Investigation of Various Types of Waste Oil for Transesterification into Biodiesel

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1. Abstract

This project aimed to provide new insights into environmentally friendly alternative sources of renewable energy, and reduce the amount of waste cooking oil, therefore saving the environment. Palm oil, soya bean oil and sunflower oil were transesterified into biodiesel with potassium hydroxide as the catalyst. Tests were carried out on these finished biodiesel products to determine their yield, density, closeness to ASTM Standards (3/27 biodiesel conversion test), heat of combustion and glycerol content. Results showed that palm oil yielded the most biodiesel, and also contained the most energy per unit mass. Furthermore, this biodiesel has passed the 3/27 biodiesel conversion test and the free and total glycerol tests, which means palm oil biodiesel has great potential for commercial use. Palm oil is a promising complement or even substitute as fuel for vehicles or machines that run on an internal combustion engine as it has good quality and can be easily found all over the world.

2. Introduction

2.1. The Fossil Fuel Problem

There are 3 types of fossil fuels: coal, crude oil, and natural gas. Coal is cheap and abundant, but it releases a lot of pollutants when burned. Crude oil is rarer but is typically cleaner than coal. It can also be pumped through pipelines and easily refined into fuels like petrol or kerosene. Natural gas is relatively inexpensive and less polluting than coal or crude oil (Metcalfe, 2019). But when these fossil fuels are burned, they release carbon dioxide and other greenhouse gases, which trap heat in our atmosphere, therefore they are the primary contributors to global warming and climate change (Nunez, 2019). Mankind's dependence on natural gas and oil is responsible for a 1.7% increase of carbon dioxide gases in the atmosphere. In 2018, it is estimated that 8.7 million deaths were linked to fossil fuel emissions alone (Kottasova & Dewan, 2021). This is why alternative energy sources such as solar panels, wind turbines and hydroelectricity become viable to prevent further climate change effects on Earth (Gosheva, 2021).

However, certain qualities of fossil fuels are difficult to replicate, such as their energy density and their ability to provide very high heat. This means that using wind and solar power to replace fossil fuels may not be suitable in every situation as alternatives that mimic these qualities of fossil fuels are needed (Gross, 2020).

2.2. Biofuels

Biofuel is derived from biomass, therefore it can be replenished readily. It is considered to be a source of renewable energy, and is commonly advocated as a cost-effective and environmentally benign alternative to fossil fuels, particularly within the context of rising petroleum prices and increased concern over global warming (Lehman & Selin, 2020).

The U.S Department of Energy reported that a biofuel like ethanol produces up to 48% less carbon dioxide than conventional petrol while the use of biodiesel releases only one fourth the amount of carbon dioxide that conventional diesel releases, making it a much more environmentally friendly option as compared to fossil fuels. Also, unlike non-renewable fossil fuels, biofuels come from crop oils, so it can always be produced without exhaustion (Teo, 2017). Biofuel refineries are more environmentally friendly as they release less carbon dioxide and other greenhouse gases into the atmosphere. Biofuels also improve a vehicle's fuel economy by 30%, and minimizes engine wear as it lubricates the engine (Perritano, 2011).

2.3. Production of Biofuels

There are 2 main kinds of biofuels: ethanol and biodiesel. Ethanol is made by fermenting starch or sugar, and it is typically blended with petrol to form a fuel mixture that is most common in the United States and Brazil. Biodiesel is made from oily plants such as soya bean or oil palm, and other oily sources such as waste cooking oil, it can be blended with petrol and diesel to form a fuel mixture too (Lehman & Selin, 2020).

A study published in the Proceedings of the National Academy of Sciences reported that biodiesel provides 93% more net energy per gallon than is required for its production, while ethanol only generates 25% more energy. The study further showed that biodiesel, when compared with petrol, reduced greenhouse emissions by 41%, while ethanol yielded only a 12% decline. Therefore biodiesel is a more energy efficient biofuel than ethanol.

Despite that, an estimated 420 gallons of ethanol can be produced per acre of corn, but only 60 gallons of biodiesel can be produced per acre of soybeans (Uldrich, 2007). This means that biodiesel requires larger land to mass produce compared to ethanol.

In summary, biodiesel is more energy-efficient, although it is limited by its efficiency of production. Therefore, this project will focus on biodiesel instead of ethanol.

2.4. Waste Oil for Biodiesel Production

The production of biodiesel requires oil. There are many types of oil, including lubricating oil, trap grease and cooking oil. However, after these oils are used, most of them are not disposed of properly. Some are washed into sewers, affecting water treatment plants. Some are dumped directly onto the ground to kill weeds or poured onto dirty roads or deserts, where they contaminate surface and groundwater. It can be seen that the disposal of waste oil creates environmental hazards (Katiyar & Husain, 2010).

Recycling these waste oil brings environmental benefits. The Terrapure North Vancouver facility conducted an assessment, where it determined that emissions from the production of recycled lubricating oils were approximately 44700 tonnes less carbon dioxide per year, than if the same volume was produced through refining of virgin crude oil. Recycling waste oil also produces approximately 94000 tonnes less carbon dioxide than if the oil is burned for heat value (Barker, 2021). It is therefore beneficial to recycle waste oil.

Waste oil can be recycled by turning it into biodiesel. As for used lubricating oils including crankcase oils, transmission fluid and hydraulic oil, it is estimated that as much as 700,000,000 gallons of waste lubricating oils are generated annually in the United States, it is important to minimize this large figure as much as possible, through processes like converting them into biodiesel. However, the conversion into biodiesel would mean some challenges such as increasing costs associated with re-refining to remove spent additives and impurities for the biodiesel to be suitable for use (Chansky et al., 1974). This is why converting waste lubricating oil into biodiesel is not a feasible option for this project.

Trap grease can be used to produce biodiesel too. Trap grease is a type of kitchen waste generally disposed at wastewater treatment plants and landfills. A 1998 study by the National Renewable Energy Laboratory of 30 metropolitan areas found that 13 pounds of trap grease are generated per person per year, and about 7.5 pounds of trap grease is needed to make 1 gallon of biodiesel. This means that it is easy to produce high amounts of biodiesel using a limited amount of trap grease. Despite that, challenges to using trap grease for biodiesel production include foul odour, as it has up to 98% FFA, food, trash and water contamination, heavy emulsification and cold flow issues. The trap grease collection system is disorganized and fragmented too (Van Gerpen, 2019), thus making collection of trap grease tough. Hence, production of biodiesel from trap grease is not feasible too.

Another waste oil would be waste cooking oil. According to a comparative study by Lapuerta, Rodriquez-Fernandez and Agudelo in 2008, waste cooking oil is easy to collect from other industries such as domestic usage and restaurants, and also cheaper to produce in small quantities than the refined oil. Converting waste cooking oil into biodiesel reduces production cost, and it prevents environmental pollution as this oil can be recycled instead of being disposed of into the environment and harming it (Ojiego et al., 2014). Additionally, cooking oil such as palm oil is used a lot all across the globe - over 73 million tons in 2020 (Robins, 2021), which creates a lot of waste oil available for production of biodiesel. Palm oil, soya bean oil and sunflower oil are among the most consumed cooking oils in recent years (Shahbandeh, 2021). This is why this project will focus on producing biodiesel from waste palm, soya bean and sunflower oil.

2.5. Introduction (Rationale)

There are multiple types of transesterification, but the most common types are acid-catalysed and base-catalysed transesterification. Acid-catalysed transesterification produces more yield, but the reaction is slow and takes more than a day (Canakci & Van Gerpen, 2003). Base-catalysed transesterification is faster and can be conducted at low temperatures (303-308 K) and pressure (0.1 MPa) (Vekateswarulu et al., 2014). Base-catalysed transesterification is more commonly used commercially (Nimvari, 2015), and among people producing homemade biodiesel (Decker, 2009) as this method is simple and economical (Eckles, 2015).

Organic oils are three long strings of fatty acids attached to a glycerol molecule. The fatty acids can vary in length and in how they are bonded or put together. Different oils have different blends of fatty acids, and therefore they have different chemical properties (Da Tech, n.d.). The biodiesels produced using these oils will have different properties too.

Hence, this project will investigate base-catalysed transesterification on the production of different types of used cooking oil into biodiesel, and study their properties and their use in daily life.

3. Objectives

- To carry out base-catalysed transesterification on different types of waste cooking oil: palm oil, sunflower oil and soybean oil.
- This project will investigate which of these biodiesels have the highest quality.
    - Quality refers to: density, closeness to ASTM standards for biodiesel, heat of combustion, free and total glycerol content.
    - ASTM standards refer to ASTM D6751, where properties such as flash point, viscosity, cloud point, and iodine value are used to determine biodiesel quality (Jääskeläinen, 2009).

4. Hypothesis

4.1 Hypothesis

Biodiesel produced by palm oil will have the highest heat of combustion, lowest free and total glycerol content, and it will be closest to the ASTM standards for biodiesel. This is followed by soybean oil and then sunflower oil.

4.2. Saturated and Unsaturated Fats

Palm oil has the most saturated fats and the least unsaturated fats, followed by soya bean oil and then sunflower oil. Having low unsaturated fats results in a low iodine value, while having high saturated fats results in a high cetane number.

Oil	**Saturated / %**	**Unsaturated / %**
Sunflower Oil	10	86
Soya Bean Oil	16	81
Palm Oil	45	46

4.2.1. Iodine Value

Biodiesels with low iodine value are generally more combustible and efficient than those with higher values. The iodine value of the biodiesel produced increases as the degree of unsaturation increases (Folayan et al., 2019).
This means that palm oil is the most combustible, followed by soya bean oil and then sunflower oil.

4.2.2. Cetane Number

Higher cetane number corresponds to better ignition quality. Cetane number increases with degree of saturation (Folayan et al., 2019).
Palm oil should have the best ignition quality, followed by soya bean oil and then sunflower oil.

5. Materials and Methods

5.1. Materials and Apparatus

The sunflower and soya bean oil were retrieved from SRC, while the palm oil was bought from a local supermarket. Methanol, potassium hydroxide, thymol blue and isopropyl alcohol for the transesterification process were taken from SRC. The dichloromethane, sodium thiosulfate, periodic acid and potassium iodide for the glycerol tests were taken from SRC too.

5.2. Methodology

5.2.1. Base-catalysed Transesterification

100 ml cooking oil was poured into a beaker, and heated to 50-60 °C. 22 ml methanol was poured into another beaker. 1 g of potassium hydroxide was added to 1 l of water in a beaker. This is the titrant solution. 10 ml isopropyl alcohol, 1 ml cooking oil and 4 drops thymol blue were added into a measuring cylinder. The titrant solution was added to the alcohol, oil and thymol blue solution little by little using a burette until it turned blue. The reading of how much titrant solution added was taken. The reading was used to determine how much potassium hydroxide should be dissolved in 22 ml of methanol. The methanol and potassium hydroxide solution was added to the 100 ml cooking oil. The mixture was stirred, dissolved and refluxed for 1 hour for the transesterification process. The cooking oil, methanol and potassium hydroxide solution was poured into the separatory funnel and waited for the time to separate the biodiesel from glycerol.

5.2.2. Test for Yield

The biodiesel produced was put into a beaker. The mass (g) of the biodiesel produced was measured using an electronic balance. The yield % was calculated by the percentage of the mass of the biodiesel produced compared to the mass of the feedstock used.

5.2.3. Test for Density

5.00 cm^3 of biodiesel sample was put into a weighing bottle using a pipette. The mass (g) of the biodiesel sample was measured using an electronic balance. The process was repeated 3 times with new weighing bottles and new 5.00 cm^3 of biodiesel samples. The average across the 3 measurements was taken to determine the mass of 5.00 cm^3 of biodiesel sample. Density of biodiesel sample was calculated as $\frac{average\ mass\ of\ biodiesel\ sample\ (g)}{5.00cm^3}$.

5.2.4. 3/27 Biodiesel Conversion Test

3 ml of biodiesel was added to 27 ml of methanol in a measuring cylinder. The mixture was shaken and stirred for about 30 s and allowed to sit for 300 s.

5.2.5. Heat of Combustion

10.00 g of biodiesel was weighed into a crucible. A wick was added into the fuel and the total mass was measured. A clean test tube was filled with 10.00 g of deionised water and placed into a calorimeter. The initial temperature of the test tube was measured. The crucible was positioned under the test tube. The wick in the biodiesel was lit. The flame was extinguished when the temperature of the test tube reached 90 °C. The final mass of biodiesel was measured. Heat of combustion was calculated using this equation: $\frac{mc\Delta\theta}{mass\ of\ biodiesel\ used}$, where m = 10.00 g, c = 4.186 $Jg^{-1}K^{-1}$

5.2.6. Free Glycerol Test

A sample of 5.00 g of biodiesel was transferred to a 100 ml volumetric flask. 9.0 ml of dichloromethane, followed by 50.0 ml of water were added. The mixture was shaken and deionised water was added to the solution until the solution had a volume of 100 ml. On standing, the mixture separated into 2 layers, the aqueous and oil layer. 25.0 ml of periodic acid and 25.0 ml of the aqueous layer were added into a conical flask. 10 ml of potassium iodide was added and the mixture was allowed to stand for 60 s. The mixture was topped up to 125 ml with deionised water and the mixture was titrated with sodium thiosulfate using starch as indicator. A blank with the same reagents, but without the sample was prepared. The volume of sodium thiosulfate required was used to calculate the percentage of free glycerol. The following formula was used to calculate the percentage of glycerol: Glycerol (%) = $\frac{[B-S][M][0.0230]}{[W]*0.294} \times 100\%$ where B = ml of thiosulfate to titrate the blank, M = normality of thiosulfate, S = ml of thiosulfate to titrate the sample, W = weight of sample extracted in g.

5.2.7. Total Glycerol Test

A 5.00 g sample of biodiesel with 15.0 ml of 0.7 mol dm^{-3} potassium hydroxide in 95% ethanol was refluxed for 30 minutes. The mixture was transferred to a 100 ml volumetric flask, 9 ml of dichloromethane and then 2.5 ml of acetic acid were added. The total glycerol content was determined as described in the free glycerol test.

6. Results and Discussion

6.1. Data Analysis

6.1.1. Yield of Biodiesel

	Sunflower	**Soya Bean**	**Palm**
Mass Of Biodiesel / g	49.95	54.78	67.96
Mass Of Oil Used / g	91.9	91.7	90.4
Yield Percentage / %	54.4	59.7	75.2

From this table, it can be seen that palm oil biodiesel had the highest yield percentage, followed by soya bean oil biodiesel and sunflower oil biodiesel.

6.1.2. Density of Biodiesel

	$5cm^3$ Sunflower	$5cm^3$ Soya Bean	$5cm^3$ Palm
Average Mass / g	4.29	4.41	4.36
Density / g/cm^3	0.858	0.882	0.872

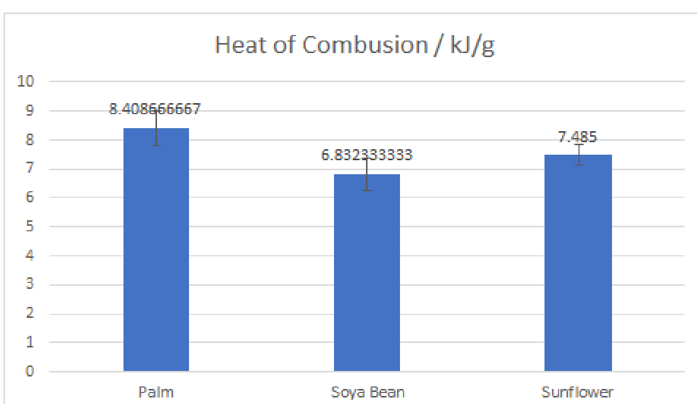
Soya bean oil biodiesel is the most dense, followed by palm oil biodiesel and sunflower oil.

6.1.3. 3/27 Biodiesel Conversion Test

From left to right: palm oil biodiesel, soya bean oil biodiesel, sunflower oil biodiesel

All biodiesel samples are translucent with no fallout. This means all the oils have been reacted to form biodiesel and the biodiesel samples are likely to pass the ASTM D6584 test (*How To Test Biodiesel For Conversion*, 2013).

6.1.4. Heat of Combustion



Palm oil biodiesel has the highest heat of combustion, followed by sunflower oil biodiesel and then soya bean oil biodiesel.
However, these values of heat of combustion is only a fraction of that of petrol, which is around 47.3 kJ/g (*Heat of Combustion*, 2011). This is because the method of burning biodiesel to heat water in a calorimeter was not very efficient, as not all energy from the biodiesel was used to heat the water.
Nonetheless, this method is still used to determine the heat of combustion of biodiesel, and the results hover around the 7.6 kJ/g mark for biodiesel (American Chemical Society, 2013).

6.1.5. Free Glycerol Test

Blank: 23.6 ml

	Sunflower 4.998 g	**Soya Bean 5.014 g**	**Palm 4.991 g**
Sodium Thiosulfate required for titration / ml	23.45	23.40	23.35
Free Glycerol / %	0.0235	0.0312	0.0392

According to the EN 14214 European Biodiesel Standards (Jääskeläinen, 2009), the maximum free glycerol content of biodiesel is 0.02% wt. All three biodiesel samples did not pass this test as their free glycerol contents are higher than the maximum standard.
Nonetheless, sunflower oil biodiesel has the lowest free glycerol content, and is the closest to passing the standards. followed by soya bean oil biodiesel and then palm oil biodiesel.

6.1.6. Total Glycerol Test

Blank: 23.6 ml

	Sunflower 5.004 g	**Soya Bean 5.000 g**	**Palm 5.001 g**
Sodium Thiosulfate required for titration / ml	18.1	17.95	17.95
Total Glycerol / %	0.860	0.884	0.884

According to the EN 14214 European Biodiesel Standards (Jääskeläinen, 2009), the maximum total glycerol content of biodiesel is 0.25% wt. All three biodiesel samples did not pass this test as their total glycerol contents are higher than the maximum standard.

Nonetheless, sunflower oil biodiesel has the lowest free glycerol content, which means it is the closest to passing the standards, followed by soya bean oil biodiesel and palm oil biodiesel, which both have the same average total glycerol content.

7. Conclusion and Recommendations for Future Work

7.1. Conclusion

From the data analysis, it can be concluded that palm oil yielded the most biodiesel, but soya bean oil biodiesel has the highest density. As for heat of combustion, palm oil biodiesel gives out the most energy per unit mass. However, sunflower oil has the lowest free and total glycerol content. Nonetheless, all the biodiesel produced have passed the 3/27 conversion test, although they did not pass the free and total glycerol tests.

In conclusion, palm oil biodiesel has the highest quality, mainly because it brought the most yield and carried much energy. Sunflower oil biodiesel is close second because its energy content is almost as high as palm oil biodiesel but the yield was not ideal, although the free and total glycerol levels are high. For soya bean oil biodiesel, the energy per unit mass is low compared to the other biodiesels, and its yield was not too decent either.

7.2. Future Work

The scope of this study could be expanded by investigating the transesterification of recycled oil instead of fresh oil, as well as further evaluating other properties of biodiesel such as gel point, flashpoint, acid value and iodine number. Methods to increase yield, and heat of combustion of the biodiesels can also be studied.

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