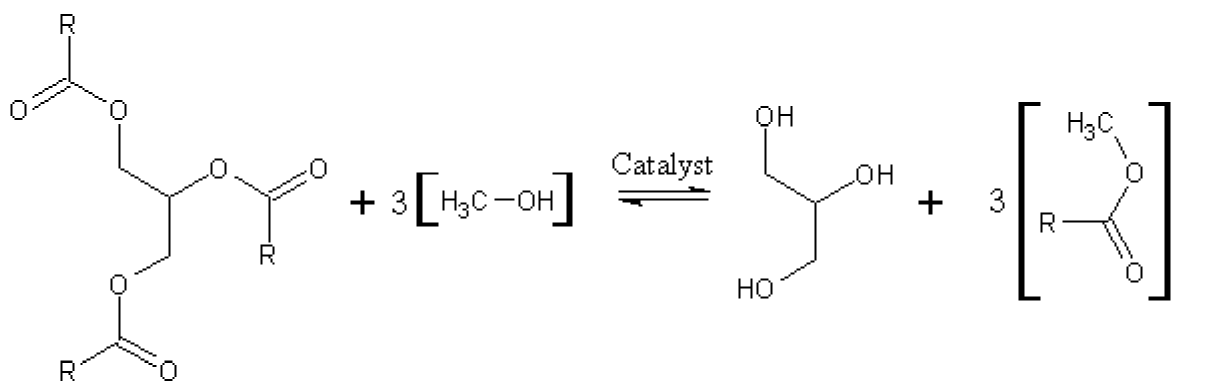
# Biodiesel

Experiment 11

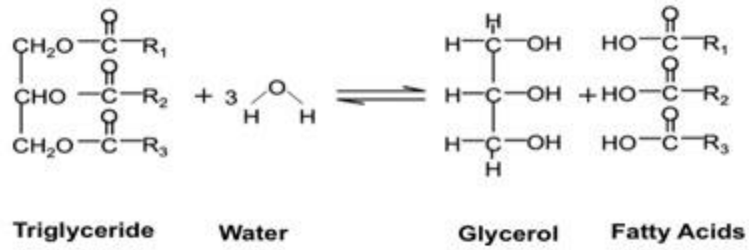
**Introduction**

*Alternative fuels* and *alternative fuel vehicles* are becoming more popular among car manufactures as the price of petroleum based fuels continue to rise. Alternative fuels are derived from resources other than petroleum. Some examples of alternative fuels are: ethanol, propane, compressed natural gas (CNG), hydrogen, and biodiesel. Alternative fuel vehicles are vehicles that either reduce or eliminate the use of petroleum based fuels. Some examples of alternative fuel vehicles include: hybrid electric, electric, solar, natural gas, biodiesel and hydrogen fuel cell cars. Some of the earlier biodiesel cars involved converting a regular diesel car into a duel tank petroleum diesel vegetable oil system which required the heating of the viscose vegetable oil before mixing with the petroleum diesel. Vehicles that involve the mixing of more than one fuel type are called *flex-fuel* vehicles (FFV). Converting vegetable oil to a less viscose substance eliminates the need of a flex-fuel system, thus may eliminate the need to use petroleum based diesel. Vegetable oil contains triglycerides which are bulky long greasy fatty acid molecules. Upon the base catalyzed *transesterification* reaction of the triglyceride with methanol, the triglyceride is converted to glycerol and the methyl ester of the fatty acid chain.




Triglyceride Methanol (3) Glycerol Methyl Esters (3)

A more *eco*-friendly approach to making biodiesel involves *upcycling* waste vegetable oil (WVO) as the source of vegetable oil. Upcycling is the process of repurposing used or waste materials into useable products. WVO can be recovered from cafeteria or fast food deep fryers. One problem with using recovered waste oil is that they decompose upon heating in the presence of water to undergo a hydrolysis reaction. The products of the hydrolysis reaction are glycerol and free fatty acids (FFA's). The free fatty acids contain a carboxylic acid group which can react with the base catalyst creating the salt of the conjugate base (soap), thus using up the catalyst and lowering the yield of the reaction.




In this experiment, you will synthesis biodiesel from waste vegetable oil. First step would be the purification of the oil by filtration. The next step would be to determine the free fatty acid content by titration with NaOH indicated with phenolphthalein. Once the correct amount of catalyst is calculated from the titration, the oil is reacted with methanol and catalyst to form glycerol and biodiesel. The products are then separated and the biodiesel is washed with water to remove the catalyst and soap.

**Equipment and Materials**

| **Day 1** | | **Day 2** |
|---|---|---|
| Waste vegetable oil | 0.1% NaOH (1g/1000mL) | activated charcoal |
| Isopropanol | 250 mL Erlenmeyer flask | Anhydrous $MgSO_4$ |
| Phenolphthalein | magnetic stir bar | filter funnel |
| Methanol | Hot/Stir plate | filter paper |
| NaOH (pellets) | 250 mL separatory funnel | Hot plate |
| 1 mL Pipet | Thermometer | Thermometer |
| filter paper | vacuum funnel | parafilm (5 x 5 square) |
| 250 ml vacuum flask | 50 mL Erlenmeyer flask | test tube (~10 mL) |

**Procedure**

**(Day 1)**

<u>Part 1 - Filtration</u>

1. Set up a vacuum filtration using the 250 mL vacuum filtration flask and filter paper with funnel.
2. Obtain approximately 150 mL of waste vegetable oil (WVO) in a 250 mL beaker. Carefully pour the waste oil into the filter.
3. Filter the oil through the filter while pulling vacuum.

<u>Part 2 – Titration</u>

1. To a 50 mL Erlenmeyer flask, pipet 1 mL of the WVO, add 10 mL of isopropanol and 2 – 3 drops of phenolphthalein indicator. Swirl the flask to make sure all of the oil dissolves.
2. Fill a buret with 0.1% NaOH solution. Record the initial buret reading.
3. Titrate the WVO dropwise with the NaOH solution until the pink end-point is reached. Record the final buret reading of the base solution.
4. Calculate amount of NaOH used in the titration.

$$\text{Volume (mL)} = V_{final} - V_{initial}$$

$$Mass\ of\ NaOH_{titration} = Volume\ (L) \times \frac{0.025\ mol}{L} \times \frac{40\ g}{mol}$$

This amount is for a 1 mL sample of WVO. Multiply this number by 100 to determine the amount needed for a 100 mL sample.

$$\text{Extra NaOH needed} = \text{mass of } NaOH_{titration} \times 100$$

Part 3 – Synthesis

1. In a 150 mL beaker on the centigram balance, weigh out 0.5 grams of solid NaOH plus the extra NaOH mass calculated from the titration.
2. Add about 20 mL of methanol to the beaker and a magnetic stir bar. Place the beaker on the stir plate and stir until all of the solids dissolve. ***Caution****: mixing is an exothermic process so make sure that proper eye protection is worn at all times*. Remove from the stir plate when all of the solids are dissolved.
3. Measure 100 mL of the filtered WVO into a 100 mL graduated cylinder. Pour into a 250 mL Erlenmeyer flask and heat on the hot plate to about 60 °C.
4. Add the methanol-NaOH solution into the Erlenmeyer flask along with the stir bar. Gently stir the solution for about 30 minutes at about 60 °C. The solution should become cloudy at this point. Try not to stir the solution too high that it becomes frothy.
5. Set up a separatory funnel on a ring stand with an iron ring. Pour the contents from the Erlenmeyer flask into the separatory funnel and allow to settle for 10 minutes. There should be two layers present. The top layer is the biodiesel and the bottom layer is the glycerol. Place the glycerol in a glycerol waste container.
6. Drain the glycerol from the bottom layer into a waste beaker. Place the glycerol in a glycerol waste container.
7. Wash the biodiesel by slowly pouring about 20 mL of DI water into the separatory funnel. Allow to settle for 10 min and drain the water (bottom layer) from the funnel. Repeat this process 2 more times.  Discard the water down the drain.
8. Transfer your biodiesel product into a clean dry 250 beaker and place a watch glass over the top. Store until next period.

**(Day 2)**

Part 4 – Purification

1. Remove the water from the biodiesel by heating the product in the 250 mL beaker on the hot plate at about 80 °C for 20 minutes. Allow to cool for 10 min.
2. Add a scoop of anhydrous magnesium sulfate ($MgSO_4$) and a scoop of activated charcoal to the biodiesel. Swirl to mix the contents.

3. Set up a filter funnel on an iron ring and place a flute folded filter paper inside. Filter the warm biodiesel product into a clean dry 250 mL flask. Clean dry biodiesel should be clear.

<u>Part 5 – Analysis</u>

*Gel Test* - If all of the water has been removed from the product, the biodiesel should gel when cooled.

1. Transfer approximately 2-3 mL of the biodiesel into a test tube. Place the test tube into a beaker containing ice and tap water. Place a thermometer into the test tube.
2. After 15 min, remove the test tube and record the temperature when the solution becomes clear. If the biodiesel does not gel, then it is not dry. Record your observations.

*Methanol Test* – If all of the WVO triglycerides were converted into glycerol and biodiesel, then the product should completely dissolve into methanol.

1. Transfer approximately 1 mL of the biodiesel into a test tube containing about 5 mL of methanol. Cover the top of the tube with parafilm and gently invert the tube several times.
2. Allow the mixture to sit for 15 min. If there is no triglyceride starting material, then all of the product should dissolve into the methanol. Record your observations.
3. Take the tube and shake several times. Allow to sit for 5 min. If there is a layer of foam, then the product contains soap impurities.

*pH Test* – Tests for the presence of NaOH.

1. To about 1 mL (20 drops) of DI water add 5 drops of biodiesel product into a test tube. Mix the components by lightly shaking from side to side.
2. Place a drop of this mixture onto a strip of universal pH paper. Record the pH value. The ideal pH of the product should be around 7.

Discard of the biodiesel into the Polyethylene Terephthalate (PETE) waste container (used soda or water bottle).

**Report**

Include all data and calculations.

Include all observations.

Discuss the purity of the product and quality of the WVO starting material.