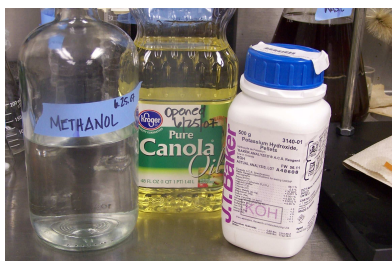


EQUILIBRIUM & LE CHATELIER: Two-Phase Method of Making Biodiesel

Georgia Performance Standards: SCSH1-9, SC2 a & b, SC5 b, SC7 b

National Science Standards: Content Standards A, B: chemical reactions, F: natural resources

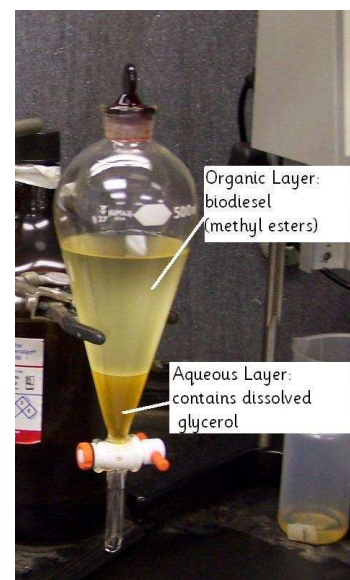
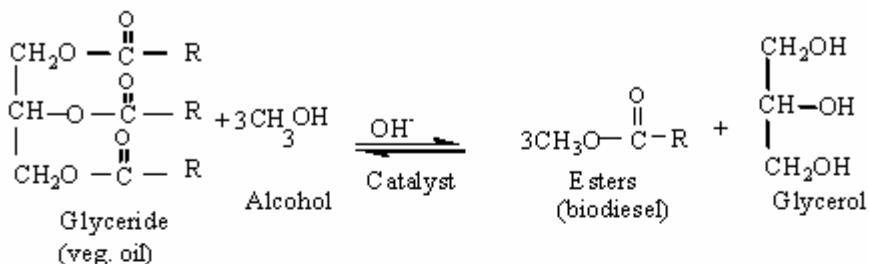


Objective: Students will make another batch of biodiesel using a two-phase process. They will compare their percent yield with the percent yield of the single phase process used in the 'stoichiometry' lab activity.

Essential Questions:

- What is LeChatelier's Principle?
- Why would using a two-phase method be more useful?

Background: Biodiesel is made by a chemical reaction called *transesterification*. It is made from a triglyceride (a fat or oil), which is sometimes called the "feedstock." The feedstock fat/oil is reacted with an alcohol, such as methanol, in the presence of a catalyst (sodium hydroxide). The reaction yields esters (methyl esters if using methanol) and glycerol.



*Above picture comes from http://www.esru.strath.ac.uk/EandE/Web_sites/02-03/biofuels/what_biodiesel.htm

The products must settle for several hours in order to separate the organic from the aqueous. This is usually done in a separatory funnel where the esters separate into the top layer and the glycerol dissolves in the bottom layer. The esters are the *biodiesel* and the glycerol can be put in a landfill or burned in boilers as fuel.

In the two-phase reaction, 80% of the methoxide is added to the oil and reacted. The glycerol is removed, and then the last 20% of methoxide is

added to what was already reacted and it is reacted again. The glycerol is then removed the second time leaving you with a batch of crude biodiesel.

- Materials:**
1. 250 mL Soybean, Canola, or other oil from a grocery store
 14. 50 mL Methanol
 15. 2.25 g KOH
 16. 125, 500 mL Erlenmeyer Flask
 17. 100 mL graduated cylinder
 18. balance
 19. Stir/Heat plate
 20. Stir bar
 21. Thermometer
 22. 250 mL or 500 mL Separatory funnel**
 23. pH paper
 24. 2 or 3 50 mL beakers

Procedure: The procedure for the two-phase method is much the same, with an extra reaction step added in. This method is used in industry regularly.

Record your hypothesis before you begin.

Part I: Reaction

1. Make sure all glassware is CLEAN and DRY.
2. Measure out 50 mL of methanol using your graduated cylinder (make sure you record the exact volume you use).
3. Weigh out 2.25 grams of KOH using the electronic balance. (Make sure you record the exact mass you use)
4. Put the methanol and KOH into the 125 mL Erlenmeyer flask. Add the magnetic stir bar and cover with Parafilm.
5. Place the flask on the stir plate and turn on to a medium speed. (You want a good vortex but without splashing.
6. Stir the mixture until the KOH dissolves into the methanol (this may take a little while).
7. Once the KOH has dissolved, you have a substance called potassium methoxide.
8. Add 250 mL of vegetable oil to a 600 mL beaker and record the actual amount of oil in your lab notebook. (To calculate the mass of the vegetable oil, use the density 0.90 g/mL). Add a stir bar and 40 mL of the methoxide mixture.

9. Turn the heat up until the mixture reaches about 55-60°C and stir at a medium speed for 30 minutes.
10. When the mixture has reacted after about 30 minutes, pour it into a separatory funnel and let it sit for about 10-15 minutes.

Part II: Separation

11. The bottom layer is the glycerol layer that you do NOT want; the biodiesel is in the top layer. Slowly drain the bottom layer into a small beaker by turning the stopcock until the liquid starts to flow. YOU MUST REMOVE THE STOPPER AT THE TOP OF THE FUNNEL TO GET IT TO DRAIN. When you see that the biodiesel layer is getting close to the bottom of the funnel, turn the stopcock so only one drip comes out at a time (you can put the top back on the funnel to help you). When you see the first drop of biodiesel leave the funnel, close the stopcock completely.
12. Empty the biodiesel layer back into the 500 mL Erlenmeyer flask (the one you used in step 8).
13. Add the remaining 10 mL of methoxide to the biodiesel you just drained, add a stir bar, and follow steps 9-11 again.
14. What you now have is CRUDE biodiesel. Since we did not wash it, it is NOT the high quality fuel you would need to put into a vehicle.
15. Weigh the clean bottle you brought (including the cap). Drain the biodiesel out into the clean bottle and mass it again (don't forget to include the cap).

Data and Calculations: This is exactly the same as the data and calculations from the first time you made biodiesel using a single-phase method.

1. In your data section make sure you have recorded any physical observations you see as you do the lab (color change, texture change, temperature change, etc). Also make sure to include the masses of all your starting materials and your final product.
2. Using the equation given to you above (take note of the coefficients – the equation is already balanced), calculate the limiting reactant and the theoretical yield of the biodiesel (methyl esters). Use the approximation of molar mass of the oil (glyceride) to be 885.4 g/mol.

3. Using your actual yield, calculate the percent yield of your reaction.

Questions:

1. Compared with the first time you made biodiesel, do you notice any difference in the physical changes you observe?
2. Explain how LeChatelier's principle is related to the two-phase method of making biodiesel.
3. What was the percent yield of your biodiesel the first time you made it? What was the percent yield of the biodiesel this time that you made it? Did you see a difference? Why?
4. Could you use a three-phase method? If so, how would that help?

Assessment: Lab report rubric

Lab Data Sheet – Equilibrium

Hypothesis:

Actual volume of methanol used: _____ mL

Actual mass of KOH used: _____ g

Actual volume of oil used: _____ mL

Calculated mass of oil used: _____ g

Show calculation here –

Mass of bottle (and cap) empty: _____ g

Mass of bottle (and cap) + biodiesel: _____ g

Physical observations:

(Note any physical differences between the two phases of the reaction as well).

Coefficients of:

Oil _____

Methanol _____

Biodiesel _____

Calculation of limiting reactant:

Calculation of theoretical yield:

Calculation of percent yield:

Make a concluding statement comparing the percent yield from the single-phase reaction with the percent yield from the two-phase reaction.

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Instructor Notes

See the instructor notes for the stoichiometry lab activity.

The optimum reaction time for the oil and methoxide is 45-60 minutes. Time constraints of class periods make this difficult so the shortened times are used. If you can break up the lab into a couple of lab periods you may be able to react the oil and methoxide for longer.

If you do not have access to separatory funnels you can use unitary wash bottles as suggested in the Stoichiometry lab.