

Chemistry of Biodiesel Production

Background and Purpose

Biodiesel is one of the fuels that are being considered as an alternative to fossil fuels. The purpose of this lab is to examine the production of various biodiesels in small scale from a chemistry standpoint. Specifically, you will be looking at:

- The chemical reaction for producing biodiesel, known as *transesterification*
- Physical properties of the reaction products
- The energy output of the biodiesel

You will use information that you gather from the lab, as well as information you find in your own research, to craft an argument for or against using biodiesel versus “regular” diesel.

Materials

Sodium hydroxide/methanol solution
Oil samples (vegetable, olive, canola, corn)
Diesel sample
Four glass vials
10 mL graduated cylinders
Transfer pipets
Masking tape
Tub/ container (for storing vials overnight)
Gloves
Test tubes
Test tube rack
Wash bottle (with water)
Iron ring
Ring stand
Wire gauze
Evaporating dish
Thermometer
Matches
Tissues (5 cm square pieces)
Watch glass

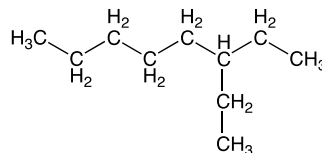
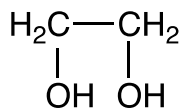
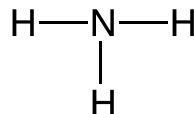
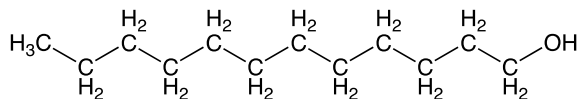
Safety

Gloves and goggles MUST be worn for both days of this lab. Use tongs to handle hot glassware/equipment. If any substance burns out of control, smother the flame using a large watch glass or sand, and notify the teacher. All waste should be dumped into the waste containers at your lab stations.

Prelab Questions

Read through both days of the lab (Day 1 AND Day 2), and answer the following questions.

1. Name the two substances that you will be adding to a vial on Day 1.
2. On Day 1, how long will you be shaking your vial containing your reaction mixture?
3. What analyses will you be doing on Day 2, and do they have to be completed in order?
4. Give one example of a polar substance, and one example of a nonpolar substance. How will these substances interact with each other if they are mixed together?
5. Look at the structural formulas below. Determine if each substance is polar or nonpolar, and explain.



Day 1: Biodiesel Synthesis - Procedure

** You will be working in groups of 4. There are four types of oils available; each group is responsible for working with one oil type. Each **person** will be responsible for working with 10 mL of your group's oil type, so each group will have 4 vials with the same oil at the end.

1. Obtain a glass vial. Fill it with 10 mL of the oil sample. Note down the type of oil that you use in the space below.
2. Into the same vial, put in 2 mL of the RED **sodium hydroxide/methanol** solution. Close the lid of the vial. Shake gently twice. Make at least two descriptions of the vial in the table below.
3. Continue to shake the vial for the next 15 minutes. About halfway through (7.5 minutes), add in another two descriptions. At 15 minutes, add in another two descriptions.
4. Let the vial sit for 15 minutes. Sketch and describe as you did before.
5. Label the top of your vial with your name and period; place the vial into the bin designated for the samples for your class.

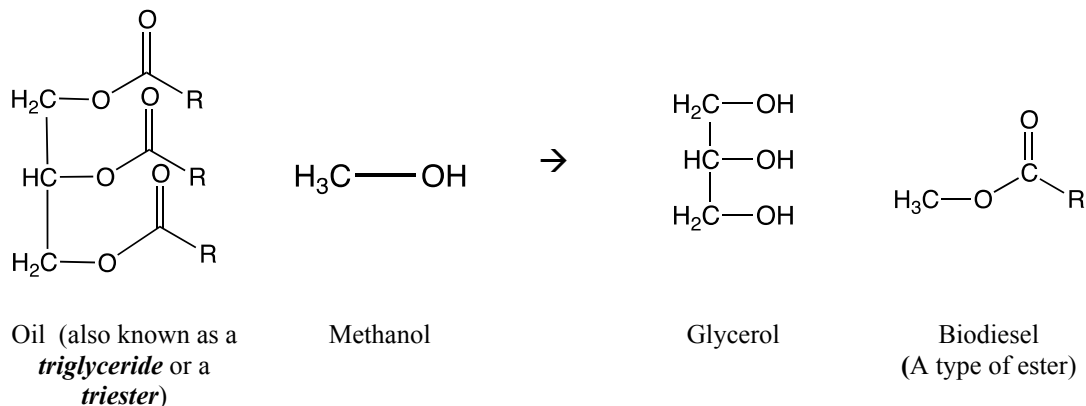
Day 1 Data Table

Oil sample type : _____

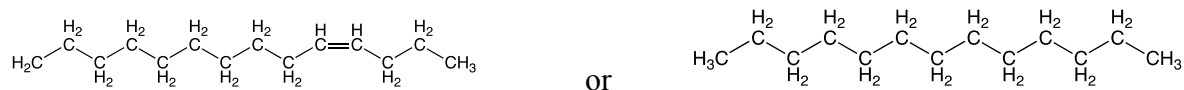
	Descriptions (2)
After 2 shakes	
7.5 minutes of shaking	
15 minutes of shaking	
15 minutes after shaking	

Day 1 Questions

The reaction for biodiesel is known as a *transesterification*. Use the equation below to answer the following questions.



Note: In the equation above, **R** stands for, generally speaking, a long-chain hydrocarbon group – for example:



Q1a: Look at the structural formulas for the products of this reaction. How do you expect the two products to behave when they are mixed together? Explain using the structural formulas for each product.

Q1b: The density of glycerol is 1.26 g/mL and the density of biodiesel is generally around 0.88 g/mL. Given your answer in Q1a, make a hypothesis as to what you should see the end of the reaction. Remember that hypotheses are three parts: *If* _____, *then* _____ *because* _____.

Q2. Draw out the part of the chemical structure that all *esters* seem to have in common [Hint: there are esters on both sides of the equation!]

Q3: Names of *alcohols* tend to have the suffix *-ol*. What chemical structure do alcohols seem to have in common?

Q4: Balance the equation for this reaction. Write the coefficients next to the structural formulas in the equation. Give the mole ratio between oil and methanol for this reaction below.

Q5: In this reaction, you used 10 mL of oil (molar mass = 900 g/mol; density = 0.92 g/mL) and 2 mL of methanol (density = 0.79 g/mL). What is the limiting reactant for this reaction? Justify with calculations.

Q6: In paragraph form, describe the transesterification reaction that you performed to make biodiesel. You will want to include information about:

- The balanced equation
- The limiting reactant for this reaction
- Esters and alcohols
- What you expect to see at the end of the reaction

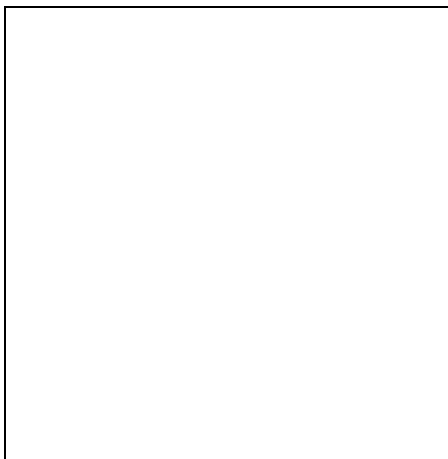
Day 2: Biodiesel Analysis - Procedure

Part A: Separating the products

**** The objective of this section is to combine all of the top and bottom layers from the vials for each person in your group. At the end of Part A, your group should have one beaker containing the top layer and one test tube containing the bottom layer. ****

1. Sketch out your reaction mixture below. Then, SLOWLY and CAREFULLY pour the top layer ONLY into one clean 100 mL beaker.
2. Tip the vial onto its side. Push the tip of a transfer pipet all the way to the bottom, and take out as much of the bottom layer as possible. Put the bottom layer into a test tube and let the solution sit inside the test tube for a few minutes.
3. After a few minutes, if two layers form in the test tube, remove the top layer from the test tube with a pipet and put it into the waste container.

Part A Observations and Questions



Sketch of reaction mixture

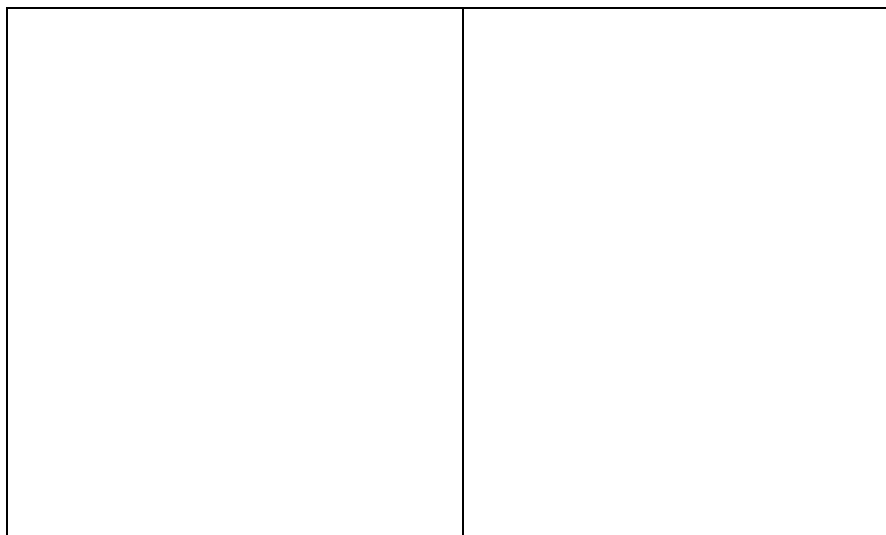
Q7: Review your responses from Q1a and Q1b (from Day 1). Propose a way to test your hypothesis that requires three steps or less, using the materials that you have in front of you. Explain your reasoning behind these procedures. **Your instructor will need to approve this before you move on!**

Part B: Analyses

The following analyses can be done in any order. Your group should split up the tasks so that you have enough time to finish.

Testing Question 7

1. Run the test that you proposed. Sketch out your observations below.



Test of top layer

Test of bottom layer

Q8: Relate what you expect to see (see Q1a/Q1b) with what you saw in your tests. What can you conclude about the top and bottom layers?

Density

1. Take an empty graduated cylinder to the electronic balance. Zero out the mass of the graduated cylinder.
2. Add 20 mL of your top layer and determine its mass.
3. Take 20 mL of your original oil, and determine its mass.
4. (IN THE FUME HOOD,) Take 20 mL of diesel, and determine its mass.
5. Determine the densities for your top layer, oil, and diesel.

Density Data

	Top layer	Diesel	Original oil
Mass			
Volume			
Density			

Q9: Give one way that this test supports what you concluded as the identity of the top layer. Give one way that this test may not support what you concluded. Explain.

Calorimetry

1. Fill a 50 mL beaker with 15 mL of water. Record the temperature of the water.
2. Set up a ring stand/ iron ring/ wire gauze assembly.
3. Put 1 mL of the biodiesel into an evaporating dish.
4. Take a small piece of tissue (5 cm square) and do a loose fanfold.
5. Place the folded tissue into the evaporating dish, on top of the liquid in the dish. Make sure that at least part of the tissue is touching the biodiesel at the bottom of the dish. Place the evaporating dish containing the biodiesel and tissue underneath the wire gauze/ iron ring. **Adjust the wire gauze/iron ring assembly is as close to the evaporating dish as possible.**
6. Light the tissue; let the tissue/biodiesel burn out. Record both start time and end time. Record the temperature of the water after burning is complete.
7. Repeat the procedure; this time, using 1 mL of diesel.
8. Calculate the amount of heat released into the water through burning the biodiesel/diesel. Show work!

Calorimetry Data

	Biodiesel	Diesel
Initial water temp		
Start time		
End time		
Total burn time		
Final water temp		
Amount of water (grams)		
Heat released into water (show work!)**		

** Remember that $q = mc\Delta T$ and the specific heat capacity of water is $4.18 \text{ J/}^\circ\text{C}\cdot\text{g}$.

Day 2 Questions

Q10: For the calorimetry section, you calculated the amount of heat that is released into the water. Calculate the amount of heat released per mL of biodiesel burned. Also calculate the amount of heat released per gram and per gallon of biodiesel burned. Note that 1 gallon is approximately 3.79 L.

Q11: How is making calculations for the amount of energy per mL/gram/gallon of biodiesel burned helpful? Give at least one specific example for each unit (mL, gram, and gallon).

Q12: “Biodiesel has the highest Btu [*British thermal units, which is another unit for energy like joule or calorie*] content of any alternative fuel, though it is slightly less than that of diesel.¹” What additional information will you need in order to evaluate this statement?

Q13: You were asked to determine the total burn time for both the biodiesel and the diesel. Recall that both biodiesel and diesel are used to fuel cars/buses. What are the consequences of having a fuel burn quickly? Slowly?

Q14: Recall how you set up the calorimetry portion of this lab. What major assumptions did we make when we calculate the amount of energy released? Were they all correct? How do these assumptions affect the ways that we interpret the results of our tests?

Q15. You performed three separate tests to analyze the biodiesel. Using the information that you have gathered, describe biodiesel. How does it compare to diesel and vegetable oil? *Based on the data from the lab*, does biodiesel appear to be a “better” fuel, or does diesel?

¹ Page 13, *Transportation Fuels: Biodiesel*. NEED Project: Manassas, VA. Retrieved on July 13, 2011, from <http://www.need.org/needpdf/Biodiesel.pdf>