calculated amount of alcohol is added to another beaker. The catalyst is then measured and completely dissolved in the alcohol.
- Once dissolved, this alcohol-catalyst mixture can be added to the heated palm oil for the reaction to take place. The hot plate and stirrer

were switched on. The speed of the stirrer was low to moderate.

- A thermometer was placed into the beaker, and the beaker was sealed with Parafilm to prevent ethanol vapour from escaping. The reaction interval was 1 hour once the temperature stabilized at 45 °C. Soon after mixing, there should be two layers visible.

- Separation and Washing of Biodiesel

Once the reaction concluded, the product was placed in a separatory funnel for 20 minutes until two distinct layers were formed. The upper layer was biodiesel, and the lower layer was glycerol. After the settling period, the funnel tap was opened, and glycerol was removed.

Deionized water was heated to 50 °C, and 10 mL was used to wash the biodiesel product. After washing, the biodiesel was left to settle for 10 minutes; the lower impurity layer was removed, and the biodiesel product was stored in a beaker ready for further washing and quality testing.

The quality was tested by mixing 100 mL each of biodiesel and deionized water, and the mixture was shaken for 30 seconds. Thereafter, it was left to settle to ensure that the biodiesel was of a suitable quality.

- Washing and drying of biodiesel

The biodiesel product was washed again with 50°C of deionized water to dissolve any impurities. The biodiesel was then placed on a hot plate to remove any remaining water. The product was then subjected to the rotary evaporator at 120°C at 50 mbar. This allows for the purification and drying of the biodiesel product.

- Property testing of biodiesel

The biodiesel product was tested for density, viscosity, refractive index, pH, cloud point, and pour point test. The equipment was set up and calibrated with the aid of the laboratory technicians.

- Density test

80 ml of biodiesel was filled in a 100 ml volumetric cylinder. A hydrometer was submerged, spun, and allowed to settle in the volumetric flask. The specific gravity was read off the hydrometer, and thereafter the density can be calculated.

- Viscosity test

7.1 mL of biodiesel was added to the metal flask of the viscometer. Thereafter, the viscometer was stabilized for around 30 minutes. After stabilization, it produced a viscosity reading.

- Refractive Index

Deionized water is used to clean the refractometer, and then a few drops are inserted onto the plate to zero the refractometer.

Similarly, four drops of biodiesel sample were placed on the plate, and the refractive index was obtained.

- PH test

A measurement probe was inserted into a 30 ml sample of biodiesel, and the pH was obtained.

- Pour Point Test

50 mL of warm biodiesel was partially submerged in a 500 mL crushed ice slurry bath. A thermometer was inserted to obtain the temperature. The sample was inspected at five-minute intervals to check for sagging of the surface. The temperature at which sagging does not occur is known as the pour point.

- Cloud point

A 50ml sample of biodiesel (containing a thermometer and sealed with Parafilm) is partially submerged in a 500 mL crushed ice slurry bath. The sample is monitored every three minutes for clouding of the sample. The clouding point is the temperature at which first clouding appears.

3. Results and Discussion

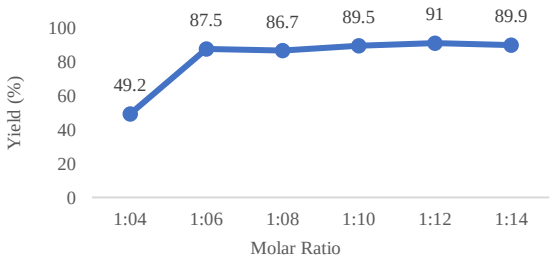


Figure 7. Biodiesel yield obtained for various oil to alcohol molar ratios.

Source: Research Findings

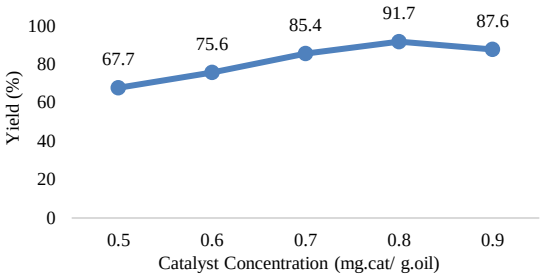


Figure 8. Biodiesel yield obtained for various Catalyst concentrations.

Source: Research Findings

3.1. Property Testing of Biodiesel Sample

Table 2. Property testing on the biodiesel sample produced and commercial petroleum diesel adapted from Crimson Renewable Energy (2006).

Property	Biodiesel Sample 1:12 at 0.8 mg.Cat/g.oil	Commercial Petroleum Diesel
pH	9.14	9.24
Refractive Index	1.394	1.42 – 1.475
Viscosity (cSt)	1.69	3.3 – 3.5
Cloud Point (°C)	12	5 - 15
Pour Point (°C)	10	15 - 35
Specific Gravity	0.828	0.858
API	39.39	33.42
Density (kg/m^3)	828	858

Source: Research Findings

3.2. Discussion

3.2.1. Suitability and the quantity of reactants required to produce biodiesel.

Before undertaking any experiments, the suitability of reactants had to be confirmed. According to Naicker, short-chain alcohols (ethanol) along with homogeneous base catalysts, such as sodium hydroxide, are appropriate reactants. Due to their molecular structure, longer carbon chains are more likely to be influenced by steric hindrance effects. This hindrance is the result of electron overlap, which prevents molecular rearrangement from taking place.

An optimum range was employed for the temperature and catalyst amount, oil to alcohol ratio used in the reaction. Based on ranges by Gabriel (2018), an initial range of 40 to 60°C, 0.6 to 1 mg.cat/g.oil and 1:6 to 1:12 was selected for the temperature, catalyst, and oil to alcohol molar ratio, respectively. Due to time constraints and the limited quantity of ethanol that was initially available, this literature range was selected as the experimental starting point instead of conducting an ANOVA analysis (requiring 24 runs) to determine optimum quantities. Using the initial range, our optimum conditions were quickly achieved by experimentation which saved time and resources. A further 10 litres of ethanol was ordered to ensure that there were no delays in the experimental work. Since many properties of the product were to be tested, a larger amount of biodiesel had to be produced. Thus, 500 ml of palm oil (458g) was used for each run. The experimental procedure was adapted from Naicker,2018. It can be seen in Appendix B that the quantity of ethanol used was determined by the reaction stoichiometry of 1 mole of oil to 3 moles of alcohol, which is then multiplied by the selected molar ratio for the experiment. Hence, a higher molar ratio required a larger amount of alcohol, which produced a larger amount of biodiesel, as seen in Figure 7.

3.2.2. Optimum conditions for transesterification and challenges encountered during experimentation.

Trial and error within the suggested range was employed to find the appropriate conditions. From the trials, it was evident that various factors such as reactant quantity, temperature, and stirrer speed directly influenced the yield of biodiesel. According to Alejandro, an excess amount of alcohol must be employed to drive the reaction forward. This can be seen from Figure 7 that

higher molar ratios (requiring a larger amount of ethanol) were needed to drive the reaction forward to obtain a high yield of 90%. The reaction was temperature sensitive and had to be constantly monitored because temperatures over 60°C did not produce two distinct layers of biodiesel. A low to medium stirrer speed aided the production of biodiesel. A fast speed resulted in the mixing of the biodiesel product with the glycerol layer, resulting in a loss of product.

During the trial run process, there were a few challenges that were encountered. The first challenge was that the base catalyst (sodium hydroxide) did not readily dissolve in the ethanol, which affected the output of biodiesel. Hence, the NaOH pellets had to be crushed and thoroughly mixed. The second challenge was the washing of the biodiesel product. The product was extremely sensitive to water, which resulted in the formation of soap. Hence, the washing had to be done dropwise to prevent the loss of the entire product. The third challenge involved the purification of the washed product, as the rotary evaporator was inoperative due to a damaged component. Thus, the washed biodiesel was initially purified at the thermodynamics laboratory rotary evaporator and heated on the hot plate as a temporary solution while the other unit was being fixed. Thus, the transesterification reaction was conducted at 45°C using a slow to medium speed with dropwise washing. 500 mL of palm oil was reacted using a 1:12 oil to alcohol ratio along with 0.8 mg.cat/g.oil of sodium hydroxide catalyst.

3.2.3. Titration of palm oil

A blank and actual titration were conducted to determine the acid number of oil. The acid number is the weight of catalyst needed to neutralize acid in 1g of oil (Ed Vitz et al. 2017). It is a measure of the free fatty acid (FFA) content that is present in palm oil. The titration is conducted in ethanol using phenolphthalein as an indicator. Appendix A shows that an average acid number of 1.2 was obtained. According to Ed Vits, a higher acid number indicates a higher acidity level, and a value of 1.2 is a small value in comparison to 31 for used frying oil. Thus, the small acid value indicates a small amount of catalyst is required to neutralize the FFAs present in the oil.

3.2.4. Biodiesel yields obtained by transesterification of palm oil.

Using the optimum conditions, three runs were conducted to further optimize the yield of biodiesel by varying the oil to alcohol ratio and the amount of catalyst used. This will prevent producing an inferior product, as these biodiesel samples were then subjected to various property tests.

It is evident from Figure 7 that as the oil to alcohol molar ratio increased, the biodiesel yield obtained also increased. The reason the higher molar ratios obtained a higher yield was due to there being enough alcohol present to drive the reaction forward. When varying the molar ratios, the amount of catalyst (0.8 mg cat/g oil) was kept constant. From Figure 7, a 1:12 molar ratio obtained the highest biodiesel yield of 91%. A molar ratio of 1:14 closely followed with a yield of 89.9% but is unsatisfactory in terms of cost as it requires more ethanol and catalyst than the optimum 1:12 ratio. Thus, the oil-to-alcohol ratio had a direct effect on the yield of biodiesel.

The trend can be observed in Figure 8, which shows that as the catalyst concentration increased, the biodiesel yields also increased. Since there is more catalyst present, the neutralization of the acid is more efficient, resulting in a higher yield being obtained. For each of the catalyst runs, the molar ratio of 1: 12 is kept constant. Figure 8 shows that 0.8 mg cat/g oil is the optimum amount of sodium hydroxide catalyst, as it obtained a 91.7% yield. Hence, there is also a direct relationship between the biodiesel yield and catalyst concentration.

3.2.5. Property testing of biodiesel and comparison to commercial diesel.

The biodiesel obtained was tested and compared to the ASTM. The ASTM provides specifications allows one to determine if the biodiesel product can be deemed satisfactory (Bureau,2018). Based on the experimental results from Table 2, upon comparison, one can see that the properties such as pH, cloud point, refractive index, and density are close (within 10% difference) to those of commercial diesel.

The biodiesel property testing produced a viscosity of 1.69 cSt differed greatly from the standard of 3.3 cSt. The pour point of 10 °C was found to be lower than the standard range of 15 to 35°C. A biodiesel density of 828 kg/m3 was close to that of 858 kg/m3 for diesel. These similarities to commercial diesel are advantageous for ethanol in terms of the future choice of ethanol as a suitable alcohol for biodiesel production. A B20 blend (20% biodiesel, 80% diesel) will greatly aid in the reduction of high fuel prices.

Due to time constraints, the gas chromatography (GC) analysis was not conducted. A GC analysis is recommended as it is helpful in identifying the compounds in the product and is useful in determining the purity.

3.2.6. Concern over biofuel competing with crop production

With the human population at 7.4 billion and projected to increase to an estimated 9 billion, there is concern over the availability of resources for food and fuel (Rulli et al,2016). Palm oil is produced is from palm seeds that grow on trees. Hence, there is major concern around land and water that could be used for food production but are rather being used to produce crops for fuel. As biofuel production increases due to the need for lower fuel costs, the poorer countries' food production line will suffer as their limited land and water resources will be competing with the transport sector. Extensive research needs to be done to properly manage the rivalry between resources and to prevent the exploitation of poorer countries.

4. Conclusions

The results of this study lead to the following principal conclusions:

- The optimum conditions were determined experimentally rather than analytically to save time and resources.
- The molar ratio affected the quantity of reactants employed in the transesterification reaction.
- The optimum conditions were 0.8 mg.cat/g. oil catalyst and a 1:12 molar ratio, conducted at 45°C using a medium stirring speed.
- The titration of palm oil produced an acid number of 1.2, indicating a lower acidity level.
- An increase in oil to alcohol molar ratio resulted in an increased biodiesel yield.
- An increase in the quantity of catalyst used increased the biodiesel yield.
- Factors such as reactant quantity, temperature, and stirrer speed directly influenced the yield of biodiesel.
- The biodiesel product, upon testing, closely resembled commercial diesel as it displayed similar properties.
- The competition between food and fuel needs to be properly managed.

A gas chromatography analyses be conducted to determine the purity and to identify the compounds present in the product. A heating plate with more accurate temperature control is required to prevent loss of biodiesel product..

Author Contributions

All authors contributed equally to the conception, preparation, and writing of this manuscript.

Conflict of Interest

Amir H. Mohammadi, in the capacity of Editorial Board, had no involvement in the selection of reviewers or the management of the peer-review process, and the other authors, while observing publication ethics in referencing, declare the absence of any conflict of interest.

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