Biodiesel Reaction Efficiency (27/3 Test)

Learning Goals:
- Students will understand variables that effect 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 make biodiesel through a transesterification reaction and test the efficiency of two different production techniques.

The two biodiesel samples will differ in that the first will consist of a single phase, stoichiometric reaction. The second sample will use a purposeful overload of chemical reagents and consist of a two stage (split 75/25) reaction with glycerin removal between stages. This second process incorporates Le Châtelier’s principle of chemical equilibrium. By disturbing chemical equilibrium (overload of reagents and removal of a product) the reaction moves closer to completion, giving us a purer biodiesel product.

This can be tested in the classroom with the 27/3 test for glycerin. The test gives a qualitative analysis of residual glycerin in the biodiesel product, a sign of incomplete reaction. By overloading the biodiesel sample with methanol we drive the reaction to completion, thus releasing any residual glycerin that is in the biodiesel (either free floating or bound in mono- or di-glyceride molecules). In theory, we will see a larger quantity of glycerin in the first biodiesel sample than the second thus demonstrating that Le Châtelier’s principle results in a more complete, efficient chemical reaction.
Materials – Sample 1:
- Ball Jar
- 500 mL Graduated Cylinder
- 50 mL Graduated Cylinder
- Scale
- 200 mL Vegetable Oil
- 25 mL Methanol
- 1.5 gram KOH or 1 gram NaOH

Materials – Sample 2:
- Ball Jar (x 2)
- 500 mL Graduated Cylinder
- 50 mL Graduated Cylinder
- Scale
- 250 mL Separation Funnel or Pipette/Eye Dropper (You will some way to remove the glycerin from the 1st reaction from off the bottom of the Ball Jar)
- 200 mL Vegetable Oil
- 40 mL Methanol (30 mL for 1st Reaction and 10 mL for 2nd Reaction)
- 1.6 grams KOH (1.2 for 1st Reaction and 0.4 for 2nd Reaction) or 1.2 gram NaOH (0.9 grams for 1st Reaction and 0.3 grams for 2nd Reaction)

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

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.
Sample 1
1. Measure methanol into Ball Jar
2. Weigh and add KOH (or NaOH, either one will work)
3. Cap the jar and shake until the KOH is fully dissolved
4. Measure and carefully pour vegetable oil into the jar
5. Cap and shake for 10 minutes
6. Allow this reaction to settle overnight
   a. The glycerin portion of the vegetable oil molecule has been removed and replaced with
      the methanol molecule. The glycerin is denser than the biodiesel so over the next few
      hours it will settle to the bottom of the jar.

Sample 2
1. Measure 1st Reaction methanol into Ball Jar
2. Weigh and add 1st Reaction KOH (or NaOH, either one will work)
3. Cap the jar and shake until the KOH is fully dissolved
4. Measure and carefully pour vegetable oil into the jar
5. Cap and shake for 10 minutes
6. Allow this reaction to settle for at least an hour
   a. Overnight would be better
   b. The glycerin portion of the vegetable oil molecule has been removed and replaced with
      the methanol molecule. The glycerin is denser than the biodiesel so over the next few
      hours it will settle to the bottom of the jar.
7. Now we need to remove the glycerin from the bottom of the jar before adding the rest of our
   chemical reagents. This can be accomplished by transferring the biodiesel and glycerin to a
   separation funnel and draining the glycerin from the bottom, or by pouring the biodiesel off the
   top of the jar into a second jar (being careful not to pour glycerin into the jar). Then use a
   pipette/eye dropper to carefully get all of the biodiesel out of the jar (try to get all of the
   biodiesel even if you get a few mL of glycerin too).
8. Measure 2nd Reaction methanol
9. Weigh 2nd Reaction KOH
10. Combine the 2nd Reaction methanol and 2nd Reaction KOH in a Ball Jar or in a graduated cylinder.
11. Allow the KOH to dissolve
12. Add to the biodiesel jar
13. Cap the jar
14. Shake for 10 minutes
15. Allow this reaction to settle overnight

Reaction Efficiency Test - 27/3 Test
Both biodiesel production techniques will produce a biodiesel (top) and glycerin (bottom) layer.
However, Sample 2 will have removed more glycerin from the biodiesel layer (total of the glycerin from
both reactions).
1. Measure 27 mL of methanol  
2. Add methanol to small sample jar  
3. Measure 3 mL of Sample 1 biodiesel  
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