Making Biodiesel from Virgin Vegetable Oil: Teacher Manual

Learning Goals:

- Students will understand how to produce biodiesel from virgin vegetable oil.
- Students will understand the effect of an exothermic reaction.
- Students will understand the distinction between reagents and products.

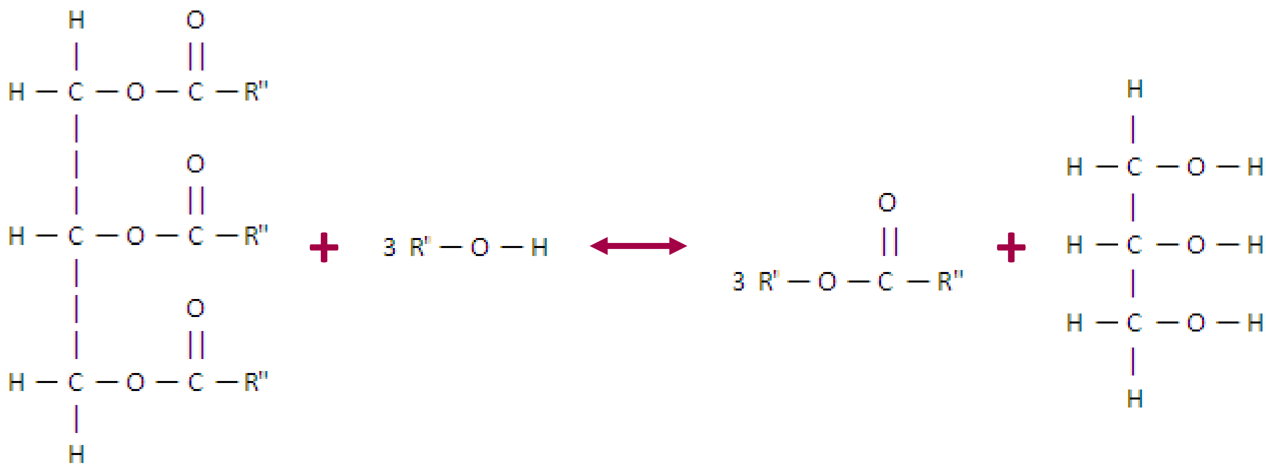
Objectives:

- Students will make biodiesel from virgin vegetable oil.
- Students will record actions, calculations, and observations in a laboratory notebook.

Extended Background:

Biodiesel is a cleaner burning renewable alternative to diesel fuel that is made from biological sources; namely vegetable oil or animal fats (triglycerides). It is mixable with diesel, stable in mixture, and can be burned in an unmodified diesel engine at any concentration.

Biodiesel is made through a **transesterification** reaction. Transesterification is the chemical process through which one *ester* (a chemical having the general structure R'COOR'') is changed into another. When the original ester is reacted with an alcohol, the process is called *alcoholysis***.** The LUC Biodiesel Laboratory makes biodiesel using vegetable oil (an ester compound) and methanol (an alcohol) as the reagents. Vegetable oil is a **triglyceride** (or triacylglycerol), which is essentially a **glycerin** (or glycerol) molecule connected via ester bonds to three **fatty acid** molecules. Vegetable oils and animal fats are composed of triglycerides. During the reaction, the fatty acids of the triglyceride molecule are cleaved and attach to the alkyl group (the part made of carbon and hydrogen) of the alcohol to form **fatty acid alkyl esters** (in our case, fatty acid **methyl** esters or *FAME*), which are biodiesel. In order to get the transesterification of vegetable oil going, at LUC we use a base **catalyst** (a substance that increases the rate of a chemical reaction but is not altered by the reaction). We use potassium hydroxide (KOH) as our base catalyst. This reaction is diagramed on the next page.



Vegetable Oil + Alcohol   Biodiesel + Glycerin

R''= *fatty acid chain of the triglyceride (this will vary for each type of vegetable oil)*

R'= *in the case of methanol, this will be methyl group (CH_3)*

Reagents		Catalyst		Products
Triglyceride (veggie oil) Alcohol (methanol)	→	KOH	→	Biodiesel (FAME) Glycerin

The transesterification reaction produces crude biodiesel. The product is considered crude because it is contaminated with methanol, basic salts, and glycerin. All of these contaminants are water soluble. Therefore, in the LUC Biodiesel Laboratory, we remove them using a water wash. The washing introduces water to the biodiesel, which decreases fuel quality and must be removed. In the LUC Biodiesel Laboratory, we bubble air through wet biodiesel to dry it. This process forces the evaporation or settling of water molecules remaining in the fuel and leaves you with a finished biodiesel fuel.

To prevent mistakes and misunderstandings and to maintain a record of any ingenious achievements, we suggest that all students take meticulous notes in a laboratory notebook.

Materials:

- Vegetable oil (200 mL per pair of students)
- 250 mL graduated cylinder
- Scale or balance
- Weigh boats
- Base (KOH) (≈ 2 g per pair of students)
- Small spatula
- Methanol (40 mL per pair of students)
- 50 mL graduated cylinder
- One mason jar per pair of students
- Large separatory funnel with ring stand
- Spray bottle with water

Preparation:

1) Set up stations for student pairs with ball jars and weigh boats.
2) Set up 1-2 stations for students to collect vegetable oil, lye, and methanol for their reactions.
3) Prepare crude biodiesel for washing (see below).

Procedure:

Calculating the amounts of reagents and catalysts required

The recipe for making biodiesel from virgin vegetable oil, using base as a catalyst, is simple. For every 1L of vegetable oil, add 0.2 L of methanol, and 8.5 g of KOH. In this lab, we will be starting with 200 mL of vegetable oil.

$$1\text{ L oil} + 0.2\text{ L methanol} + 8.5\text{ g KOH} \rightarrow 1\text{ L biodiesel} + 0.2\text{ L glycerin} + \text{soaps}$$

Have students calculate the amounts of methanol and KOH required:

200 ml vegetable oil   _____ ml methanol   _____ g KOH

(This is important information to include in the laboratory notebook. Always have students begin each entry with the batch number, name, present date and time.)

Laboratory safety

Caution: The methanol you will be working with is highly flammable and toxic and the base is caustic. Everyone should put on safety goggles and gloves. Check that you are wearing long pants and closed-toed shoes.

Making Potassium Methoxide: *Student Procedure*

1) Using a 50-mL graduated cylinder (located in the fume hood), measure out the correct volume of methanol under the fume hood. Pour the methanol into a mason jar. Seal the lid.
   a) If a fume hood is not available, place methanol in a well ventilated area.
2) Use the balance to weigh out the correct mass of lye. (Try to do this quickly. Base is hygroscopic, it will absorb water from the atmosphere after the lid is opened and change total mass).
3) Put the KOH into the mason jar, secure the lid again, and shake the jar until all of the KOH has dissolved (no particles should remain visible except sand sized particles). The reaction between the base and methanol is exothermic (it releases heat and pressure). Periodically take your jar to the fume hood and "burp it"—open the lid to release the pressure.

(Have students make descriptive notes in the laboratory notebook about this process. What did the solution look like? Did you feel heat coming off of the liquid? How long did it take to dissolve the lye? etc.)

Making crude biodiesel: *Student Procedure*

1) Using a graduated cylinder (located near the vegetable oil), measure 200 ml of vegetable oil.
2) Pour the vegetable oil into the jar with the potassium methoxide. Secure the lid.
3) Carefully and vigorously shake the mixture for at least 10 minutes.
4) Label the jar with your group name and the jar's contents and let sit.

You just made crude biodiesel! You are almost there!

(Again, have students make descriptive notes in the laboratory notebook. What did the solution look like? Did the color change through time? etc.)

What happens next?

You should begin to see a separation in the mixture you have created. The glycerin that was cleaved from the triglyceride is denser than the biodiesel and will settle to the bottom of your container. The biodiesel will float on top as in the image above.

Washing the biodiesel: *Teacher Procedure*

At this point the biodiesel you have made is crude because it contains residual base, glycerin, soap, and methanol. To remove these impurities which may negatively impact fuel performance, the crude biodiesel must be washed.

Because washing is a time intensive project, we suggest that you do a small demonstration at the front of the class for washing biodiesel. Using a large separatory funnel pour in crude biodiesel (a beaker may be substituted but a separatory funnel will show separation more clearly). This will provide a clear visualization of the separation that takes place between the crude biodiesel and crude glycerin. Allow sufficient time to separate (~30 min.). Once there has been a noticeable separation, drain the crude glycerin from the biodiesel.

Using a spray bottle filled with water, gently spray water onto the surface of the biodiesel in the separatory funnel. You should notice an immediate separation as the water moves to the bottom of the funnel. Because of the polarity of water molecules, they will pull the residual catalyst, glycerin, soap and methanol from your crude biodiesel, and leave you with a purer biodiesel. If the force of the water entering is too great, it may hydrolyze free fatty acids, which will combine with the base to form soap.

However, it should be noted that the biodiesel created in the ball jars will not be as pure as the biodiesel made on a larger scale because this process occurs at room temperature, uses only one reaction, and does not include a titration of the oil. Subsequently, there is a greater chance that this biodiesel will contain unwanted substances in suspension, despite washing it.

Note for procedure*:* If you have the time and resources, it will be beneficial to have biodiesel in the separatory funnel before the lab begins.

Clean Up

- Clean all glassware and bench space.
- Biodiesel can be used in candles or tiki torches (not suitable for use in an engine)
- **Glycerin contains excess methanol**. This can be boiled off under a fume hood (not a student activity) and the methanol-free glycerin can be used to make soap (See "Soap Lab")
- Wash water can be sent down the drain

Student Questions:

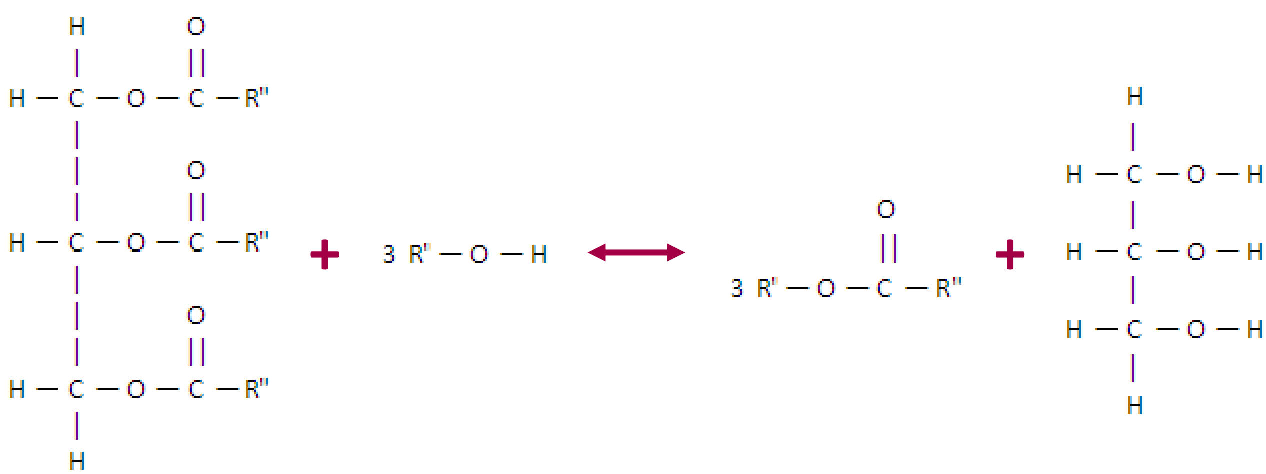
1) In this laboratory, what were the reagents and what were the products? What did you use as a catalyst? Why did you use a catalyst?
    - ***Reagents:*** *vegetable oil and alcohol (methanol)*
    - ***Products:*** *biodiesel and glycerin*
    - ***Catalyst:*** *either sodium hydroxide (NaOH) or potassium hydroxide (KOH) depending upon your procedure*
        - *A catalyst lowers the activation energy of the reaction by bonding with the methanol. It causes the reaction to move forward.*
2) What did you observe as you mixed the vegetable oil and the methoxide? Why do you think this happened?
    - *The students should observe a slight color change in the solution of methoxide and vegetable oil as the two begin to react with each other. As the reaction occurs the contents of the ball jar will change to a milky white.*
3) Describe what happened to the vegetable oil after the reaction. What did you observe in your jar?
    - *After the reaction has finished and the ball jar content has returned to being a gold-oil coloring rather than white, the contents of the jar should begin to separate out into crude biodiesel and glycerin.*
4) Why do the crude biodiesel and crude glycerin separate?
    - *The crude biodiesel and the glycerin should separate when let sit due to the differences in density. The glycerin will settle to the bottom and the biodiesel will sit on top.*
5) How did the pH of the water change after washing the biodiesel? Why did the pH gradually change after each successive washing?
    - *The pH should lower after each watching and after each washing more of the residual base glycerin and methanol will be removed from the biodiesel bringing the pH closer to seven.*

Making Biodiesel from Virgin Vegetable Oil: Student Lab

Background

Biodiesel is a cleaner burning renewable alternative to diesel fuel that is made from biological sources; namely vegetable oil or animal fats. It is mixable with diesel, stable in mixture, and can be burned in an unmodified diesel engine at any concentration.

Biodiesel is made through a *transesterification* reaction. Transesterification is the chemical process through which one ester is changed into another. The LUC Biodiesel Laboratory makes biodiesel using vegetable oil (an ester compound) and methanol (an alcohol) as the reagents. In order to get the transesterification of vegetable oil going, at LUC we use a base catalyst (a substance that increases the rate of a chemical reaction but is not altered by the reaction). We use potassium hydroxide (KOH) as our base catalyst. This reaction is diagramed on the next page.



Vegetable Oil + Alcohol Biodiesel + Glycerin

R''= *fatty acid chain of the triglyceride (this will vary for each type of vegetable oil)*

R'= *in the case of methanol, this will be methyl group (CH_3)*

Reagents		Catalyst		Products
Vegetable Oil Methanol	→	Base (KOH)	→	Biodiesel Glycerin

The transesterification reaction produces crude biodiesel. The product is considered crude because it is contaminated with methanol, base, glycerin, and soap.

To prevent mistakes and misunderstandings and to maintain a record of any ingenious achievements, we suggest that all students take meticulous notes in a laboratory notebook.

Materials:

- 250mL graduated cylinder
- 500mL graduated cylinder
- Scale or balance
- Base (KOH)
- For each pair of students:
- One mason jar
- 200 mL Vegetable oil
- ≈ 2 g Base (KOH)
- 40 mL Methanol

(In your Laboratory notebook always begin each entry with the batch number, your name, present date and time)

Laboratory safety

Caution: The methanol you will be working with is highly flammable and toxic and the base is caustic. Everyone should put on safety goggles and gloves. Check that you are wearing long pants and closed-toed shoes. Make sure your hair is pulled back away from your face and secured in a hair tie.

Preparation:

Before the procedure, the amount of reagents and catalysts needs to be calculated. The recipe for making biodiesel from virgin vegetable oil, using base as a catalyst, is simple. For every 1L of vegetable oil, add 0.2 L of methanol and 8.5 g of KOH. In this lab, we will be starting with a 200 mL of vegetable oil.

$$1\ \text{L oil} + 0.2\ \text{L methanol} + 8.5\ \text{g KOH} \rightarrow 1\ \text{L biodiesel} + 0.2\ \text{L glycerin} + \text{soaps}$$

(Use this space or your Lab notebook to calculate the amount of methanol and catalyst needed for the reaction)

200 ml vegetable oil   _____ ml methanol   _____ g KOH

Procedure:

Make potassium methoxide to be used as a reagent in your reaction. Potassium methoxide is the result of adding your catalyst (KOH) to the methanol. Remember to read through all instructions before carrying out the procedure.

Make descriptive notes in your Lab notebook about this process. Some questions to keep in mind are: What did the solution look like? Did you feel heat coming off of the liquid? How long did it take to dissolve the lye? Feel free to answer these questions in your lab notebook.

Using a 50 mL graduated cylinder (located in the fume hood), measure out the correct volume of methanol under the fume hood. Pour the methanol into a mason jar. Seal the lid.

Use the balance to weigh out the correct mass of lye. (Try to do this quickly. Base is hygroscopic, it will absorb water from the atmosphere after the lid is opened and change the total mass).

Put the base into the mason jar, secure the lid again, and shake the jar until all of the base has dissolved (no particles should remain visible except sand sized particles). The reaction between the base and methanol is exothermic (it releases heat and pressure).

Next, make crude biodiesel

Continue to make descriptive notes in your Lab notebook. Some questions to answer: What did the solution look like? Did the color change through time?

Using a 250 mL graduated cylinder (located near the vegetable oil), measure 200 ml of vegetable oil.

- Pour the vegetable oil into the jar with the potassium methoxide. Secure the lid.
- Carefully and vigorously shake the mixture for at least 10 minutes.
- Label the jar with your group name and the jar's contents and let sit.

You just made crude biodiesel!

Crude biodiesel

Crude glycerin

What happens next?

You should begin to see a separation in the mixture you have created. The glycerin that was cleaved from the triglyceride is denser than the biodiesel and will settle to the bottom of your container. The biodiesel will float on top as in the image left.

Clean up

- Wash your hands after the experiment.
- Clean all glassware and bench space you used.
- Put safety goggles in its proper place.

Questions:

1. In this laboratory, what were the reagents and what were the products? What did you use as a catalyst? Why did you use a catalyst?

2. What did you observe as you mixed the vegetable oil and the methoxide? Why do you think this happened?

3. Describe what happened to the vegetable oil after the reaction. What did you observe in your jar?

4. Why do the crude biodiesel and crude glycerin separate?

5. How did the pH of the water change after washing the biodiesel? Why did the pH gradually change after each successive washing?