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RESEARCH ARTICLE

Investigating the Quality of Biodiesel Synthesized from Used Cooking Oils

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ABSTRACT: Biodiesel, an alternative to fossil-derived diesel, offers numerous advantages and is produced from used oils through a transesterification reaction. In a transesterification reaction, the triglyceride molecules of oil break down into glycerin and three fatty acid esters (biodiesel). This process for generating biodiesel is relatively simple and cost-effective. Biodiesel contains oxygenated species (esters) and variable unsaturation; hence, the cetane numbers technique is commonly used but cannot directly quantify biodiesel quality. In this project, I used a transesterification reaction to produce seven different biodiesels from used oils. My goal in this project was to investigate various characterization analytical tools to analyze biodiesel quality and identify suitable feedstock for producing high-quality biofuel. I used proton NMR measurements to determine the quality of biodiesel. The ratio of ester protons to unsaturation protons was used to estimate the number of double bonds of freshly prepared and air-treated biodiesels. NMR results revealed that the biodiesel derived from restaurant frying oil is stable to oxidation and ranked the best in quality. The kinematic viscosity of biodiesels is between 4.7 and 6.5 at 20 °C. Biodiesels derived from restaurant-used frying oil are rated the best of all biodiesels produced.

KEYWORDS: Energy, Chemical, Alternative Fuels, Biodiesel, Transesterification.

Introduction

Diesel was invented approximately 150 years ago, and modern society relies heavily on it. It is impossible to imagine a day without diesel.[1,2] Do you know there are about 86 million diesel-engine vehicles in the US? According to the US Energy Information Administration (EIA), in 2021, the transportation sector in the US consumed about 47 billion gallons (1.11 billion barrels) of diesel, an average of about 128 million gallons per day, and diesel is commonly used in large vehicles because diesel engines produce more torque than gasoline engines. Diesel has greater compression resistance and stability.[3,4] Therefore, diesel engines are at least 33% more fuel efficient than comparably sized gasoline engines.

In today's world, diesel drives the economy, and we use diesel for everything. Most daily materials must be shipped and moved from place to place in diesel-operated vehicles. Due to international politics, diesel prices have been unpredictable, and regular diesel prices have increased drastically over the last few years. The most significant disadvantage of using traditional diesel is that it leads to environmental issues, including global warming.

To address this issue, researchers have attempted to develop an eco-friendly fuel equivalent to diesel, biodiesel, which is a form of diesel fuel derived from plants, food, or animals.[1,2] Biodiesel produced from various cooking oils can be seen in Figure 1. Something that needs to be understood is that biodiesel does not directly reduce carbon emissions, but can slow overall emissions into the environment, positively influencing climate change. Biodiesel and diesel both will expel carbon-based products into the atmosphere. Still, the key difference is where the carbon source is coming from.[3,4] When diesel burns, carbon that was once trapped underground is released into the atmosphere. On the other hand, when burning biofuels, we are simply recycling carbon through our atmospheric system. Plants absorb the carbon as they grow and then return it to the air when we burn fuel from those plants. Burning biofuels, therefore, does not increase the overall amount of CO_2 in the atmosphere and has the potential to help slow climate change. Biodiesel is superior to diesel because of its renewable nature, and biodiesel has esters; in contrast, fossil-derived diesel has no oxygens (Figure 2). Oxygen in biodiesel enhances ignition properties and ensures total, proper combustion of the hydrocarbons.

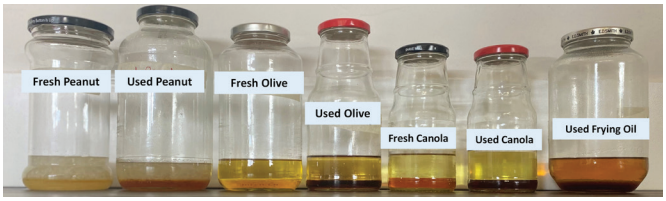

Figure 1: Biodiesels are produced from used cooking oils. The feedstock used to create each biodiesel is specified on labels on the jars. The top visible layer is the biodiesel, and the bottom visible layer is the glycerin.

Although biodiesel came into existence about 30 years ago, due to the diverse nature of the feedstocks (raw materials) and cost, the quantification of biodiesel is still not mature enough, and more work needs to be done.[5] To address this issue, in this project, I investigated the transesterification method for producing and characterizing biodiesel from used cooking oils, an inexpensive feedstock.[6,7] I also studied the factors that influence the quality of biodiesel by using various analytical methods, including Nuclear Magnetic Resonance (NMR).[8] The quality of biodiesel obtained from various cooking oils was qualitatively compared with conventional diesel and identified by the best feedstock for producing the best biodiesel.[3,4,9] I started this

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project with the hypothesis that the biodiesel made from olive oil would be better than that produced from other feedstocks (frying oil, peanut oil, and canola oil) because it has more saturated hydrocarbon linkages. The structure resembles a regular diesel but contains an additional ester moiety vital to improving combustion.

Fuel	Renewable	Structural Difference	Origin	Limited/ Unlimited	Density/ Viscosity	Freezing Point	Energy Density (MJ/Kg)
Diesel	No	No Ester Hydrocarbon	Fossil	Limited	Relatively Low	Relatively Low	~43*
Bio-Diesel	Yes	Hydrocarbon With Ester	Renewable (Plant Matter)	Unlimited	Relatively High	Relatively High	~39*

* Data based on literature.

Figure 2: Key differences and similarities between diesel and biodiesel. This chart specifies different properties of diesel and biodiesel that are important to compare. The advantages of biodiesel are clear due to its renewability and limitless supply.

In this project, biodiesel was produced using a transesterification approach. Used vegetable oils comprise triglyceride molecules, a combination of fatty acids attached to a glycerol backbone. In the transesterification reaction of used oil, a triglyceride reacts with an alcohol in the presence of a catalyst (sodium hydroxide) to produce a biodiesel mixture and glycerol. I used four different oils that differ in unsaturated fatty acid content to produce different biodiesels that differ in unsaturation. Proton nuclear magnetic resonance (NMR) and density techniques have been used to evaluate the biodiesel quality.

Methods

All cooking oil materials used in this project were purchased from local grocery stores. Methanol and sodium hydroxide were from Sigma Aldrich.

For the creation of biodiesel, the following materials were used: canola oil (150 mL), used canola oil (150 mL), oil from a restaurant [highly used frying oil blend] (150 mL), peanut oil (150 mL), used peanut oil (150 mL), fresh olive oil (150 mL), used olive oil (150 mL), methanol, sodium hydroxide (NaOH), glass jars.

Transesterification:

In this project, I used a transesterification reaction (Figures 3, 4) in which methanol and the catalyst sodium hydroxide were used to perform transesterification on used and fresh cooking oils.[10,11] Oils contain triglyceride molecules, which are esters of three fatty acids linked to glycerol. When alcohol, such as methanol, is introduced and reacts with triglyceride in the presence of a base such as sodium hydroxide (catalyst), the fatty acids (saturated, unsaturated, and polyunsaturated) break away from the glycerin as methyl esters and become the biodiesel (Figures 4, 5).[12] The glycerol, NaOH, and other waste products solidify at the bottom, making the biodiesel easily accessible at the top. The key difference in the feedstocks used is the amount of oil saturation. Saturated fats and oils contain only single bonds, and upon transesterification, they yield biodiesels resembling diesel.[11] On the other hand, some of the oils used contain monounsaturated fatty acids that contain one double bond and polyunsaturated fatty acids that contain more double bonds between carbon atoms, which are not present in diesel.

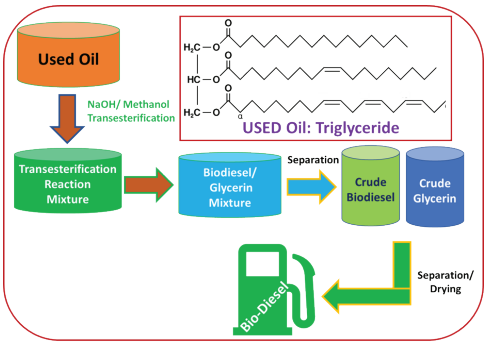

Figure 3: Experimental setup for producing biodiesel. The method of producing usable biodiesel from the feedstock of used oil is outlined. A chemical diagram of a molecule of crude oil is also shown. The black is the glycerol part, while the blue chains become the biodiesel.

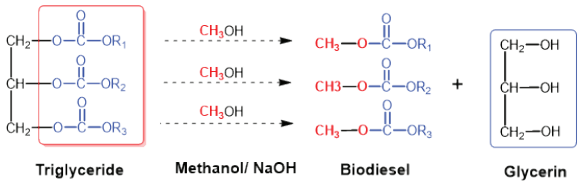

Figure 4: Illustration of the transesterification reaction. *R1, R2,* and *R3* are representatives of 3 different carbon chains. These figures illustrate the reaction that creates the biodiesel and glycerin molecules.

Procedure for Producing Biodiesel:

To the contaminant-free fresh or used cooking oil (150 mL) in a glass jar was added a freshly prepared clear solution comprising 33 mL of methanol and 1.05 g of NaOH. A clear solution of methanol and NaOH was obtained by continuous stirring using a magnetic stir bar. After the addition, the final mixture was vigorously shaken for 5 min to initiate the transesterification process. The resulting two-phase solution was allowed to sit for about 48 hours (2 days) to complete the transesterification reaction. The biodiesel (methyl esters) formed are on the top layer, and glycerin, a transesterification by-product, is at the bottom of the jar. This process was repeated the same way for all seven different feedstock cooking oils, ensuring biodiesel production from each.

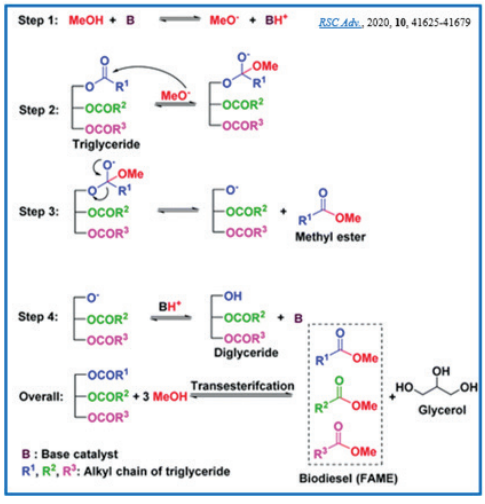

Figure 5: Detailed outline of the chemical reaction for obtaining biodiesel. This figure portrays the steps taken to convert the triglycerides from the oil into biodiesel.

Procedure for Conducting Flash Point and Carbon Residue Tests:

The open cup method determined the flash point and carbon residue test. For each Biofuel, one measurement of the flash point was made. In the case of biodiesel-derived canola oil, two measurements were made to validate the measurements. Biodiesel samples of known weight were placed in an evaporating crucible on a hot plate with a heating ramp rate of 10 ˚C/ min. The flame source was placed above the evaporating crucible; when the biodiesel reached the flash point, a flash occurred, called the flash temperature. The biodiesel was allowed to burn until it was entirely consumed. The weights of the evaporating crucible were compared before and after to determine the carbon residue.

Procedure for Oxidation Stability Tests:

Oxidation stability tests of biodiesels were conducted to test the stability of the biodiesels to air at room temperature and high temperature. The biodiesels were kept in the air for three days for room temperature oxidation stability tests. NMR analyzed the samples to compare ester protons to unsaturated protons to determine the number of double bonds that ultimately define the oxidation stability of biodiesels. For high-temperature oxidation stability tests, the biodiesels were heated at 75 ˚C for 2 hours, and samples were allowed to cool to room temperature. The methanol contaminant was also removed by heating the biodiesel. The samples were analyzed by NMR (Nuclear Magnetic Resonance) to compare ester protons to unsaturated protons.

Procedure for Conducting Copper Corrosion Test:

For the corrosion test, a specific number of strips of copper metal were placed in biodiesels and allowed to be in contact with them for seven days. The weight of the metal before and after it was put in the biodiesel was checked to see if there was any corrosion after seven days. If there is corrosion, metal oxide will form on the copper's surface, and the metal's weight will increase after corrosion.

Procedure for Conducting Viscosity Tests:

A Lovis 2000 ME Viscometer was used to measure the viscosity tests. The density of the biodiesel is used to measure kinematic viscosity. Kinematic viscosity is a measure of the resistance to flow of a fluid under the influence of gravity.

Procedure for Conducting NMR Test:

An Anasazi Instruments EFT-90 MHz NMR spectrometer was used to measure the NMR of all biodiesels. About 400 microliters of CDCl3 with TMS standard and 400 microliters of biodiesel were added to the NMR, and NMR measurements were obtained.

■ Results and Discussion

Risk and Safety:

For my experiments, I worked with various fresh and used oils. I also used methanol and sodium hydroxide as catalysts for the transesterification reaction. Proper protective equipment, like gloves, lab coats, and goggles, was used while experimenting. Many precautions were taken to prevent spills or mistakes by doing the reactions in a safe, secure space where spills can be cleaned up rapidly and not disturbed when stored. In the process, the mixing of the two solutions was done in a glass jar. One last potential hazard is the flammability of the substances being made and tested. The biodiesel flash point is high, and it was stored in safe locations to ensure that it would never reach a point of combustion when not desired. Carbon residue and flash point tests were conducted in a laboratory hood at Columbia Basin College (CBC), WA. All waste (used oil, biodiesel, glycerol, sodium hydroxide, and methanol) was disposed of appropriately.

Data Analysis:

The following feedstock (oil) parameters influence the overall quality of the biodiesel derived after transesterification. The parameters are:

1. Carbon chain length in fatty acids (8 to 22 carbons long)
2. Amount of unsaturation or the number of double bonds in fatty acids (saturated, monounsaturated, polyunsaturated)
3. Location of unsaturation in fatty acids
4. Simple triglycerides (made up of the same three fatty acids) vs. mixed triglycerides (made up of different fatty acids).

Because of these variations, the biodiesel derived from these diverse feedstocks can be very different from the actual structure of diesel. Some of these properties favor ignition characteristics, but some do not. Therefore, quantifying the combustive properties of these biodiesels remains a huge issue today.

Cetane numbers are currently considered the gold standard for quantifying the quality of fossil diesel (Figure 6).[13] The cetane number is measured using a scale that reflects auto-ignition properties. The cetane number scale is based on the auto-ignition characteristics of two reference fuels.[14-16] Hexadecane ($C_{16}H_{36}$) with spontaneous autoignition property is assigned a cetane rating of 100. Heptamethylnonane ($C_{16}H_{34}$) has a much lower autoignition property and is rated 15.

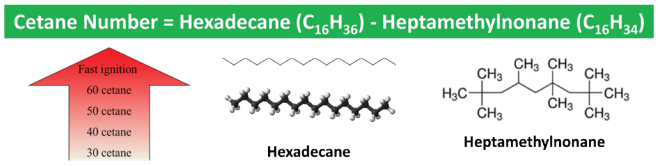

Figure 6: Illustration of Cetane number. The figure displays what the cetane number is and how it can be calculated. The higher the cetane number, the faster the ignition.

However, determining cetane numbers is an intense, challenging, and expensive process involving high-end equipment in highly controlled environments. More importantly, the cetane number heavily relies on the property of hexadecane, which is one of the ingredients in biodiesel. Because the amount and nature of fatty acids differ in different oils, the structural and functional properties of biodiesels derived from these feedstocks differ significantly from oil to oil or between batches (Figure 7).[17] Therefore, determining the cetane numbers may not be sufficient to quantify the quality of the

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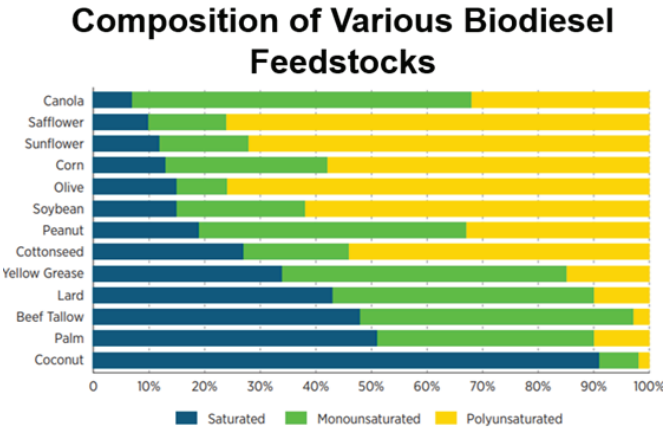
biodiesels derived from different feedstocks. Due to the vast structural and functional variation that occurs due to the nature of fatty acid characteristics of the feedstocks, it is hard to narrow down the quality based on one technique alone, such as the cetane number.



➢ **Biodiesel feedstock has significant differences in saturation to unsaturation.**

Figure 7: Composition of various biodiesel feedstocks. The figure outlines the different levels of saturation present in different feedstocks. The different proportions will affect the quality of the biodiesels.

In this science fair project, I attempted to utilize commonly available characterization methods and analytical tools to assess the quality of biodiesel qualitatively.[18] These methods and tools include Infrared Spectroscopy (IR), Nuclear Magnetic Resonance spectroscopy (NMR), density, freezing point, flash point, carbon residue, corrosion test, oxidation stability, and viscosity measurements (Figure 8).[17,18]

DOE Requirements for 100% Biodiesel (B100)

Property	ASTM Limits	Units
Flash Point	130	°C
Methanol	0.2	% Mass
Water	0.05	% Volume
Sulfur	0.02	% Mass
Cetane	47	
Carbon Residue	0.05	mg/g
Acids	0.8	% Mass
Glycerin	0.24	% Mass
Phosphorus	0.001	% Mass
Distillation Temperature	360	°C

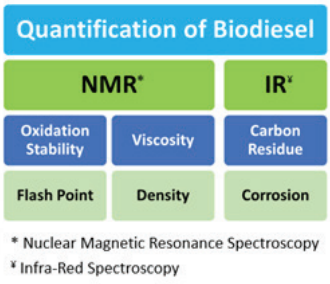


Figure 8: The requirements set by the US Department of Energy (DOE) for commercially usable Biodiesel. The table on the right specifies the exact tests that I ran.

NMR is an analytical chemistry technique used primarily in quality control and research to determine a sample's content and molecular structure. I utilized the NMR characterization to verify the spectral variations in the unsaturated and esters region of different synthesized biodiesels.[8,19] The NMR spectra have offered excellent information and guidance on the presence of saturation and unsaturation in biodiesel (Figure 9).[20] I also utilized Infrared Spectroscopy (IR) to understand the presence of unsaturation in biodiesels. The C- H and C=C stretching peaks in the IR of different biodiesels offered insights into unsaturation in biodiesel. On the other hand, regular diesel is entirely composed of saturated carbons, meaning the molecule has only single bonds (C-C) and no double bonds.

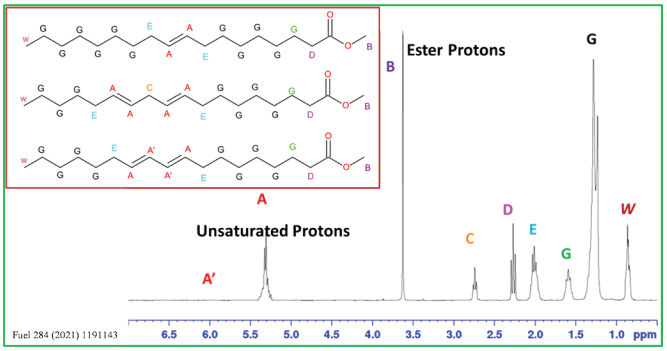


Figure 9: Sample proton NMR of sample biodiesel. These figures demonstrate how I analyzed the NMR of the biodiesels to determine their quality.

To compare the properties of biodiesels with regular diesel, I also performed density experiments that provided some insights into the flow properties. The effect of freezing temperatures on different biodiesels was also tested by keeping the samples outside overnight. The viscosity of cold biodiesel was tested to see if it could withstand the cold temperatures as much as diesel. Interestingly, the biodiesel derived from three different feedstocks has different freezing points and viscosity properties. However, regular diesel has a very low freezing point; it does not freeze under the same conditions.

I also investigated many other physical attributes, including the carbon residue of the biodiesel, to verify the safety qualifications and ensure the synthesized biodiesel meets the Department of Energy's (DOE's) specifications requirements for biodiesel. I conducted flash point testing to determine at what temperature the different biodiesels are ignitable in a controlled environment under expert supervision. This is important because flash points of biodiesel below room temperature will be more hazardous. I also conducted oxidation stability experiments on biodiesels because their double bonds oxidize due to their high reactivity with oxygen in the atmosphere. Additionally, I conducted viscosity measurements of seven biodiesels at room temperature and performed viscosity measurements of the best biodiesel at high temperatures. Viscosity measurements are critical because highly viscous fuels often produce large spray droplets on injection during combustion, resulting in poor performance. I also conducted corrosion resistance experiments on copper to determine the impact of biodiesel on metals. All the above-discussed tests have been performed to investigate whether biodiesel synthesized from used oils meets the requirements set by the DOE.

Findings and Analysis:

Proton NMR analysis was used to quantify the ratio of unsaturated protons to ester protons. Figure 9 illustrates the sample NMR of biodiesel, which provides insight into NMR analysis to understand what peaks represent different protons and carbons to analyze structural properties such as double bonds (unsaturation) and esters.

The amount of unsaturation from the proton NMR spectra can be used to evaluate the quality of different biodiesels (Figures 10, 11).[18] It is reported that biodiesels with mono-unsaturated fatty acids (MUFAs) and an ester in a 1:1 ratio generally offer higher cetane numbers.[21] This means only one double bond for every hydrocarbon chain with one ester. Since every triglyceride produces three separate hydrocarbon chains after the transesterification process, each with an ester (3 Protons), I used this information in my NMR analysis. I compared the ratio of ester to unsaturated protons to rank the biodiesel by quality.[21] Biodiesels with higher unsaturation tend to have better cold flow properties and faster ignition rates, which result in better engine performance.[8]

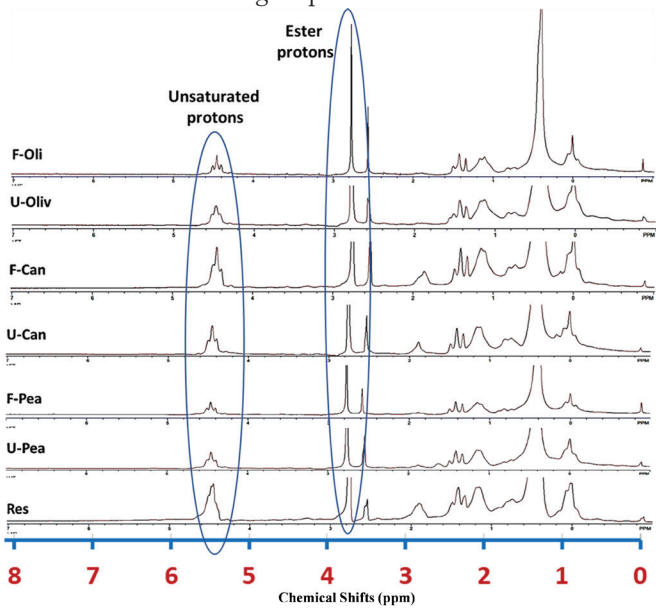


Figure 10: Stacked proton NMR of all freshly synthesized biodiesel samples. It provides a relative number of electrons with similar frequencies, which can be used to calculate the # of esters to the # of unsaturated protons ratio.

Biodiesel	Unsaturation Proton's	Ester Proton's	Ester/ Unsaturation	# of Unsaturated Protons*	# of Double Bonds*	Ranking of Bio-Diesel Quality
BD-F Canola	0.17	0.12	0.71	12.75	6.38	3
BD- U Canola	0.18	0.13	0.72	12.46	6.23	2
BD-F Olive	0.22	0.09	0.41	22.00	11.00	6
BD-U Olive	0.18	0.07	0.39	23.14	11.57	7
BD-F Peanut	9.32	4.92	0.53	17.05	8.52	5
BD- U Peanut	0.19	0.1	0.53	15.55	7.77	4
BD-U Restaurant	0.2	0.17	0.85	10.59	5.29	1

* In every 3 fatty acids, esters (biodiesel molecules) derived from a Triglyceride

Figure 11: Analysis of proton NMR of biodiesel samples. The table shows the calculations for understanding the saturation level of all the biodiesels tested. Biodiesel from used restaurant oil performed the best.

Oxidation stability tests were performed by exposing the samples to air for three days (Figures 12-14). I further tested the oxidation stability by heating the biodiesel at 75 ˚C for 2 hours.[22-24] NMR analysis was performed on all the samples to compare the ester proton to unsaturation proton ratio in the three conditions (fresh, air, heated). Biodiesel derived from highly used restaurant oil has the highest oxidation stability, showing the least change in all three conditions tested. The biodiesels derived from fresh and used olive oils have the least oxidation stability.

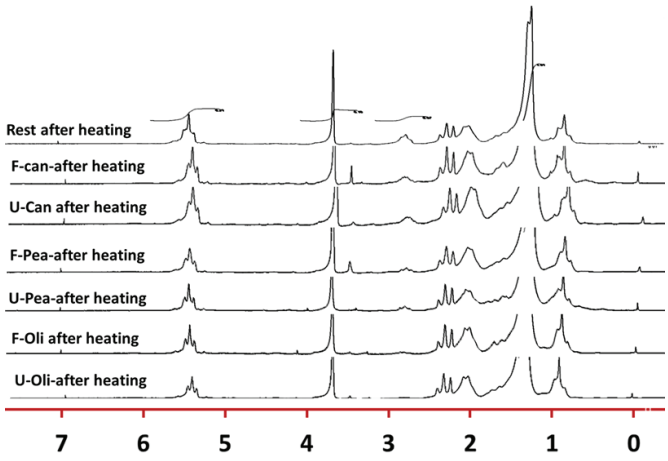


Figure 12: NMR spectra of all heat-treated biodiesel samples (oxidation stability). This can be used to determine which biodiesel is the most stable by comparing it with the NMRs of raw samples.

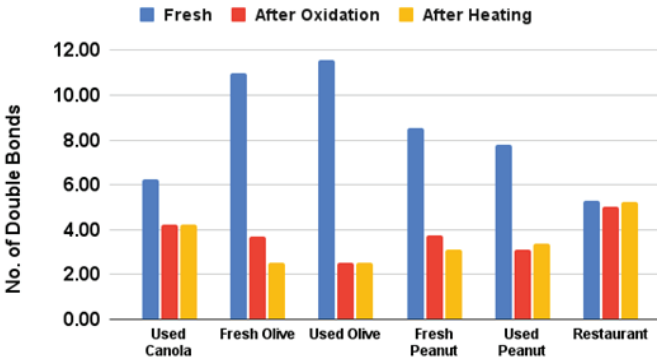


Figure 13: Oxidation stability of biodiesels. This graph proves that biodiesel from used restaurant oil is the most stable due to the least change from fresh to after oxidation and heating.

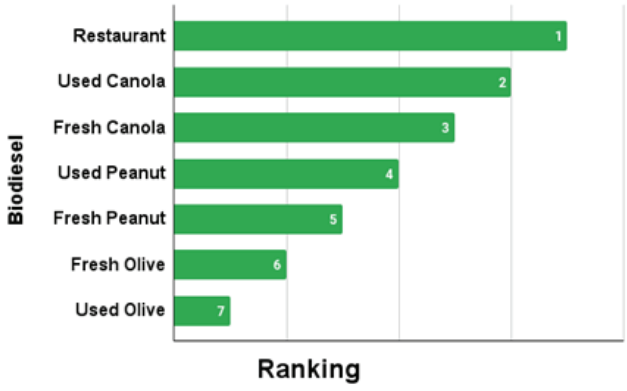


Figure 14: Ranking of produced biodiesels based on Oxidation stability.

The DOE recommended range for kinematic viscosity is 4-6 at 40 ˚C. Biodiesels synthesized in this project have DOE-specified kinematic viscosity between 4-6.[25, 26] Temperature affects viscosity. As the temperature increased, overall viscosity decreased. The viscosity difference between biodiesel and fossil diesel becomes much smaller at high temperatures. At 60 ˚C, the kinematic viscosity is 2.8 (Figures 15-17).

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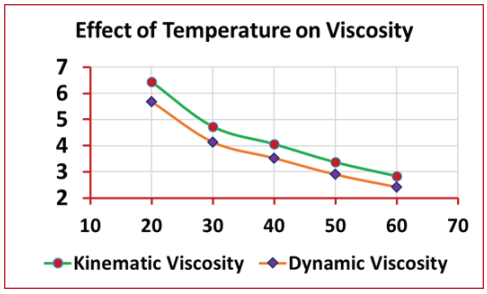


Figure 15: Temperature effect on biodiesel viscosity. This graph shows how the viscosity of biodiesels, on average, reduces as temperature increases.

Figure 16: Image of a viscometer, an instrument used to measure viscosity.

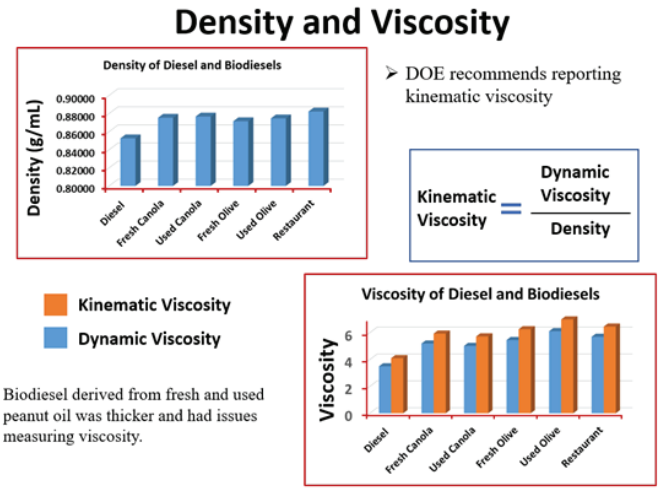
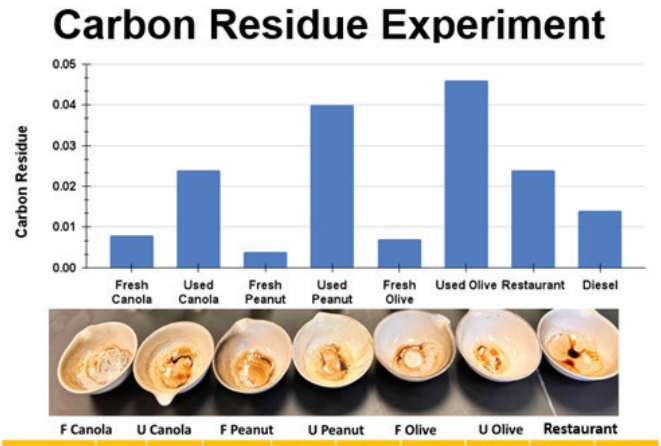


Figure 17: Density and viscosity measurements for the biodiesel samples compared to diesel.

The flash point is the lowest temperature at which the vapors of biodiesel will ignite. The flash point of biofuels is important for understanding safety-related fuel processing, transportation, and storage properties. Biodiesel derived from fresh olive oil has the lowest flash point. Biodiesel derived from highly used oil from restaurants has the highest flash point. The higher the flash point, the less flammable the liquid. The flash point of biodiesel derived from highly used restaurant oil is the highest (199.5 °C), and that of fresh olive oil is the lowest (133.2 °C) (Figure 18). Using an open cup method, the amount of carbon residue obtained for biodiesels aligns with what is expected for diesel (Figure 19).

Property	ASTM Limits	Units	Diesel	BD-F-Canola	BD-U-Canola	BD-F-Peanut	BD-U-Peanut	BD-F-Olive	BD-U-Olive	BD-R-Rest
Flash Point	130	°C	85	148.7	180.3	149.6	161.5	133.2	146.7	199.5

Figure 18: Flash point testing experimental setup and results. This shows the stability of the biodiesel and whether it meets the ASTM limit.



Property	Units	Diesel	BD-F-Canola	BD-U-Canola	BD-F-Peanut	BD-U-Peanut	BD-F-Olive	BD-U-Olive	BD-R-Rest
Carbon Residue	g	0.014	0.008	0.024	0.004	0.04	0.007	0.046	0.024

Figure 19: Carbon residue test results. This shows the visible results and the mass of the carbon residue after testing. This test is done to determine safety and waste consumption.

Conclusion

I used a transesterification reaction to produce seven different biodiesels from several fresh and used oils in this project. Feedstock oils are diverse and contain different amounts of unsaturation and hydrocarbon chain length. Due to this, the biodiesel derived from these various oils is different from the actual structure of diesel. Although some of these characteristics favor ignition properties, some do not, making it very challenging to quantify the combustion properties of biodiesels. Biodiesel contains oxygenated species (esters) and unsaturation, so the cetane number, currently considered the gold standard for quantifying the quality of fossil-derived diesel, cannot be directly used to quantify biodiesel quality. I used non-destructive methods such as Infrared Spectroscopy (IR), Nuclear Magnetic Resonance spectroscopy (NMR), density, and freezing measurements to assess the quality of biodiesel. From NMR data, I compared the ratios of ester protons (fixed at 9 per chain) to unsaturated protons (varying per chain) to determine the amount of unsaturation. Out of seven biodiesels produced in this project, biodiesel derived from highly used frying oil is closest to a monounsaturated fatty acid ester with a 1:1 ester to double bond ratio. Biodiesels with about one double bond (unsaturation) per chain tend to have better cold flow

properties and faster ignition rates, which results in better engine performance.

Biodiesel derived from highly used restaurant oil has the highest oxidation stability, as it showed the least change in all three conditions tested (freshly synthesized, oxidized by air, and heated in air). Biodiesels synthesized in this project have a kinematic viscosity in the range of 4–6 as specified by US-DOE. As the temperature increased, overall viscosity decreased, and the viscosity difference between biodiesel and fossil diesel became much smaller at high temperatures (at 60 °C). The flash point of biodiesel derived from highly used restaurant oil is the highest (199.5 °C), and fresh olive oil has the lowest (133.2°C). The amount of carbon residue obtained for biodiesels using an open cup method is in line with that of diesel.

My experimental data and analysis revealed that used frying oil from restaurants yielded the highest quality biodiesel. This is likely because restaurant frying oil has already undergone many cycles of heating, which can reduce the polyunsaturated bonds and increase the proportion of more stable monounsaturated fatty acids. As a result, the resulting biodiesel contains a better ester-to-unsaturation ratio, giving it higher oxidation stability, improved ignition properties, and overall making it have superior fuel quality. These results proved that my hypothesis that olive oil would be the best feedstock was wrong. NMR analysis offered insights into carbon structure and oxidation stability by using the number of double bonds per ester to quantify biodiesel quality. With further optimization, these tests and analysis methods can produce biodiesel that meets industry quality standards. My results show that using biodiesel derived from restaurant oil could be a viable alternative to diesel. This can have many environmental and economic benefits. Reducing the use of diesel is beneficial to the environment, and using waste cooking oils prevents waste build-up and expensive disposal processes. The conversion to biodiesel is much cheaper. Doing this can have a positive impact on the world.

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