Institute of Environmental Sustainability

Biodiesel Labs

Teacher Manual with Student Documents

Loyola University of Chicago
Institute of Environmental Sustainability
Biodiesel Program

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Welcome.

This series of labs focuses on building an environmental sustainability component into the high school classroom. In the following pages you will find both teacher and student documents for labs that focus on biodiesel production, testing, and use.

Our goal is to provide a comprehensive collection of activities that teach scientific principles through the lens of alternative fuel production. We continue to develop new labs, lessons, and supplies in the Searle Biodiesel Lab, but we also encourage teachers to submit their ideas and materials to us as well. Please help us to build a library of biodiesel, alternative energy, and environmental sustainability labs and lessons.

Beyond this collection, the Biodiesel Program also offers support to schools interested in going beyond simple labs. We offer guest speakers (Skype), tours of our lab (in person or Skype), Q&A sessions (Skype), and biodiesel production equipment loans to schools in the Chicagoland area. Please visit our website to learn more about the resources available through our program and feel free to contact us with any questions, concerns, or ideas: www.luc.edu/biodiesel.

I would like to extend thanks to the Environmental Protection Agency for funding this project in its start-up and the Institute of Environmental Sustainability at Loyola University Chicago for their support. We would also like to extend our thanks to you, the teachers. Your commitment to teach about the environment and sustainable practices is leading us to a brighter future.

Sincerely,

Zach Waickman
Biodiesel Lab Manager
Institute of Environmental Sustainability
Loyola University Chicago
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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 alcoholyis. 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 \[ R'' = \text{fatty acid chain of the triglyceride (this will vary for each type of vegetable oil)} \]
\[ R' = \text{in the case of methanol, this will be methyl group (CH}_3 \text{)} \]
Biodiesel + Glycerin

<table>
<thead>
<tr>
<th>Reagents</th>
<th>Catalyst</th>
<th>Products</th>
</tr>
</thead>
<tbody>
<tr>
<td>Triglyceride (veggie oil)</td>
<td>KOH</td>
<td>Biodiesel (FAME)</td>
</tr>
<tr>
<td>Alcohol (methanol)</td>
<td></td>
<td>Glycerin</td>
</tr>
</tbody>
</table>

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:

\[
\text{200 mL vegetable oil} \quad ____ \text{ ml methanol} \quad ____ \text{ g KOH}
\]

(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 washing 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.

\[
\begin{align*}
\text{Vegetable Oil} & \quad + \quad \text{Alcohol} & \quad \text{Biodiesel} & \quad + \quad \text{Glycerin} \\
R'' & = \text{fatty acid chain of the triglyceride (this will vary for each type of vegetable oil)} & \quad R' & = \text{in the case of methanol, this will be methyl group (CH}_3) \\
\end{align*}
\]

<table>
<thead>
<tr>
<th>Reagents</th>
<th>Catalyst</th>
<th>Products</th>
</tr>
</thead>
<tbody>
<tr>
<td>Vegetable Oil</td>
<td>Base (KOH)</td>
<td>Biodiesel</td>
</tr>
<tr>
<td>Methanol</td>
<td></td>
<td>Glycerin</td>
</tr>
</tbody>
</table>

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
  - \( \approx 2 \) 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)

\[
200 \text{ ml vegetable oil} \quad _____ \text{ ml methanol} \quad _____ \text{ g KOH}
\]

**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!

**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?
Waste Vegetable Oil and Titrations: Teacher Manual

Goals
- Understand how the frying process alters the composition of vegetable oil.
- Understand why titration is performed on WVO before introducing it to the transesterification reaction.
- Understand how to perform a titration.
- Understand why additional base is added to the transesterification reaction.

Objectives
- Students will perform multiple titrations on samples of WVO.
- Students will use their results to calculate the amount of additional base needed to neutralize FFAs in the WVO.
- Students will product biodiesel from WVO.
- Students may be involved in the WVO collection and pre-treatment processes.

Materials
- Waste vegetable oil (WVO)
- Large container for heating oil
- Stove burner or hot plate
- Sock filter(s)
- Scale(s)
- KOH
- Weigh boats
- Distilled water
- 1.5 L beaker
- Funnel

For each student group:
- 1 burette
- 3 small (50 ml) flasks
- 1 graduated pipette
- Phenolphthalein pH Indicator
- 60-mL Isopropyl alcohol
- 10-ml graduated cylinder
- Small funnel

Preparation and procedures
The process of converting waste vegetable oil (WVO) to biodiesel is essentially the same as that of converting virgin vegetable oil, but working with WVO requires a few extra steps:

1. Collection
2. Filtration
3. Heating and settling
4. Titration
5. Calculating the amount of additional base catalyst
Collection
To convert WVO to biodiesel, oil must first be collected from a deep fryer. The first step in collecting WVO is to establish a relationship with your provider. If you have fryers at your school, it should be easy enough to collect enough oil for a classroom experiment. We encourage students to be involved in this process.

The Biodiesel Program currently collects WVO from cafeterias on campus, major universities in Chicago, all of the museums in Chicago, and from local restaurants. Kitchens often change fryer oil on a regular schedule, so you should be aware of this as you prepare the lesson. A system or protocol for pick-up may need to be established. We leave collection pails with the cafeterias on campus, and our restaurant providers typically return the fryer oil to its original vessel once it has cooled. **Caution: Never collect hot oil.**

Filtration
The frying process often introduces food particles to the oil, and the oil must be filtered before undergoing the transesterification reaction. We typically pre-filter the oil through a 100 micron sock filter. Old t-shirts make a good, quick substitute for sock filters. The filtration process should be performed in advance of a transesterification reaction, and we encourage the involvement of students in this process.

Heating and Settling (Optional)
In addition to food particles, foods introduce water to the fryer oil. We pre-heat our WVO to 70°C which allows water (and additional food particles) to settle to the bottom of the vessel. Excess water and solid material will settle on the bottom of the oil container. Pre-heating should be started at least one day before a transesterification reaction.

Titration
WVO is typically more acidic than virgin vegetable oil. When foods containing water are fried in hot oil, some of the water reacts with triglyceride molecules to form free fatty acids (FFAs) (Fig 1). FFAs are fatty acid molecules that are not bound to glycerin. These acids react with the base catalyst to form soap (Fig 2), effectively leaving less catalyst available for the transesterification of triglycerides to biodiesel. The result is a less complete transesterification reaction.
FFAs, however, can be neutralized simply by adding additional base catalyst so that they don’t interfere with the transesterification reaction. In this case, FFAs are intentionally converted to soap. (Subsequently, this method is impractical for oils with very high FFA content.) To determine the amount of additional catalyst, one must determine the acid content of the WVO. This can be accomplished by titration.

Titration allows one to determine the concentration of acid in a known volume of WVO by neutralizing it with a reference solution (or titrant) of known base concentration in the presence of a pH indicator. The following titration procedure has been popularized among biodiesel homebrewers for its ease of use. Conveniently, the number of milliliters of reference solution needed to neutralize the analyte corresponds directly with the grams of additional base needed per liter to neutralize the FFAs in the WVO. The process is as follows:

**Preparing the reference solution**

This basic solution of known concentration will allow determination of the acid concentration of the WVO in the analyte. This recipe prepares a 0.1% KOH solution. The reference solution can be prepared either in advance by the teacher or during the lab by the students.

1. Dissolve 1 g of KOH in 1 L of distilled water (or tap water if purified water is not available).
2. Pour the reference solution into a burette.

**Preparing the analyte: Student Procedure**

1. Using a graduated cylinder, measure 20 ml of isopropyl alcohol.
2. Pour the isopropyl alcohol into a 50 ml flask.
3. Add 2-3 drops of phenolphthalein solution to the alcohol.
4. Swirl to mix.
5. Add 1 ml of WVO. Swirl until thoroughly mixed (you should start to see the oil bubbles disassociate and begin to mix with the isopropyl).
6. Repeat steps 1-5 two more times. You will need three separate flasks of analyte.

**Titration procedure: Student Procedure**
1. Place a flask under the burette containing reference solution.
2. Record the initial volume of reference solution in the burette.
3. Slowly add reference solution (one drop at a time) to the analyte solution.
4. Swirl the beaker between each addition.
5. Continue to add reference solution to the oil/alcohol solution until it turns pink and stays pink for ~30 seconds while swirling.
6. Stop.
7. Record the quantity of reference solution used, where \( V_0 \) is initial volume, \( V_f \) is final volume, and \( T \) is total volume of reference solution:
   \[
   T = V_0 - V_f
   \]
8. Repeat titration procedures for each flask.
9. Calculate the average value for \( T (\bar{T}) \) from the three trials.

**Calculating the amount of additional base catalyst: Teacher Procedure**
1. Either assign a volume of WVO for reactions by students, or have them determine the quantity of WVO appropriate for their jar (remember that you will also need to add methanol and lye).
2. Have students determine the quantities of base and methanol needed to react with your WVO.

<table>
<thead>
<tr>
<th>Biodiesel Ingredients and Proportions</th>
</tr>
</thead>
<tbody>
<tr>
<td>Using Waste Vegetable Oil</td>
</tr>
<tr>
<td>____ L feedstock oil (( \text{Hint: } 1 \text{ gal} = 3.79 \text{ L} ))</td>
</tr>
<tr>
<td>____ mL methanol = 0.2 * ( x ) L of oil * 1000</td>
</tr>
<tr>
<td>____ g KOH = (8.5 g KOH + ( T )) * ( x ) L of oil</td>
</tr>
</tbody>
</table>

**Making Biodiesel from WVO**
At this point, students can proceed with the transesterification reaction as outlined in the Biodiesel Program “Making Biodiesel from Virgin Vegetable Oil” lab activity with the addition of the extra base that they have calculated.
Questions:
1) If the WVO were to contain a higher number of free fatty acids, how would that affect the amount of base used in the reaction? Why?

➢ *It would require the addition of more base to neutralize the acids. If additional base is not added, the conversion of FFAs to soap will make less base catalyst available to the transesterification reaction, and an incomplete conversion of vegetable oil to biodiesel will result.*

2) Why is it important to perform the titration three times and use the average $T$ value?

➢ *Repeating trials and using an average value minimizes the chances of experimental error in determining the $T$ value.*

3) Why is it more important to perform titration when using WVO than when using virgin vegetable oil?

➢ *WVO is more likely to have a high FFA content, due to the application of high heat and the introduction of water, which causes the hydrolysis of triglycerides to FFAs.*

References:
Waste Vegetable Oil and Titrations: Student Lab

Ever been waiting behind a bus, bus driver hits the gas, and all of a sudden you’re hungry for French fries? Well, if that hasn’t happened to you yet, it will. We’re going to turn this French fry grease into fuel! Waste to energy! Unfortunately, as you might notice, used fryer oil is often pretty cruddy. That’s why it’s getting thrown away. Fortunately, with a few steps, we can make waste vegetable oil (WVO) suitable for conversion to biodiesel fuel.

**Background:**
First, we have to get this crud out of the WVO. Much of this crud is food particles left in the oil. Technically, these food particles are called chunkles®, and by technically we mean we made up that word. How are we going to get the chunkles® out of there? You got it – a chunkle® filter! The Biodiesel Program uses a 100 micron chunkle® filter to filter our oil before processing it. No filter? No problem, just use an old t-shirt!

Second, most food stuff contains water. When food is fried, it introduces water to the oil. We can separate most of the water from the oil by gently heating the oil to about 70°C, which causes the water to settle to the bottom. Now why does that happen? And once it’s there how do we remove it from the oil? These are the kinds of problems faced by people trying to save the planet.

Additionally, the chemical composition of vegetable oil changes when it is used in a deep fryer. The combination of water and heat causes the vegetable oil (triglyceride) molecules to break apart and form fragments called free fatty acids (FFAs). FFAs will react with the base catalyst to form soap. Soap! When this reaction takes place, it uses up catalyst that was intended to convert triglycerides into biodiesel, and we don’t get a complete conversion of WVO to biodiesel. So, we have to add more than 8.5 g of KOH per liter of oil to neutralize the extra acid in WVO. This will turn the FFAs into soaps, and still leave plenty of catalyst for the transesterification reaction.

But how much additional KOH do we have to add? (Another problem faced by somebody trying to save the planet.) Fortunately, chemists have devised a simple technique to address this problem. It’s called *titration*. In our case, titration can be used to determine the amount of extra base required to neutralize the FFAs. Titration is based on the idea that one molecule of base will neutralize one molecule of acid.

So, if we know the amount of base required to neutralize a sample of oil, we should know the amount of acid in the oil. Additionally, if we know how much basic *reference solution* (or *titrant*) was required to neutralize the solution (or *analyte*) that contains the oil, we should know how much additional base catalyst we need to add to our reaction to neutralize the FFAs in the oil. In this titration, the milliliters of
reference solution you use will correspond directly to the additional grams of base catalyst you need to add to the reaction.

When we do our titrations, we need to know when all of the acid in the oil has been neutralized. We can use a pH indicator to help us determine when this has happened. In this lab, we’ll use a pH indicator called phenolphthalein, which turns pink in a basic solution.

When performing a titration, you will drip the reference solution into the analyte until the color of the solution just begins to turn pink and remains pink after gently swirling for about 30 seconds. You will then record the amount of reference solution added. When making biodiesel from WVO, the Biodiesel Program uses the following recipe:

**For every 1 L feedstock oil (WVO) use:**

\[
0.2 \text{ L methanol (CH}_3\text{OH)}
\]

\[
8.5 \text{ g KOH } + \bar{T}
\]

(where \(\bar{T}\) = average milliliters of reference solution used in titration to neutralize the analyte)

---

**Laboratory safety**

Put on safety goggles and gloves.

**Procedure:**

**Preparing the reference solution**

This basic solution of known concentration will allow determination of the acid concentration of the WVO in the analyte. This recipe prepares a 0.1% KOH (or NaOH) solution.

1. Dissolve 1 g of KOH in 1 L of distilled water or isopropyl alcohol.
2. Pour the reference solution into a burette.

**Preparing the analyte**

1. Using a graduated cylinder, measure 20 ml of isopropyl alcohol.
2. Pour the isopropyl alcohol into a 50 ml flask.
3. Add 2-3 drops of phenolphthalein solution to the alcohol.
4. Swirl to mix.
5. Add 1 ml of WVO. Swirl until thoroughly mixed (you should start to see the oil bubbles disassociate and begin to mix with the isopropyl).
6. Repeat steps 1-5 two more times. You will need three separate flasks of analyte.

**Titration procedure**

1. Place a flask under the burette containing reference solution.
2. Record the initial volume of reference solution in the burette.
3. Slowly add reference solution (one drop at a time) to the analyte solution.
4. Swirl the beaker between each addition.
5. Continue to add reference solution to the oil/alcohol solution until it turns pink and stays pink for ~30 seconds while swirling.
6. Stop.
7. Record the quantity of reference solution used, where $V_0$ is initial volume, $V_f$ is final volume, and $T$ is total volume of reference solution:
   \[ T = V_0 - V_f \]
8. Repeat titration procedures for each flask.
9. Calculate the average value for $\bar{T}$ from the three trials.

Calculating the amount of additional base catalyst

<table>
<thead>
<tr>
<th>Biodiesel Ingredients and Proportions</th>
</tr>
</thead>
<tbody>
<tr>
<td>Using Waste Vegetable Oil</td>
</tr>
<tr>
<td>____ L feedstock oil (Hint: 1 gal = 3.79 L)</td>
</tr>
<tr>
<td>____ mL methanol = 0.2 * x L of oil * 1000</td>
</tr>
<tr>
<td>____ g KOH = (8.5 g KOH + $\bar{T}$) * x L of oil</td>
</tr>
</tbody>
</table>

Making Biodiesel from WVO
You are now prepared to make biodiesel from waste vegetable oil! Proceed as you would with virgin vegetable oil, but be sure to add the extra base catalyst that you’ve calculated based on your titration results.

Questions:
1). If the WVO were to contain a higher number of free fatty acids, how would that affect the amount of base used in the reaction?

2) Why is it important to perform the titration three times and take the average $T$ value?

3) Why is it more important to perform titration when using WVO than when using virgin vegetable oil?
Viscosity Lab Procedure: Teacher Manual

Goals:
- Understand what viscosity is.
- Understand what factors govern viscosity.
- Understand the importance of viscosity in relation to a fuel system.
- Understand how to change the viscosity of fluids.

Objectives:
- Students will test the viscosities of fluids through experimentation.
- Students will collect and record data from their experiments.
- Students will analyze the data by graphing it.
- Students will express their understanding of viscosity and their experiments by answering questions.

Background

Diesel engines
Diesel engines convert chemical potential energy in the form of fuel into mechanical kinetic energy in the form of moving parts. This is achieved through the combustion of fuel into heat, light, and gas in a combustion chamber called a cylinder. When the fuel explodes inside of the cylinder, it forces a piston outward producing mechanical motion. The linear motion of the piston is then converted to rotational motion via a crankshaft. This rotational motion ultimately turns the wheels of a vehicle.

The uniqueness of the diesel engine derives from the fact that compression alone within the cylinder creates sufficient heat to ignite the fuel. This characteristic distinguishes the diesel engine from the gasoline engine, which relies on a spark for the ignition of fuel within the chamber. The high compression of diesel engines causes them to be about 15% more efficient on average than gasoline engines.

Due to their high combustion temperatures, diesel engines have always had the capacity to utilize a variety of fuels. In 1909, a diesel engine fueled by peanut oil was demonstrated at the World Fair in Paris. Using vegetable oil-based fuels mitigates many of the negative impacts associated with fossil fuels including greenhouse gas emissions and dependency on imported oil.

During the early 20th Century, however, as petroleum became less expensive and more available, engineers increasingly designed automobiles to use only petroleum-based fuels. Almost all diesel fuel systems built in the last one hundred years were designed to optimize the properties of petroleum diesel. For an alternative fuel to function effectively in an unmodified diesel automobile, it must
duplicate the properties of petroleum diesel. The major physical property affecting the use of vegetable oil in unmodified diesel engines and fuel systems is viscosity.

**Viscosity**

Viscosity is the resistance of a fluid to flow. It is often referred to as “fluid friction” and commonly thought of as the “thickness” of a fluid. A fluid like honey that is very thick has a high viscosity, and a fluid like water that is relatively thin has a low viscosity. Viscosity is commonly measured in units called Pascal seconds (Pa • s).

In an engine, fuel is delivered to the cylinders via a fuel system. The major components of the fuel system include the fuel tank, fuel lines, the fuel pump, the fuel filter, and fuel injectors. When you pump gas into a vehicle, it enters the fuel tank. Fuel is then pumped out of the tank when you drive, through fuel lines and through the fuel filter to fuel injectors, which inject a fine spray of fuel into the cylinders at exactly the right moment. The fuel then explodes. The components of the fuel system are designed to distribute a certain amount of fuel at a certain rate, which is affected by fuel viscosity.

Viscosity is governed by a combination of three major factors:

- **Intermolecular forces**
  - The stronger the bonds between molecules, the more viscous the fluid.
- **Molecular size**
  - Smaller molecules flow past one another more easily than larger molecules.
- **Molecular shape**
  - This property can be tricky. Sometimes, linear molecules flow more easily past each other than branched molecules. On the other hand, sometimes linear molecules can more easily stack on top of one another than branched molecules, which can increase the intermolecular bonding between linear molecules.

Vegetable oil is typically ten times more viscous than petroleum diesel. Using vegetable oil in an unmodified diesel fuel system would be a lot like shooting vegetable oil through a squirt gun. It won’t work very well. Furthermore, it will cause damage to the engine and fuel system. In order to utilize a vegetable oil-based fuel in an unmodified diesel fuel system, the vegetable oil itself must be modified to acquire a viscosity very close to that of petroleum diesel.

**Teacher Preparation**

In this lab, students will compare the viscosities of vegetable oils and biodiesel at different temperatures. We have suggested WVO, biodiesel, corn oil, and soy oil, but we encourage you to be creative. We advise having pairs of students or small groups conduct trials on only one of the fluids and then comparing the results as a class. It is a good idea to prepare a demonstration in the front of the classroom to show the viscosity of all of the different fluids, in case the students didn’t get to see them.
all during the lab period. Make available at each student work bench each of the items in the Materials list below.

1) Prepare lab stations for each student group with the items listed in the Materials list below.

2) Give one of the samples to each group in a container – note the groups will only perform trials on one of the samples. At the end of the class gather all of the data and share.

3) At the end of class if you wish to calculate viscosity use the equation:

\[ n = \frac{2(\Delta p)g r^2}{9v} \]

With this equation \( n = \text{Viscosity in Pa}\text{s} \)

\( \Delta p = \text{the change of the density of the fluid and the sphere} \)

\( g = \text{acceleration of gravity (9.8 m/s}^2) \)

\( r = \text{radius of the sphere in meters} \)

\( v = \text{velocity of the sphere’s descent through the liquid in m/s} \)

**Materials:**

- 1000 mL (glass) graduated cylinder
- 1000 mL sample of one of the following
  - waste vegetable oil
  - biodiesel
  - corn oil
  - soy oil
- Hot plate
- Thermometer
- 1000 mL Erlenmeyer flasks
- Teflon balls (5/16” diameter) Funnel
- Steel wool
- Stopwatch
- Stirring Rod
- Wax pencil

**Safety Precautions:**
Wear gloves and goggles at all times. When you are handling hot oils wear heat resistant gloves.

**Student Procedure:**

1. Fill the graduated cylinder with your liquid sample to the 1000 mL mark.
2. Draw two lines on your column with the wax pencil, one near the top of the oil and one near the bottom.
3. Measure the distance between the two lines in meters and record the length below. You will need this measurement later for your data table.
Column height ___________ meters

(Note: there are 100 centimeters in one meter)

4. Take the temperature of the liquid at room temperature (in °C) and record in the data table.
5. Use a stopwatch to time the Teflon ball as it drops through the oil. Drop the Teflon ball into the oil and measure the time it takes the ball to travel from the top line to the bottom line. Try to drop the ball as close to the liquid’s surface as you can. Conduct total of three trials and record the times in seconds in your data table.
6. Pour the liquid from the graduated cylinder into the Erlenmeyer flask until you have emptied most of the fluid. Before the balls drop, place a sample of steel wool into a funnel to catch the Teflon balls.
7. Heat the liquid in the Erlenmeyer flask on the hot plate to a temperature of 60°C. You can mix the liquid with a stirring rod.
8. Pour the heated liquid from the flask into the graduated cylinder up to 1000 mL mark (Note: oil may have expanded during heating. If so take a new height measurement).

New Column height ___________ meters

8. Drop a Teflon ball into the oil in the cylinder. Using a stopwatch, measure the time it takes the ball to travel from the top line to the bottom line. Try to drop the ball as close to the liquid’s surface as you can. Conduct a total of three trials and record the times in seconds in your data table.
9. Transfer the heated liquid to the original Erlenmeyer flask from the beginning and begin your calculations while the liquid cools. Do not clean up the liquid until its temperature has dropped below 50°C.
10. When everyone is finished, collect the data on the other sample liquids from the other groups.

**Data Sheet**

<table>
<thead>
<tr>
<th>Sample Material</th>
<th>Time to travel X-meters in Y-seconds at Room Temperature (_______ ° C)</th>
<th>Time to travel X-meters in Y-seconds at 60° C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Waste Vegetable Oil</td>
<td>m/ sec</td>
<td>m/ sec</td>
</tr>
<tr>
<td>Biodiesel</td>
<td>m/ sec</td>
<td>m/ sec</td>
</tr>
<tr>
<td>Corn Oil</td>
<td>m/ sec</td>
<td>m/ sec</td>
</tr>
<tr>
<td>Soy Oil</td>
<td>m/ sec</td>
<td>m/ sec</td>
</tr>
</tbody>
</table>
Questions

1) What factors affected how quickly the ball moved through each of the samples?

- The viscosity of the fluid, resulting largely from the shape and size of the component molecules.
- The temperature of the fluid. Higher temperature fluids have a lower viscosity.

2) Can you hypothesize why the ball moved more quickly through the heated samples than through the samples at room temperature?

3) How did the viscosity of the biodiesel compare to the other samples? Why might this be important when considering it as a fuel to be combusted within engines?

- Biodiesel typically has a lower viscosity than vegetable oils at lower temperatures. At higher temps, the viscosities become close. The viscosity of biodiesel in the liquid phase typically changes less than that of vegetable oil as temperature increases.

4) In addition to viscosity, what are some other important properties or qualities to consider in the development of alternative fuels?

- Emissions, environmental impact, energy density, transportability, cost, etc.
Viscosity Lab Procedure: Student Lab

Introduction

When diesel engines were invented in the late 1800s, they were intended to be able to run on a variety of fuels, and they could. The first diesel engines ran on coal dust, and shortly thereafter, people were running them on peanut oil. Petroleum, however, was rapidly emerging as a relatively cheap and energy dense source of fuel, which it has been for about the past 100 years or so. Today, most diesel vehicles today are designed to operate on petroleum diesel, and their designs optimize the properties of that fuel.

As you probably know, however, petroleum is a non-renewable resource, and it will become less and less available and more and more expensive in the next 100 years. Additionally, as you probably also know, it contributes to global climate change and pollution. For those reasons, we’re looking for alternative, renewable energy sources. To be used in a modern diesel vehicle, however, these energy sources must duplicate the properties of petroleum diesel fuel, and one of the most important properties to duplicate is viscosity.

Viscosity is the resistance of a liquid to flow. A good way of imagining viscosity is to think of pouring out a teaspoon of honey and a teaspoon of water. The honey has a much higher viscosity, or resistance to flow, in comparison to the water.

In this lab you will be comparing the viscosity of multiple oils in order to determine relative viscosity as well as how heating a sample affects the overall viscosity of the sample.

Safety Precautions:

Wear gloves and goggles at all times. When you are handling hot oils wear heat resistant gloves.
Materials:
- 1000 mL
- (glass) graduated cylinder
- 1000 mL sample of one of the following:
  - waste vegetable oil
  - biodiesel
  - corn oil
  - soy oil
- Hot plate
- Thermometer
- 5 x 1000 mL Erlenmeyer flasks
- Teflon balls (5/16” diameter) Funnel
- Steel wool
- Stopwatch
- Stirring Rod

Procedure
1) Fill the graduated cylinder with your liquid sample to the 1000 mL mark.
2) Measure the length of the column of oil in meters and record the length below. You will need this measurement later for your data table.
   Column length _________ meters
   (Note: there are 100 centimeters in one meter)
3) Take the temperature of the liquid at room temperature (in °C) and record in the data table.
4) Drop a Teflon ball into the oil in the cylinder while using a stopwatch to time from when the ball enters the oil to when it hits the bottom of the cylinder. Try to drop the ball as close to the liquid’s surface as you can. Conduct total of three trials and record the times in seconds in your data table.
5) Pour the liquid from the graduated cylinder into the Erlenmeyer flask until you have emptied most of the fluid. Before the balls drop, place a sample of steel wool into a funnel to catch the Teflon balls.
6) Heat the liquid in the Erlenmeyer flask on the hot plate to a temperature of 60°C. You can mix the liquid with a stirring rod to meet the required temperature.
7) Pour the heated liquid from the flask into the graduated cylinder up to 1000 mL mark (Note: oil may have expanded during heating. If so take a new length measurement).
   New Column length _________ meters
8) Drop a Teflon ball into the oil in the cylinder. Using a stopwatch, time from when the ball enters the oil to when it hits the bottom of the cylinder. Try to drop the ball as close to the liquid’s surface as you can. Conduct total of three trials and record the times in seconds in your data table.
9) Transfer the heated liquid to the original Erlenmeyer flask from the beginning and begin your calculations while the liquid cools. Do not clean up until the liquid temperature has dropped below 50°C.
10) When everyone is finished, collect the data on the other sample liquids from the other groups.
Data Sheet

<table>
<thead>
<tr>
<th>Sample Material</th>
<th>Time to travel X-meters in Y-seconds at Room Temperature (_______ °C)</th>
<th>Time to travel X-meters in Y-seconds at 60° C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Waste Vegetable Oil</td>
<td>m/ sec</td>
<td>m/ sec</td>
</tr>
<tr>
<td>Biodiesel</td>
<td>m/ sec</td>
<td>m/ sec</td>
</tr>
<tr>
<td>Corn Oil</td>
<td>m/ sec</td>
<td>m/ sec</td>
</tr>
<tr>
<td>Soy Oil</td>
<td>m/ sec</td>
<td>m/ sec</td>
</tr>
</tbody>
</table>

Questions

1) What factors affected how quickly the ball moved through each of the samples?

2) Can you hypothesize why the ball moved more quickly through the heated samples than through the samples at room temperature?

3) How did the viscosity of the biodiesel compare to the other samples? Why might this be important when considering it as a fuel to be combusted within engines?

4) In addition to viscosity, what are some other important properties or qualities to consider in the development of alternative fuels?
Goals

- Students will differentiate between density and viscosity.
- Students will separate and identify liquids by density.
- Students will predict viscosity of biodiesel.

Background

Density and viscosity are essential properties in characterizing liquids, and the differences between the two are important. Density is the amount of matter in a given space (mass per unit volume), and viscosity is the resistance of a liquid to flow. Unfortunately, the terms “thick” and “thin” are frequently used as synonyms for both dense/not dense and viscous/not viscous.

Students confuse density and viscosity, or they simply do not possess a separate viscosity concept. They are inclined to think that oils are denser than water, because oils do not flow as easily as water. They need the idea of viscosity as a distinct property, and if the ideas of viscosity and density are introduced in close proximity with deliberate comparisons, long term confusion might be avoided. The table below gives the densities and viscosities of six liquids arranged in order of viscosity. Though the organic substances follow the same order for viscosity and density, the inorganic substances, water and mercury, do not.

Table of Liquid Viscosities and Densities

<table>
<thead>
<tr>
<th>LIQUID</th>
<th>VISCOSITY (Pa-s)*** (at 20°C)</th>
<th>DENSITY (g/mL)*** (at 20°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Methanol</td>
<td>$5.9 \times 10^{-4}$</td>
<td>0.79</td>
</tr>
<tr>
<td>Water</td>
<td>$1.003 \times 10^{-3}$</td>
<td>1.00</td>
</tr>
<tr>
<td>Mercury</td>
<td>$1.55 \times 10^{-3}$</td>
<td>13.53</td>
</tr>
<tr>
<td>Biodiesel*</td>
<td>$34 \times 10^{-4}$ - $51 \times 10^{-4}$</td>
<td>0.86 - 0.90</td>
</tr>
<tr>
<td>Corn oil</td>
<td>$3.1 \times 10^{-2}$</td>
<td>0.92</td>
</tr>
<tr>
<td>Glycerin</td>
<td>$1.42$</td>
<td>1.26</td>
</tr>
</tbody>
</table>

*Varies by source, e.g., corn or soy

**Densities are given in g/mL, because they are more intuitive for students than the SI kg/m³. They can be changed by multiplying by 1000, e.g., 0.79 g/mL becomes 790 kg/m³.


Should students wish to explore other liquids, the website given as a footnote to the table provides common examples. It should be noted that in articles that deal with viscosity, in particular those concerned with biodiesel technology, two types of viscosity are usually mentioned: dynamic or absolute viscosity and kinematic viscosity. Dynamic or absolute viscosity is the number used here. (Kinematic viscosity is the ratio of dynamic viscosity to density.)
**Materials**
- Water (tap water works)
- Methanol (about 150 mL per student team)
- Corn oil (about 150 mL per student team)
- Biodiesel (about 150 mL/team; from previous lab?)
- Glycerin (about 150 mL/team; if from previous lab, this will be a glycerin/methanol mixture)
- 250 mL beakers (3/team)
- 100 mL graduated cylinders (1/team)
- Scales for finding mass
- Plastic balls, small enough to drop easily in the cylinder (5/team)
- Stopwatches (1/team)
- Printed lab sheets for Exploration and Experiment

**Preparation**
On Day 1 prepare a chart (chalkboard, overhead, or computer/projector) to record and share data on densities and ball drop times for water and methanol. On Day 2 a similar chart will be needed for corn oil, glycerin, and biodiesel. DATA FOR WATER AND METHANOL SHOULD BE INCLUDED ON THE DAY 2 CHART FOR COMPARISON.

This lab is meant to follow one in which students make biodiesel and glycerin from corn oil and methanol. If the products from this lab are used, the glycerin will really be a glycerin/methanol mixture. A sample of pure glycerin could be used for comparison. If this lab has not been completed, samples of biodiesel and glycerin will need to be obtained elsewhere, and students can skip the question about which product, biodiesel or glycerin, contains the waste methanol.

**Procedure (See also student version)**

**Exploration (Day 1)**
1) Students will find the densities of 2 liquids, water and methanol, by measuring 100 mL of each into graduated cylinders that have been massed. They find the mass of cylinder plus liquid and then the mass of the liquid by difference. They divide by 100 to find the density of each liquid in grams/milliliter. (This procedure is used to build an instinctive sense of liquid density, and it could be modified for students who are already very familiar with the density concept.)

2) Using the same cylinders with 100 mL of liquid, students will mark cylinders for each liquid by placing a piece of masking tape vertically on the cylinder. They will mark a starting point about 1 centimeter below the level of the liquid and a stopping point 10 centimeters below the starting point. They then use a stopwatch to find the time it takes a small ball to drop through the measured distance in the cylinder.

3) Class Discussion: Compare data posted by each group. It should be clear that water has a density greater than methanol (about 1 g/mL for water compared to about 0.8 g/mL for methanol). It should also be clear that the ball sinks faster in the methanol than in water. Students should
form a hypothesis for Day 2, remembering that a hypothesis has reason and a prediction. A good initial “reason” for the differences in time for the ball to sink is density. Students should be able to predict accordingly that denser liquids will have times greater than less dense liquids. A good hypothesis statement would be, “If denser liquids cause the ball to drop more slowly, then times for the ball to drop should be greater as density increases. (The density reason is NOT correct, but students should discover that for themselves on Day 2.)

**Experiment (Day 2)**
1) Students will find the densities of corn oil, biodiesel, and glycerin using the same procedure as Day 1.

2) Ball dropping is timed as in Day 1.

3) Students arrange data in a table according to ball drop times. It should become clear that the densities do not go in the same order.

4) Each group adds densities and times to the class chart for comparison.

5) The hypothesis has been disproven, because water is not in order. Students might need to be reminded that it only takes one piece of data to disprove a hypothesis.

6) Introduce the term viscosity: resistance to flow.

**Questions**
1. You found that density was not a dependable way to predict the time a ball takes to drop through a liquid. Instead, you found that there was another property of liquids called viscosity. In your own words, what is viscosity? (Answers may vary, but will probably include resistance to flow or stickiness.)

2. The liquids in the table below are arranged from lowest viscosity to greatest viscosity. How would the time for a ball to drop through mercury compare to that for water? How would it compare to corn oil? How does the density of mercury compare to water and corn oil? (Mercury is more viscous than water, so the ball would drop more slowly. Mercury is less viscous than corn oil, the ball would drop more quickly through mercury. Mercury is much more dense than either water or corn oil.)

<table>
<thead>
<tr>
<th>LIQUID</th>
<th>VISCOSITY (Pa·s)*** (at 20°C)</th>
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</tr>
<tr>
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<td>?</td>
<td>0.86-0.90</td>
</tr>
<tr>
<td>Corn oil</td>
<td>$310 \times 10^{-4}$</td>
<td>0.92</td>
</tr>
<tr>
<td>Glycerin</td>
<td>$1420 \times 10^{-4}$</td>
<td>1.26</td>
</tr>
</tbody>
</table>
3. Do you think heating a liquid would change the viscosity? Give a reason for your answer.  
(Heating will lower the viscosity. A possible reason for the answer is experience heating syrup; syrup flows more easily when it’s heated.)

4. When you made biodiesel and glycerin by mixing corn oil and methanol, you added more methanol than needed. Some methanol did not react, and it was left in one of the products. Based on the densities in the table and the densities you found for your biodiesel and glycerin samples, do you think the extra methanol was mixed with the biodiesel or the glycerin? Explain.  
(Answers may vary, but logically methanol will reduce the density of the product with which it mixes, so if students found a density lower than the density in the table, they should guess that that product contains the methanol. In fact, the methanol is found mixed in the glycerin.)
Properties of Liquids: Student Lab Day 1

EXPLORATION (DAY 1)

Question: How is density related to movement in liquid?

Materials / Equipment:
Your team will need
- 250 mL beakers, 2
- Tap water - a little more than 100 milliliters
- Methanol - a little more than 100 milliliters
- 100 mL graduated cylinder
- Nylon balls, 5
- Stopwatch

You will also be using a balance scale.

Procedure
1. Find the density of water by the following method:
   a. Use a 250 mL beaker to obtain a sample of tap water, slightly more than 100 mL.
   b. Find the mass of a dry 100 mL graduated cylinder. (Record below.)
   c. Pour exactly 100 mL of the water into the 100 mL graduated cylinder.
   d. Find the mass of the graduated cylinder with water. (Record below.)
   e. Subtract to find the mass of water alone.
   f. Find the density of water in g/mL (grams per milliliter).
   g. Pour the water back into its beaker for later use.

2. Find the density of methanol by the same method used for water. Complete the table below. Save the methanol in its beaker.

<table>
<thead>
<tr>
<th>WATER</th>
<th>METHANOL</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mass of cylinder with 100 mL liquid</td>
<td></td>
</tr>
<tr>
<td>Mass of dry cylinder</td>
<td></td>
</tr>
<tr>
<td>Mass of 100 mL liquid</td>
<td></td>
</tr>
<tr>
<td>Density of liquid (g/mL) Note: You know the mass of liquid in 100 grams. This will be the mass of liquid in 1 gram.</td>
<td></td>
</tr>
</tbody>
</table>

3. Record the densities in the class data table.

4. Use the following method to find how long it takes a nylon ball to fall 10 centimeters through water:
a. Place a piece of masking tape vertically on the graduated cylinder.
b. Mark a starting point about 1 centimeter below the 100 mL mark.
c. Mark a stopping point 10 centimeters below the starting point.
d. Put 100 mL of water in the cylinder.
e. Drop a ball in the water. Start the stopwatch (or other timer) when the ball reaches the starting point on the masking tape. Stop the stopwatch when the ball reaches stopping point on the masking tape.
f. Record the time below.
g. Repeat 2 more times and average the 3 times for water.

5. Use the same method to find how long it takes the ball to fall through methanol. You may discard the water, but DO NOT discard the methanol.

<table>
<thead>
<tr>
<th>TIME FOR BALL TO FALL 10 CENTIMETERS</th>
<th>WATER</th>
<th>METHANOL</th>
</tr>
</thead>
<tbody>
<tr>
<td>Trial #1</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Trial #2</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Trial #3</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Average time</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

6. Record the average times in the class data table.

**Class Discussion**

1. Based on data from the whole class, how do the densities of water and methanol compare?

2. Based on data from the whole class, how do the ball drop times for water and methanol compare?

3. How might you predict ball drop times for other liquids? Write this in the form of a hypothesis, remembering that the hypothesis gives reasoning for believing the prediction might be true, and it is testable.
Properties of Liquids: Student Lab Day 2

EXPERIMENT (DAY 2)

Question: How is density related to movement in liquid?

Materials/Equipment:
Your team will need
- 250 mL beakers, 3
- Corn oil - a little more than 100 milliliters
- Biodiesel - a little more than 100 milliliters
- Glycerin - a little more than 100 milliliters
- 100 mL graduated cylinder
- Nylon balls, 5
- Stopwatch

You will also be using a balance scale.

Hypothesis (Use the hypothesis you developed on Day 1, or change it if you wish.)

Procedure
1. Use the same method you used in the Exploration to find the time it takes for a nylon ball to fall 10 centimeters through each of the following liquids: corn oil, glycerin, and biodiesel. Put your data in Table 1.
2. Use these times and the times for water and methanol found in the Exploration (Day 1). Arrange the liquids in Table 3 from least time to greatest time.
3. Use the same method you used in the Exploration to find the densities of each liquid. Use Table 2 for your raw data, and then put the densities in Table 3.
4. Add your times and densities to the class data table.
Results
Table 1: Ball drop times.

<table>
<thead>
<tr>
<th>TIME FOR BALL TO FALL 10 CENTIMETERS</th>
<th>CORN OIL</th>
<th>GLYCERIN</th>
<th>BIODIESEL</th>
</tr>
</thead>
<tbody>
<tr>
<td>Trial #1</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Trial #2</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Trial #3</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Average time</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Table 2: Densities.

<table>
<thead>
<tr>
<th>Mass of cylinder with 100 mL liquid</th>
<th>CORN OIL</th>
<th>GLYCERIN</th>
<th>BIODIESEL</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mass of dry cylinder</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Mass of 100 mL liquid</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Density of liquid (g/mL)</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Table 3: Liquids from least to greatest ball drop time.

<table>
<thead>
<tr>
<th>LIQUID</th>
<th>BALL DROP TIME</th>
<th>DENSITY</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
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<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Conclusion: Explain whether the data in Table 3 supports your hypothesis or not. Then answer the beginning question.

_____________________________________________________________________________

_____________________________________________________________________________

_____________________________________________________________________________
Properties of Liquids: Post Lab

Post Lab Questions

1. You found that density was not a dependable way to predict the time a ball takes to drop through a liquid. Instead, you found that there was another property of liquids called viscosity. In your own words, what is viscosity?

2. The liquids in the table below are arranged from lowest viscosity to greatest viscosity. How would the time for a ball to drop through mercury compare to that for water? How would it compare to corn oil? How does the density of mercury compare to water and corn oil?

<table>
<thead>
<tr>
<th>LIQUID</th>
<th>VISCOSITY (Pa·s) (at 20°C)</th>
<th>DENSITY (g/mL) (at 20°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Methanol</td>
<td>$5.9 \times 10^{-4}$</td>
<td>0.79</td>
</tr>
<tr>
<td>Water</td>
<td>$10.03 \times 10^{-4}$</td>
<td>1.00</td>
</tr>
<tr>
<td>Mercury</td>
<td>$15.5 \times 10^{-4}$</td>
<td>13.53</td>
</tr>
<tr>
<td>Biodiesel*</td>
<td>$34 \times 10^{-4} - 51 \times 10^{-4}$</td>
<td>0.86 - 0.90</td>
</tr>
<tr>
<td>Corn oil</td>
<td>$310 \times 10^{-4}$</td>
<td>0.92</td>
</tr>
<tr>
<td>Glycerin</td>
<td>$1420 \times 10^{-4}$</td>
<td>1.26</td>
</tr>
</tbody>
</table>

*Varies by source, for instance, corn or soy

3. Do you think heating a liquid would change the viscosity? Give a reason for your answer.

4. When you made biodiesel and glycerin by mixing corn oil and methanol, you added more methanol than needed. Some methanol did not react, and it was left in one of the products. Based on the densities in the table and the densities you found for your biodiesel and glycerin samples, do you think the extra methanol was mixed with the biodiesel or the glycerin? Explain.

Goals:
- Students will understand how matter and energy are affected by combustion.
- Students will understand the difference between complete and incomplete combustion.
- Students will understand the energy content and combustion emission differences between diesel and biodiesel fuel.

Background
Combustion involves a series of chemical reactions between a fuel (i.e. a hydrocarbon, or an organic compound containing only carbon and hydrogen) and oxygen. The result is a major reorganization of both matter and energy.

Matter
Combustion can be complete or incomplete depending on how much oxygen is present. The diagrams below represent complete combustions, which happens in the presence of ample oxygen. When complete combustion occurs, all of the carbon atoms in a fuel (i.e. the diesel and biodiesel molecules below) will be converted to carbon dioxide molecules. Also, the hydrogen atoms that were attached to each carbon atom in the fuel bind with oxygen to form water.

Complete combustion:

\[
\text{Fuel} + \text{Oxygen} \rightarrow \text{Carbon Dioxide} + \text{Water} + \text{Energy}
\]

Complete combustion of diesel:

\[
\text{C}_{12}\text{H}_{24} + 36\text{O}_2 \rightarrow 12\text{CO}_2 + 12\text{H}_2\text{O} + \text{Energy}
\]

Complete combustion of biodiesel:

\[
\text{H} \quad \text{O} \\
\text{H—C—O—C—R} + \text{O}_2 \quad \text{H} \\
\text{Biodiesel} + \text{Oxygen} \rightarrow \text{CO}_2 + \text{H}_2\text{O} + \text{Energy}
\]

Note that \( R \), the hydrocarbon chain, will vary in length depending on the feedstock used to make the biodiesel (it will be greater than 10 carbons).
When combustion is incomplete (due to lack of oxygen), other products such as carbon monoxide and particulates form in addition to carbon dioxide and water.

Incomplete combustion:

\[
\text{Fuel} + \text{Oxygen} \rightarrow \text{Carbon Dioxide} + \text{Water} + \text{Energy} + \text{Soot} + \text{Carbon Monoxide}
\]

Incomplete combustion is common. You may have heard of the dangers of carbon monoxide poisoning that are associated with furnaces incompletely combusting natural gas in a low-oxygen environment. You may have also seen the effects of incomplete combustion in a smoky campfire (combusting biomass releases a lot of particulates or soot). In this lab, you will qualitatively compare the completeness of combustion of diesel and biodiesel by visually examining the emissions produced during combustion.

Energy

As you may have noticed in the diagrams above, combustion also releases energy stored in fuel. In the case of a hydrocarbon, the energy was stored as chemical potential energy in hydrocarbon bonds. The more hydrocarbon bonds, the more energy stored in a hydrocarbon molecule. Through combustion, that chemical potential energy is converted to light and heat.

The energy released from the combustion of fuel within the diesel engine drives the car. When diesel or biodiesel has been injected into an engine cylinder and is combined with oxygen taken in through the engine’s air intake valves, combustion occurs. The explosion moves the piston which powers the crank shaft. This eventually results in the wheels moving.

A fuel that has more stored energy will mean more miles per gallon and power for the driver. In this lab, you will quantitatively compare the energy contents (by measuring heat of combustion) of biodiesel and diesel using principles of calorimetry.

Calorimetry

Calorimetry is the science of measuring the heat evolved or required in a chemical reaction. In this lab you will construct a simple calorimeter to measure the heat released when combusting diesel and biodiesel. Specifically, you will burn the two fuels in a consistent manner and measure the temperature increase in a known volume of water. If your calorimeter was a closed system, all of the heat released would be transferred to the water. Obviously, some heat in your system will be lost to the environment, but you should still be able to take meaningful relative measurements.
Materials
- Emptied and cleaned 12-oz aluminum soda cans (two per student group)
- Pencil (one per group)
- Ring stand with ring attached (one per group)
- Oil burners (two per group—one filled with diesel and one filled with biodiesel)
- Small funnel (one per group)
- Lighter (one per group)
- 250 ml Graduated cylinder (one per group)
- Alcohol thermometer (one per group)
- Balance accurate to 0.01g (Weight capacity: ~500 g)
- Ruler
- Calculator
- Marker (for labeling)

Safety
Because we are burning fuels, this laboratory is best done outdoors or underneath a fume hood to avoid inhalation of fumes. Also, to protect yourself, make sure to wear goggles and gloves at all times.

In addition, it is important to make sure that equipment is clean and that no oil has been spilled over the outside of the burners in order to prevent any accidental flames from forming anywhere besides the wick of the burner.

Procedure
Note: You have one oil burner filled with either diesel or biodiesel in front of you. Begin the procedure using that burner. Repeat the procedure using an oil burner filled with the other fuel (you will need to obtain this from another group).

1. Determine the mass of the oil burner and fuel (with the cap on) using the balance. Record the mass in the data table on the next page.
2. Measure 200 ml of water using a graduated cylinder. Using a funnel, pour the water into the soda can. Record the mass of water in the can on the data table (Note: 1 ml H₂O = 1 g H₂O at room temperature).
3. Fasten the soda can to the ring stand by placing a pencil through the can tab. Let the can fall through the ring until it is suspended by the pencil on the ring stand.
4. To measure the initial temperature of the water, hold an alcohol thermometer in the water so that it does not touch the sides of the soda can. Record the temperature in the data table.
5. Place burner under can. Adjust the ring height so that the top of the wick is a measured 3 cm below bottom of the can (Figure 1). Center the oil burner under the can.
6. Remove the cap from the oil burner and light the wick using a lighter. (You may need to adjust the ring stand to light the burner. Be sure to quickly reposition after the flame is lit.)
7. Use a thermometer to measure the temperature of the water. Use the thermometer to stir the water periodically.
8. As you burn your fuel make observations on your data table about associated smells, and the nature of the flame and smoke.
9. Continue heating and stirring the water until the temperature has increased by ~25°C. At that point, raise the ring (with the can still attached) and quickly extinguish the flame by placing the burner cap over the wick.
10. Continue to gently stir the water and record the maximum temperature the water reaches.
11. Determine the final mass of the oil burner (with the cap) and the remaining fuel using the balance. Record the final mass in the data table.
12. Note the quantity of soot on the soda can in your data table (under observations).
13. Repeat steps 1-13 with your other fuels.
14. Clean all equipment.

Data table
Use the following table to record your data and emissions observations as well as data gathered during Data analysis (after Data table). Step by step calculations can be found on the next page.

<table>
<thead>
<tr>
<th></th>
<th>Biodiesel</th>
<th>Diesel</th>
</tr>
</thead>
<tbody>
<tr>
<td>Initial water temp. (°C)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Final water temp. (°C)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Change in water temp. (°C) (Δ t)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Heat (J, calculated using q=mCΔt)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Initial mass of oil burner (g)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Final mass of oil burner (g)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Mass of fuel burned (g)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Heat of combustion (J/g)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Observations:</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Data Analysis
Use the information in your data table to calculate the amount of energy released as heat, heat of combustion (J/g), by diesel and biodiesel.

1) For each fuel calculate change in temperature (\(\Delta t\)) and the mass of fuel burned (g). Record your results into your data table.

\[ \Delta t (°C) = \text{Final Temperature} - \text{Initial Temperature} \]

With biodiesel:

With diesel:

\[ \text{Mass of fuel burned (g)} = \text{Final mass of oil burner} - \text{Initial mass of oil burner} \]

Biodiesel:

Diesel:
2) The amount of heat transferred to the water can be determined by using the following equation: 
\[ q = mC\Delta t \], where \( q \) is heat in Joules, \( m \) is mass in grams of water, \( C \) is the heat capacity of water (4.18 J/g°C), and \( \Delta t \) is the temperature change of the water in degrees Celsius. Record your results into your data table.

With biodiesel, \( q = \)

With diesel, \( q = \)

3) Calculate the heat of combustion by dividing \( q \) by the mass of fuel of used. Record your results into your data table.

Heat of combustion (biodiesel)

Heat of combustion (diesel)
Questions
1) Where did the energy you measured as heat come from?

2) What do these results indicate about the relative energy content of diesel and biodiesel?

3) How do your results compare to Argonne Laboratory’s heat of combustion results for diesel and biodiesel (see table on the next page)?

4) Why do you think your results differ from the Argonne Laboratory results?

<table>
<thead>
<tr>
<th>Fuel</th>
<th>Your heat of combustion (J/g) value</th>
<th>Argonne Laboratory’s heat of combustion (J/g) value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Biodiesel</td>
<td></td>
<td>40,160</td>
</tr>
<tr>
<td>Diesel</td>
<td></td>
<td>45,759</td>
</tr>
</tbody>
</table>

5) Did you witness complete or incomplete combustion of diesel and biodiesel? How do you know?
6) According to the US Environmental Protection Agency, biodiesel produces 47% less particulate matter and 48% less carbon monoxide emissions than diesel. Are the EPA particulate matter results aligned with your observations of combustion?

7) Can you explain the differences in the combustion results described above (Hint: look at the molecular formulas of diesel and biodiesel)?

8) What are the comparative advantages of using biodiesel instead of regular diesel?
Combustion of a Renewable and Fossil Fuel: Student Lab

Background
Combustion involves a series of chemical reactions between a fuel (i.e., a hydrocarbon, or an organic compound containing only carbon and hydrogen) and oxygen. The result is a major reorganization of both matter and energy. Combustion can be complete or incomplete depending on how much oxygen is present. When complete combustion occurs, all of the carbon atoms in a fuel (i.e., the diesel and biodiesel molecules) will be converted to carbon dioxide molecules. When combustion is incomplete (due to a lack of oxygen), other products, such as carbon monoxide and particulates, form in addition to carbon dioxide and water. In this lab, you will qualitatively compare the completeness of combustion of diesel and biodiesel by visually examining the emissions produced during combustion.

Materials
- 2 – 12-oz aluminum soda cans
- Pencil
- Ring stand with ring attached
- Oil burner (1 Biodiesel, 1 Diesel)
- Small funnel
- Lighter
- 250 ml Graduated cylinder
- Alcohol thermometer
- Ruler
- Calculator
- Marker

Procedure
Note: You have one oil burner filled with either diesel or biodiesel in front of you. Begin the procedure using that burner. Repeat the procedure using an oil burner filled with the other fuel (you will need to obtain this from another group).

1. Determine the mass of the oil burner and fuel (with the cap on) using the balance. Record the mass in the data table on the next page.
2. Measure 200 ml of water using a graduated cylinder. Using a funnel, pour the water into the soda can.
3. Record the mass of water in the can on the data table (Note: 1 ml H₂O = 1 g H₂O at room temperature).
4. Fasten the soda can to the ring stand by placing a pencil through the can tab. Let the can fall through the ring until it is suspended by the pencil on the ring stand.
5. To measure the initial temperature of the water, hold an alcohol thermometer in the water so that it does not touch the sides of the soda can. Record the temperature in the data table.
6. Place burner under can. Adjust the ring height so that the top of the wick is a measured 3 cm below bottom of the can (Figure 1). Center the oil burner under the can.
7. Remove the cap from the oil burner and light the wick using a lighter.
   a. You may need to adjust the ring stand to light the burner. Be sure to quickly reposition after the flame is lit.
8. Use a thermometer to measure the temperature of the water.
   a. Use the thermometer to stir the water periodically.
9. As you burn your fuel make observations on your data table about associated smells, and the nature of the flame and smoke.
10. Continue heating and stirring the water until the temperature has increased by ~25°C. At that point, raise the ring (with the can still attached) and quickly extinguish the flame by placing the burner cap over the wick.
11. Continue to gently stir the water and record the maximum temperature the water reaches.
12. Determine the final mass of the oil burner (with the cap) and the remaining fuel using the balance. Record the final mass in the data table.
13. Note the quantity of soot on the soda can in your data table (under observations).
14. Repeat steps 1-13 with your other fuels.
15. Clean all equipment.

**Data table**

Use the following table to record your data and emissions observations as well as data gathered during Data analysis (after Data table). Step by step calculations can be found on the next page.

<table>
<thead>
<tr>
<th></th>
<th>Biodiesel</th>
<th>Diesel</th>
</tr>
</thead>
<tbody>
<tr>
<td>Initial water temp. (°C)</td>
<td></td>
<td></td>
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<tr>
<td>Final water temp. (°C)</td>
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<td></td>
</tr>
<tr>
<td>Change in water temp. (°C) (Δ t)</td>
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</tr>
<tr>
<td>Heat (J, calculated using q=mCΔt)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Initial mass of oil burner (g)</td>
<td></td>
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</tr>
<tr>
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<td></td>
</tr>
<tr>
<td>Mass of fuel burned (g)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Heat of combustion (J/g)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Observations:</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Data analysis
Use the information in your data table to calculate the amount of energy released as heat, heat of combustion (J/g), by diesel and biodiesel.

1) For each fuel calculate change in temperature (Δt) and the mass of fuel burned (g). Record your results into your data table.

\[ \Delta t \ (°C) = \text{Final Temperature} - \text{Initial Temperature} \]

With biodiesel:

With diesel:

\[ \text{Mass of fuel burned (g)} = \text{Final mass of oil burner} - \text{Initial mass of oil burner} \]

Biodiesel:

Diesel:

2) The amount of heat transferred to the water can be determined by using the following equation:

\[ q = mC\Delta t \]

where \( q \) is heat in Joules, \( m \) is mass in grams of water, \( C \) is the heat capacity of water (4.18 J/g°C), and \( \Delta t \) is the temperature change of the water in degrees Celsius. Record your results into your data table.

With biodiesel, \( q = \)

With diesel, \( q = \)
3) Calculate the heat of combustion by dividing q by the mass of fuel used. Record your results into your data table.

Heat of combustion (biodiesel)

Heat of combustion (diesel)

Questions
1) Where did the energy you measured as heat come from?

2) What do these results indicate about the relative energy content of diesel and biodiesel?

3) How do your results compare to Argonne Laboratory’s heat of combustion results for diesel and biodiesel (see table on the next page)?

4) Why do you think your results differ from the Argonne Laboratory results?
5) Did you witness complete or incomplete combustion of diesel and biodiesel? How do you know?

6) According to the US Environmental Protection Agency, biodiesel produces 47% less particulate matter and 48% less carbon monoxide emissions than diesel. Are the EPA particulate matter results aligned with your observations of combustion?

7) Can you explain the differences in the combustion results described above (Hint: look at the molecular formulas of diesel and biodiesel)?

8) What are the comparative advantages of using biodiesel instead of regular diesel?
Biodiesel Reaction Efficiency (27/3 Test)

Learning Goals:
- Students will understand variables that affect reaction efficiency
- Students will understand test methods for determining reaction efficiency
- Students will be introduced to Le Châtelier's principles of chemical equilibrium

Background:
In this lab we are going to test the efficiency of the transesterification reaction. We will compare two different samples of biodiesel. These samples should differ in some way: made by different students, different amounts of catalyst, different oils, etc.

The test gives a qualitative analysis of residual glycerin in the biodiesel product, a sign of incomplete reaction. The glycerin will settle on the bottom of the test jar in small droplets. More glycerin droplets indicates a less complete reaction. If glycerin is observed, the biodiesel is highly likely to fail industrial standard testing. If no glycerin is observed, the biodiesel sample is of high quality but may not pass industrial standards. To get a definitive answer you need a quantitative result instead of the qualitative result returned by this test. Gas chromatography is used to get quantitative results of residual free glycerin, monoglycerides, diglycerides, and triglycerides.

Materials:
- 50 mL Graduated Cylinder
- Small Sample Jar with Lid (x 2)
- 54 mL of Methanol (27 mL for each test)
- 3 mL of Biodiesel Sample 1
- 3 mL of Biodiesel Sample 2

Procedure:

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.
Reaction Efficiency Test – 27/3 Test

1. Measure 27 mL of methanol
2. Add methanol to small sample jar
3. Measure 3 mL of biodiesel sample 1
4. Add to the sample jar
5. Cap the jar
6. Shake thoroughly
7. Allow the sample to settle for 15 minutes
8. Repeat procedure for Sample 2

After 15 minutes, compare the test jars. We are looking for small droplets of glycerin that will form along the bottom of the small test jar. More glycerin indicates a less complete biodiesel reaction and results in a low quality fuel. If no glycerin settles out then the biodiesel is very high quality.

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
Making Liquid Soap: Teacher Manual

Learning Goals:
- Students will understand how to produce liquid soap from glycerin.
- Students will understand how to adjust variables to alter the physical properties of a product.
- Students will understand the distinction between reagents and products.

Objectives:
- Students will make liquid soap from glycerin they made in a biodiesel reaction.
- Students will record actions, calculations, and observations in a laboratory notebook.

Extended Background:
Making biodiesel can be a simple, effective way to produce liquid transportation fuel that has both financial and environmental benefits. Another advantage of biodiesel is that it can be made from waste cooking oil after it has already been used for cooking foods in the kitchen, cafeteria, or restaurant. The oil molecule is still suitable for biodiesel production so this waste can be utilized to make fuel. Unfortunately, the chemical process of making biodiesel, transesterification, results in two products: biodiesel and glycerin. The biodiesel is the primary product, but the glycerin is very useful as well. Additionally, we don’t want to take one waste product (cooking oil) and create another waste product (glycerin). Therefore we refer to the glycerin produced during the biodiesel reaction as a “by-product”.

By-products retain both financial and environmental value that we can capture through other chemical processes. Glycerin has hundreds of uses from food sweetener to heart medication depending on the quality. Glycerin derived from biodiesel production is a low quality product because it contains many contaminants from the biodiesel reaction. Biodiesel glycerin is actually a mixture of free fatty acids (FFA) that were neutralized during transesterification, soaps, water, catalyst (NaOH or KOH depending on what was used to make the biodiesel), methanol, and glycerin. The only contaminant that poses a risk is the methanol. To obtain a high conversion of oil into biodiesel we use a purposeful excess of methanol. The excess methanol settles into the glycerin layer and must be removed to make the glycerin safe to handle.

All residual methanol must be removed before the glycerin can be used for making soap. Methanol will boil off at temperatures above 68°C. The methanol can be distilled out of the glycerin to capture the methanol or simply heated under a fume hood to remove methanol. Allow 45 minutes of boiling at temperature to ensure all methanol is driven off.

Once the methanol is removed, the glycerin is safe to handle and is suitable for making soap. The remaining contaminants are all ingredients in soap. Producing soap is the easiest way to capture the value of the glycerin at the small scale. The following lab is designed to show how glycerin, from biodiesel made with KOH, can be turned into a liquid soap with a multitude of uses from hand soap to glass cleaner.
Materials:
- Glycerin, Methanol Removed (200 grams per pair of students)
- Essential Oil (optional)
- Coconut Oil (25 g per pair of students)
- Citric Acid
- 250 mL graduated cylinder
- Scale or balance
- Potassium Hydroxide (KOH)
- 1,000 mL beaker
- 250 mL beaker x 2
- Water (DI or Distilled if available)
- Hot Plates
- Thermometer
- pH Strips or Meter

Preparation:
Ensure all residual methanol is removed from glycerin. Methanol will boil off at temperatures above 68°C. Allow 45 minutes of boiling at temperature to ensure all methanol is driven off.

Procedure:
1. Prepare glycerin
   a. Measure 200 grams of glycerin into a 1,000 mL beaker
   b. Heat glycerin to 60°C
   c. Stir glycerin gently and check temperature with thermometer
2. Prepare coconut oil
   a. Weigh 25 grams of coconut oil in a 250 mL beaker
   b. Heat coconut oil enough to melt the oil
   c. Add to glycerin
3. Prepare potassium hydroxide (KOH) solution
   a. Measure 75 mL of water into a 250 mL beaker
      i. Use DI or distilled water if available
      ii. Fewer impurities in water, especially if you have hard water, will improve the quality and clarity (color) of the soap.
   b. Measure 25 grams of KOH
      i. KOH is hydroscopic. It will absorb moisture from the air causing it to weigh more and be less effective. Keep KOH covered.
      ii. KOH dust can irritate the nostrils and throat. Avoid breathing the dust or fumes when mixing KOH solutions.
   c. Add the measured KOH to the water and swirl to dissolve
      i. If available, perform this step under a fume hood
4. Mix Soap
   a. Gently pour KOH/Water solution into the hot glycerin
   b. Heat soap and maintain temperature of 60-70°C
   c. Stir soap constantly
      i. Mix for 5 minutes
      ii. The soap will start to thicken as the glycerin and coconut oil are saponified
d. Add 250 mL of dilution water to the soap  
   i. Use DI or distilled water if available  
   ii. Fewer impurities in water, especially if you have hard water, will improve the  
       quality and clarity (color) of the soap.

e. Continue to heat and stir soap for an additional 20 minutes

f. After 20 minutes:
   i. Take soap off of the heat
   ii. Allow to cool to room temperature
   iii. Let soap rest overnight if possible

5. pH Balance Soap
   
a. Test the pH of your soap  
   i. Use pH test strips and/or a pH meter  
   ii. Target pH is 9.75

b. The pH is likely to be too high, but this was done purposefully to ensure that all of the  
   glycerin and coconut oil were saponified.

c. Lower pH by adding 20% citric acid solution  
   i. Dissolve 20 grams of citric acid in 80 grams of water  
   ii. Slowly add small amounts to the soap  
   iii. Stir  
   iv. Retest pH  
   v. Continue this until you reach your target pH of 9.75

6. OPTIONAL:
   
a. At this point you can add a few grams of essential oil to scent the soap
   b. Concentrated essential oil is very strong and a little goes a long way

7. Test
   
a. Test the soap on your hands, floors, tables...almost anything can be cleaned with this  
   universal soap!
   b. Observe the soap over the next couple days and weeks. The soap should not form any  
   layers and the pH should remain stable.

Questions:

What is the purpose of adding glycerin to the soap? Essential oil?

What would happen if we dissolved the soap paste in more/less water?

What could this soap be used for?

How does making soap fit in with making biodiesel?
Making Liquid Soap: Student Lab

Learning Goals:
- Students will understand how to produce liquid soap from glycerin.
- Students will understand how to adjust variables to alter the physical properties of a product.
- Students will understand the distinction between reagents and products.

Objectives:
- Students will make liquid soap from cooking oil and glycerin they made in a biodiesel reaction
- Students will record actions, calculations, and observations in a laboratory notebook.

Extended Background:
Making biodiesel can be a simple, effective way to produce liquid transportation fuel that has both financial and environmental benefits. Biodiesel can be made from waste cooking oil, but we don’t want to take one waste product (cooking oil) and create another waste product (glycerin). Therefore we refer to the glycerin produced during the biodiesel reaction as a “by-product”.

By-products retain both financial and environmental value that we can capture through other chemical processes. Biodiesel glycerin is actually a mixture of free fatty acids (FFA) that were neutralized during transesterification, soaps, water, catalyst (NaOH or KOH depending on what was used to make the biodiesel), methanol, and glycerin.

Once the methanol is removed, the glycerin is safe to handle and is suitable for making soap. The remaining contaminates are all ingredients in soap making soap production the easiest way to capture the value of the glycerin. The following lab is designed to show how glycerin, from biodiesel made with KOH, can be turned into a liquid soap with a multitude of uses from hand soap to stainless steel cleaner.

Materials:
- Glycerin, Methanol Removed (200 grams per pair of students)
- Essential Oil (optional)
- Coconut Oil (25 g per pair of students)
- Citric Acid
- 250 mL graduated cylinder
- Scale or balance
- Potassium Hydroxide (KOH)
- 1,000 mL beaker
- 250 mL beaker x 2
- Water (DI or Distilled if available)
- Hot Plate
- Thermometer
- pH Strips or Meter
Procedure:

1. Prepare glycerin
   a. Measure 200 grams of glycerin into a 1,000 mL beaker
   b. Heat glycerin to 60°C
   c. Stir glycerin gently and check temperature with thermometer

2. Prepare coconut oil
   a. Weigh 25 grams of coconut oil in a 250 mL beaker
   b. Heat coconut oil enough to melt the oil
   c. Add to glycerin

3. Prepare potassium hydroxide (KOH) solution
   a. Measure 75 mL of water into a 250 mL beaker
      i. Use DI or distilled water if available
      ii. Fewer impurities in water, especially if you have hard water, will improve the quality and clarity (color) of the soap.
   b. Measure 25 grams of KOH
      i. KOH is hygroscopic. It will absorb moisture from the air causing it to weigh more and be less effective. Keep KOH covered.
      ii. KOH dust can irritate the nostrils and throat. Avoid breathing the dust or fumes when mixing KOH solutions.
   c. Add the measured KOH to the water and swirl to dissolve
      i. If available, perform this step under a fume hood

4. Mix Soap
   a. Gently pour KOH/Water solution into the hot glycerin
   b. Heat soap and maintain temperature of 60-70°C
   c. Stir soap constantly
      i. Mix for 5 minutes
      ii. The soap will start to thicken as the glycerin and coconut oil are saponified
   d. Add 250 mL of dilution water to the soap
      i. Use DI or distilled water if available
      ii. Fewer impurities in water, especially if you have hard water, will improve the quality and clarity (color) of the soap.
   e. Continue to heat and stir soap for an additional 20 minutes
   f. After 20 minutes:
      i. Take soap off of the heat
      ii. Allow to cool to room temperature
      iii. Let soap rest overnight if possible

5. pH Balance Soap
   a. Test the pH of your soap
i. Use pH test strips and/or a pH meter
ii. Target pH is 9.75
b. The pH is likely to be too high, but this was done purposefully to ensure that all of the glycerin and coconut oil were saponified.
c. Lower pH by adding 20% citric acid solution
   i. Dissolve 20 grams of citric acid in 80 grams of water
   ii. Slowly add small amounts to the soap
   iii. Stir
   iv. Retest pH
   v. Continue this until you reach your target pH of 9.75

6. OPTIONAL:
   a. At this point you can add a few grams of essential oil to scent the soap
   b. Concentrated essential oil is very strong and a little goes a long way

7. Test
   a. Test the soap on your hands, floors, tables...almost anything can be cleaned with this universal soap!
   b. Observe the soap over the next couple days and weeks. The soap should not form any layers and the pH should remain stable.

Questions:
What is the purpose of adding glycerin to the soap? Essential oil?

What would happen if we dissolved the soap paste in more/less water?

What could this soap be used for?

How does making soap fit in with making biodiesel?
Learning Goals:

- Students will understand the underlying rationale and basic process of life cycle analysis
- Students will understand points of differentiation in the life cycles of biodiesel and ethanol

Background:

Ethanol

Ethanol is a colorless transportation fuel that mixes with gasoline. Low concentrations of ethanol, such as E10 (10% ethanol, 90% gasoline) can be burned in most gasoline engines. A flex-fuel vehicle is required to use higher concentrations of ethanol, such as E85.

Ethanol can be made from many plant-based sources. In the United States, it is most commonly produced from starch in corn grains. In Brazil, ethanol is commonly made from sugar cane. Although the technology is being developed for large-scale production, ethanol can also be made from cellulose, a structural compound found in plants. Examples of feedstocks currently being investigated for cellulosic ethanol production include fast-growing grasses and trees, as well as waste products such as corn stover (the stems left on the field after harvest) and wood chips. Because ethanol is made from plants that can be regenerated, it is a renewable fuel.

To make ethanol, the starches or cellulose are first converted to sugars. The sugars are then fermented (recall that fermentation is a form of anaerobic respiration) to produce alcohol. The alcohol is purified via distillation, a process that separates liquids based on their boiling points.

Biodiesel

Biodiesel is a liquid transportation fuel that mixes with diesel (not compatible with gasoline engines). It can be burned in any concentration (B1 to B100) in most unmodified diesel engines.

Like ethanol, biodiesel is also made from organic sources. Biodiesel can be made from plant-, algae- or animal-derived fats. Common plant-based biodiesel feedstocks include soy (the most common biodiesel feedstock used in the United States), canola, and palm oil. Other biodiesel producers use waste products such as waste vegetable oil from restaurant fryers or rendered animal fat. The large-scale production of biodiesel from algal oil is still being developed. Because biodiesel can be produced from a variety of organic sources, it is also a renewable fuel.

To make biodiesel the oils or fats, both of which are composed of triglyceride molecules, are modified through a chemical transformation called transesterification. In transesterification, the glycerin portion of the triglyceride molecule is separated from and the fatty acid chains and the fatty acid chains are methylated forming fatty acid methyl esters (or biodiesels!!).
Biofuels and life cycle analysis
Ethanol and biodiesel have both received praise and skepticism. Their supporters tout the fuels as solutions to the environmental problems associated with burning gasoline and diesel, such as global climate change. This is because biofuels are thought to reduce net emissions of carbon dioxide because plant-based feedstocks capture carbon dioxide from the atmosphere via photosynthesis during their growth. However, skeptics believe that the benefits of this “carbon credit” are dwarfed by emissions incurred during the production, distribution, and use of biofuels. One tool, life cycle analysis (LCA), has been particularly useful in generating information about the environmental benefits and constraints of biofuels.

LCA examines the environmental impact of a product from “the cradle to the grave”. Specifically, LCAs quantify and evaluate all raw materials and energy consumed as well as wastes discharged beginning with product design and the sourcing of the raw materials from the earth through manufacturing, distribution, use, maintenance, and disposal.

LCAs can be used in various ways. They are often used to compare two products. For example, questions about the environment impacts of paper vs. plastic bags have been explore using LCA. LCAs can be used by a product’s manufacturer to identify processes or materials used that have a high environmental impact and make substitutions to lower the impact, or to educate consumers about a product. In this activity, you will construct the life cycles of two renewable fuels, ethanol and biodiesel. Then, using information from published LCAs, you will evaluate and compare their environmental impacts.

Materials:
- Colored Beads – 20 of Each per Group
  - Green, Blue, Red, Orange, and Yellow

Activity:
1.) The steps, inputs, and outputs involved in the production of ethanol and biodiesel are listed below. On the next page, arrange these processes or materials in a diagram that you feel best describes the life cycle of each fuel.
For corn grain ethanol and biodiesel from soy:
Ethanol/Biodiesel production at a plant
Consumer vehicle operation
Fertilizer
Pesticides
Energy use
Materials
Refueling station
Carbon sequestration
GHG Emissions
Transportation
Corn/Soybean cultivation and production
Water

**NOTE:** Some of these are processes and some are physical materials. Also, some items may be used once and some will be used multiple times.

2.) Draw your diagram below:
Wait for further instructions before proceeding

4.) Your diagrams illustrate that in the production and use of biofuels environmental impacts are incurred. However, the amount of resources used or pollutants generated varies greatly between fuels. You will now represent the relative amounts of resources used or pollutants generated by ethanol and biodiesel production and use in your diagrams.

You have 20 green, blue, red, orange, and yellow beads. Each represents a different resource used or pollutant generated in your life cycle diagram.

- Green = Fertilizer Use (20 beads)
- Blue = Water Use (20 beads)
- Red = Pesticide Use (20 beads)
- Orange = Energy Use (20 beads)
- Yellow = GHG Emissions (20 beads)

As a group, divide the beads among your two diagrams based on your predictions of the relative amounts of a pollutant produced or resource used. For example, you have 20 beads representing fertilizer use. Do you know something about corn (ethanol) that would lead you to believe that its cultivation requires more fertilizer than that of soy (biodiesel)? If so, place more green beads on your drawing representing ethanol production and use. Distribute all of your beads in a similar matter.

Tell your instructors when you have finished distributing the beads between your ethanol and biodiesel production diagrams. They will give you some actual values of the relative amounts of GHG emissions, and fertilizer, pesticide, energy, and water use involved in the life cycle of the two fuels. Use this data to redistribute the beads between your diagrams.

**Proper Bead Distribution:**

**Ethanol – 75 Beads Total**

- Green – 17 Beads
- Blue – 10 Beads
- Red – 17 Beads
- Orange – 15 Beads
- Yellow – 16 Beads

**Biodiesel – 25 Beads Total**

- Green – 3 Beads
- Blue – 10 Beads
- Red – 3 Beads
- Orange – 5 Beads
Questions:

How did your predictions compare to the actual values?

Are their certain inputs or outputs that you feel are less harmful for the environment? Justify.

What do you conclude about the relative environmental impacts of biodiesel and ethanol? Justify.

References:

Biofuel Life Cycle Analysis: Student Activity

Learning Goals:
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- Students will understand points of differentiation in the life cycles of biodiesel and ethanol

Background:

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Questions:

How did your predictions compare to the actual values?

Are their certain inputs or outputs that you feel are less harmful for the environment? Justify.

What do you conclude about the relative environmental impacts of biodiesel and ethanol? Justify.
Renewable Energy Debate: Student Activity

Learning Goals

- Students will understand the need to move beyond a petroleum based energy system.
- Students will understand the need to diversify the global energy portfolio.
- Students will be able to effectively communicate this new information by having a thorough understanding of differing viewpoints.

Scenario

In a heroic attempt to curb climate change, the City Council of Nocarbondale, Illinois is meeting to devise a plan to restructure their entire transportation system with economic stimulus funds. The entire town has been invited to this meeting, along with representatives from all of the major energy sectors and transportation niches.

The City Council (your teachers) is giving each group a chance to make a case for their approach to Nocarbondale’s new transportation plan. There will be time for discussion and questions after each presentation. At the end of the meeting the group will be asked to come to a consensus as to how the city will spend its funds. Will we be purchasing a whole fleet of ethanol ready cars for the town? Will we all be using public transportation? Will we be using petroleum for the rest of our lives?

Procedure

1. Students should organize themselves into teams.
2. Each team will be assigned an energy source or transportation alternative to research.
3. Teams will conduct Internet research on their topic. The goal of this research is to prepare a 5 minute presentation advocating your assigned topic. The team should also be prepared to answer questions and address concerns that may be raised by other teams. We have provided each group with a list of Internet resources to help you get started. Feel free to seek out additional sources.
   
   Research should focus on the cost, environmental impacts, and social impacts pertaining to:

   - Short-term benefits
   - Long-term benefits
   - Short-term concerns
   - Long-term concerns

4. Once your team is prepared for your 5 minute presentation and follow-up questions from the audience, sign-up for a presentation slot.
Sources of Information

Petroleum

http://www.eia.doe.gov/oil_gas/petroleum/info_glance/petroleum.html
http://www.goarticles.com/cgi-bin/showa.cgi?C=651190
http://www.opec.org/home/
http://www.api.org/

Public Transportation

http://www.publictransportation.org/
http://www.transitchicago.com/
http://people.hofstra.edu/geotrans/eng/ch6en/conc6en/ch6c4en.html

Natural Gas

http://www.eia.doe.gov/oil_gas/natural_gas/info_glance/natural_gas.html
http://www.eia.doe.gov/kids/energyfacts/sources/non-renewable/naturalgas.html
http://www.naturalgas.org/environment/naturalgas.asp
http://www.energyjustice.net/naturalgas/
http://www.afdc.energy.gov/afdc/fuels/natural_gas.html

Ethanol

http://www.afdc.energy.gov/afdc/ethanol/index.html
http://www.eia.doe.gov/kids/energyfacts/sources/renewable/ethanol.html
http://www.nrel.gov/learning/re_biofuels.html
http://www.eia.doe.gov/oiaf/analysispaper/biomass.html
Biodiesel

http://biodiesel.org/what-is-biodiesel/biodiesel-fact-sheets
http://www.afdc.energy.gov/afdc/fuels/biodiesel.html
http://www.nrel.gov/learning/re_biofuels.html
http://www.eia.doe.gov/oiaf/analysispaper/biodiesel/

Electric Car

http://www.fueleconomy.gov/Feg/evtech.shtml
http://www.afdc.energy.gov/afdc/vehicles/electric.html
http://www.evworld.com/index.cfm
http://phev.ucdavis.edu/

Fuel Cells

http://www.ucsus.org/clean_vehicles/technologies_and_fuels/hybrid_fuelcell_and_electric_vehicles/
http://www.fueleconomy.gov/feg/fuelcell.shtml
http://www.afdc.energy.gov/afdc/vehicles/fuel_cell.html
http://www.fuelcells.org/
http://www.nfcrc.uci.edu/2/default.aspx
Questions

1. During the debate which transportation alternative seemed like the most viable option? Why?

2. What was the final consensus of the group? Do you agree? Why, why not?

3. Popular media and scientific research both point to a future without petroleum based fuels. Is the only option to abandon petroleum as a fuel source? Why has petroleum been such a success as a transportation fuel?

4. What is the value of understanding all of the alternative transportation options available? Why not just pick the best one and throw everything behind it?