Chemistry of Biodiesel Production

Teacher Notes

DAY 1: Biodiesel synthesis (50 minutes)

NOTE: The lab preparation instructions / lab protocol assumes classes of 32 students, with 8 groups of 4 students each.

MATERIALS LIST (for 1 class of 32 students/ 8 groups, @120% amount needed)

- 80 mL methanol
- 1.4 g sodium hydroxide pellets
- Red food coloring
- Vegetable oil (80 mL)
- Corn oil (80 mL)
- Canola oil (80 mL)
- Olive oil (80 mL)
- 40 glass vials (20 mL capacity or above, with screwcaps;)
- Graduated cylinders – one per reagent aliquot
- Transfer pipets – one per reagent aliquot
- Masking tape
- Tub/ container (for storing vials overnight)
- Gloves

TEACHER PREPARATION

1. Dissolve 1.4 g sodium hydroxide pellets in 80 mL of methanol in a 250 mL Erlenmeyer flask. Put in a few drops of red food coloring. Let the solution sit overnight; the sodium hydroxide pellets should dissolve. Alternatively, sodium hydroxide pellets can be pulverized and dissolved in methanol the day of the lab; allow for about 15 minutes with stirring. Label this solution **METHANOL/ SODIUM HYDROXIDE**. On the day of the lab, split this amount into at least 2 aliquots; for each aliquot, provide one transfer pipet and one 10 mL graduated cylinder.

2. Split each kind of oil into at least two aliquots; for each aliquot, provide one transfer pipet and one 10 mL graduated cylinder.

3. Spread out the reagents (methanol/sodium hydroxide; the different oils) strategically throughout your lab area.

4. [Optional] For each lab station, set out 4 vials and masking tape (for labeling the vials)

DAY 2 LESSON PLAN

Time	Teacher/student actions
45 minutes	**Lab Day 1** Each group of students will be synthesizing biodiesel with 4 different types of oils: 1. Olive oil (unused) 2. Canola oil (unused) 3. Vegetable oil (unused) 4. Corn oil (unused) Each student group will be responsible for one oil. Each student will contribute by making ~10 mL of biodiesel. Students will pool their products to get ~ 40 mL biodiesel. See lab handout for protocol. TECHNICAL/PROCEDURE NOTES [unfinished!]: 1. Gloves are strongly recommended. This minimizes contact with the methanol/sodium hydroxide solution as well as any reaction mixture that can spill out as students shake the vials. 2. The food coloring was introduced into the sodium hydroxide/methanol mixture to increase contrast between the biodiesel layer and the glycerol layer at the end of the reaction. After 24 hours, the food coloring should settle to the bottom, along with the glycerol; the biodiesel layer should be on top. We expect approximately a 10:1 biodiesel:glycerol ratio by volume. 3. The solution should be turbid initially. The will be a noticeable change in viscosity about 2-3 minutes into the shaking. 15 minutes after the shaking stops, a red layer will settle out of solution, along with a light red liquid at the top. Over 24 hours, all of the red coloring will settle to the bottom, with most of the liquid volume inside the vial being yellow.
5 minutes	**Assign homework** Have students finish questions for homework. They will need the answers to two of the questions in order to continue with the lab on Day 3.

DAY 3: Biodiesel analysis (50 minutes)

NOTE: The lab preparation instructions / lab protocol assumes classes of 32 students, with 8 groups of 4 students each.

MATERIALS LIST (for 1 class of 32 students/ 8 groups, @120% amount needed)
Biodiesel vials (from last period)

Electronic balances (4 minimum)
Transfer pipets (40)
Waste beakers (8)
Matches (8 boxes)
Tissue paper strips (40), cut at 5 cm square. One-ply toilet paper will work.
Ring stand (8)
Wire gauze (8)
Wash bottles (8)
50 mL beakers (8 minimum, more if available)
10 mL graduated cylinders (8 minimum, more if available)
50 mL graduated cylinders (8)
Small test tubes (80)
Test tube racks (8)
Oil samples (from last period)
12 mL diesel sample (buy from gas station)

TEACHER PREPARATION

1. **Buy diesel sample from gas station. For 5 classes of 32, you will need a total of about 60 mL **
2. Leave oil setups from last period.
3. For each lab group, set out the following:
    - 5 transfer pipets
    - 1 waste beaker
    - 1 box of matches
    - 5 strips of tissue paper
    - 1 ring stand
    - 1 wire gauze
    - 1 wash bottle
    - 1-4 - 50 mL beakers
    - 1-4 - 10 mL graduated cylinders
    - 1 - 50 mL graduated cylinder
    - 1 test tube rack
    - 10 small test tubes
4. Set out balances and biodiesel vials.
5. Set out at least 2 aliquots of diesel strategically around the lab area.

DAY 2 LESSON PLAN

Time	**Teacher/student actions**
50 minutes	**Lab Day 2** Each group will perform the following analyses on their samples of biodiesel: 1. Solubility 2. Density 3. Calorimetric (burn analysis) See lab handout for protocol.

	TECHNICAL/PROCEDURE NOTES: 1. The intention behind having students separate the two layers as the protocol described is so that they can do solubility testing on the two layers. The red layer technically can be discarded, as it is not the target product [glycerol, food coloring] 2. You will need to sign off on students' procedures on how they will test for polarity differences between the biodiesel [top] layer and the glycerol [bottom] layer. 3. **IMPORTANT: Depending on your student population, this day may take one or two 50-minute periods. You will want to tell students to organize themselves efficiently and split up tasks among themselves. *** 4. Since all groups will be using different types of oils, you may want to pool the data for your classes for your different types of oils/ biodiesels for comparative purposes.

CLEANUP/DISPOSAL:

Sequester all waste.
Used glassware should be soaked in soapy water and then washed.

Chemistry of Biodiesel Production KEY

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.

Methanol/sodium hydroxide; vegetable oil

2. On Day 1, how long will you be shaking your vial containing your reaction mixture?

15 minutes

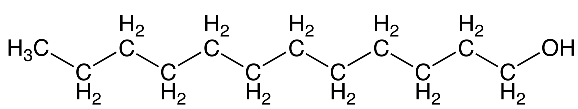
3. What analyses will you be doing on Day 2, and do they have to be completed in order?

Testing Question 7, density, calorimetry

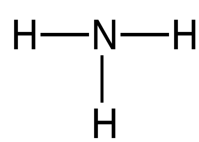
4. Give one example of a polar substance, and one example of a nonpolar substance. How will these substances interact with each other if they are mixed together?

Polar substance: water; nonpolar substance: oil. If mixed together, these substances will form two layers/ will not dissolve in each other (immiscible).

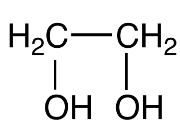
5. Look at the structural formulas below. Determine if each substance is polar or nonpolar, and explain.



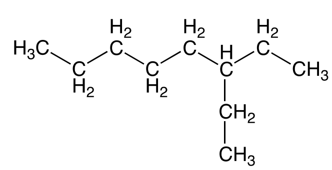
nonpolar: even with OH group at one end, long-chain C-H → nonpolar



Polar: N-H



Polar: multiple OH groups; relatively short C-H chain



nonpolar: C-H only

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

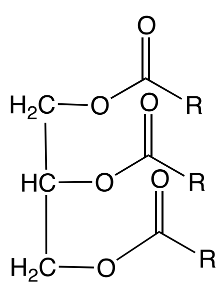
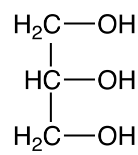
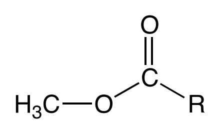
Oil sample type :____

	Descriptions (2)
After 2 shakes	**Red layer should remain floating at the top, with a few droplets dispersed throughout bottom** Mixture hard to mix (viscous)
7.5 minutes of shaking	Red liquid; cloudy; somewhat less viscous (easier to shake).
15 minutes of shaking	Red liquid; relatively clear; somewhat less viscous (easier to shake).

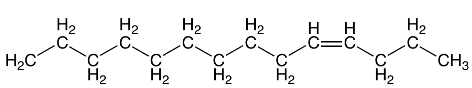
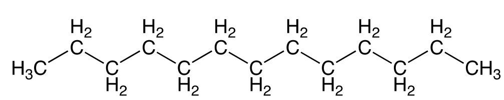
15 minutes after shaking	Red liquid with darker red layer settling at the bottom; clearer than at 7.5 minutes

Day 1 Questions

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

	H_3C—OH	→		
Oil (also known as a ***triglyceride*** or a ***triester***)	Methanol		Glycerol	Biodiesel **(**A type of ester)

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

 or 

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.

Glycerol has a short C-H chain with OH groups; biodiesel has a relatively large C-H chain. These two products will not dissolve in each other (will not be miscible in each other) because we can predict that glycerol is most likely a polar molecule and biodiesel a nonpolar one.

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* _________.

If glycerol and biodiesel were put into the same mixture, then they will not be miscible in each other and will separate into two layers – with biodiesel as the top layer and glycerol as the bottom layer. This is because glycerol and biodiesel are polar and nonpolar compounds, respectively, and glycerol has a higher density than biodiesel.

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!]

-C(O)OR

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

--- OH

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.

Coefficients are 1, 3, 1, 3. Mole ratio is 1:3.

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.

LR is oil

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

Answers will vary.

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

Two separate layers; one yellow and one red. Yellow layer has about 10x more volume than red layer.

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!**

Add water to both.

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
Immiscible in water.	Miscible in 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?

The top layer should be biodiesel, while the bottom layer should be glycerol.

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			

Answers will vary, but density of biodiesel should be about 0.88, diesel about 0.85, and vegetable oil should be about 0.92.

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.

We know that the top layer is definitely not glycerol (density figure not close to density of glycerol). We also do not know whether the top layer is actually biodiesel or is vegetable oil, since the densities are within error.

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!)**		

(temp change should be about 15 deg C for both biodiesel and diesel, but diesel should have a faster burn time).

** Remember that q = mc DT and the specific heat capacity of water is 4.18 J·g°C.

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.

Answers will vary.

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).

Calculating amount of energy per mL is helpful because there is a relative measurement of energy per unit volume. We extend this to gallons because in the United States, fuel is sold per gallon; elsewhere in the world, fuel is sold per L, so the mL measurement is also helpful in that sense. Since there is also a weight consideration to transportation (more weight = more energy needed to transport) and fuel does have weight, a calculation of energy released per gram is also helpful.

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?

We also need a relative measurement – that is, we need to know whether this amount is per unit volume or per unit mass or some other "amount" measurement.

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?

A fuel that burns quickly could mean that safety is an issue - that it is much more easily ignitable. A fuel that burns too slow may not release energy quickly enough.

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?

We assumed that all of the energy released from the fuel burn is transferred into the water, which is not correct. We also assumed that the energy released from the burning was all from the diesel/biodiesel burning, but the burning of the wick also releases energy. We cannot look at the amount of energy as is; we can only make a comparison between diesel and biodiesel. That is, we cannot conclusively say that biodiesel releases X energy per mL because we did not "capture" a significant amount of it.

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?

Answers vary.

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