

## **Biodiesel Mini-Batch Procedure**

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### **Be Safety Conscious**

Before beginning any experiment, it is critical to follow proper safety procedures. Locate emergency eyewash and shower stations prior to beginning the experiment. Locate fire extinguishers and make sure they have current inspection tags. Read all Material Data Safety Sheets (MSDS) as well as safety labels on any other materials being used in the experiment. For this experiment, MSDS sheets for methanol ( $\text{CH}_3\text{OH}$ ), sodium hydroxide ( $\text{NaOH}$ ), and potassium hydroxide ( $\text{KOH}$ ) should be reviewed prior to beginning the experiment.

These are dangerous and poisonous chemicals, and therefore the utmost care should be given to safety.

**Methanol** is a poison that can be absorbed through your skin and has the potential to be inhaled (vapor form). Methanol can cause blindness or death and is as flammable as gasoline. Cartridge respirators do not work with methanol.

**Sodium hydroxide** ( $\text{NaOH}$ ) and potassium hydroxide ( $\text{KOH}$ ) are from the caustic soda family of chemicals and can cause severe burns and death.

### **Wear Protection Gear**

All individuals involved in the experiment should wear protective gear that protects eyes (safety goggles), hands (solvent resistant gloves), and one's body (lab coats or aprons). Have running water readily available to wash off any splashes immediately.

**Do not proceed without proper protective gear and trained supervisors.**

### **Review all Relevant Safety Related Protocols before Beginning the Experiment**

After making sure all safety precautions are in place, the biodiesel making process can begin.

## 1-liter Mini-Batch Procedure

### Step #1

Obtain vegetable oil for conversion. This can be either filtered waste vegetable oil (WVO) or virgin cooking oil (VCO). If using WVO, make sure it has been filtered, i.e., it is free of particulates.

### Step #2

Measure 1-liter of WVO or VCO in a clear glass or plastic mixing container that has a minimum capacity of 1.5 liters (1.58 quarts).

### Step #3

Preheat the oil to about 48.9 °C (120 °F). This step ensures that the chemical reaction (transesterification) will be complete.

### Step #4

Review the titration procedure thoroughly and make sure that all the necessary supplies for the titration procedure are readily available.

### Step #5

Using a 1-ml disposable pipette, disperse 1-mL (one milliliter) of preheated oil to a paper cup #1.

### Step #6

Measure 10-mL of methanol (using a graduated cylinder) and pour it into paper cup #1 that contains the 1-mL of oil (from step #5).

### Step #7

Using the disposable 1-ml pipette, agitate the mixture by squeezing the pipette repeatedly until the methanol (10-mL) and oil (1-mL) are mixed thoroughly. When they are mixed properly, the mixture should have a milky white color and no globules of oil are visible.

### Step #8

Place about 10-mL of Titration Solution (0.01 % Sodium Hydroxide Solution – made by mixing 1 gram of pure NaOH with 1-L of distilled water) paper cup #2.

### Step #9

Using a pH testing kit, place 3 drops of the pH indicator into the oil/methanol mixture in paper cup #1 and stir with a clean stirring rod. The mixture will usually turn orange, indicating an acidic initial pH.

**Step #10**

Using a 5 mL pipette, withdraw 5 mL of the Titration Solution (0.01% NaOH) from paper cup #2. Slowly add the titration solution to the oil/methane mixture in paper cup #1, until solution changes from orange to a blue-green color. The blue-green color indicates the pH is about 9 (slightly basic).

**Step #11**

Once the mixture has been titrated to a pH of 9, record the quantity of NaOH (mL) dispersed from the 5-mL pipette.

**Step #12**

Now add 3.5 to the quantity pipetted during the titration, e.g., if the sample required 1.6 ml of NaOH solution for titration, one would need to add  $1.6 + 3.5 = 5.1$  grams of NaOH (crystalline form – like table salt) to the 200 ml of methanol.

**Step #13**

Measure the amount of NaOH, as determined in Step 12, and mix with 200 ml of methanol (being careful not to breathe any vapors from the methanol). This mixture creates a methoxide solution necessary for transesterification. Transesterification is the process that breaks the fatty acid chains (biodiesel) from the triglyceride molecule backbone.

**Step #14**

Using a hand blender, slowly mix the 200 ml of methoxide with the 1-liter of oil.

**Step #15**

For proper transesterification, the contents should be stirred for 15 minutes or more.

**Step #16**

After removing the blender/mixer, allow the biodiesel solution to set overnight (8 hours or more).

**Step #17**

After settling, one should see a distinct separation line between the biodiesel and the glycerol (the dark substance at the bottom of the mixing container).

**Step #18**

Pour off (decant) the biodiesel, being careful not to pour any of the glycerol into the receiving container.

**Step #19**

Properly dispose of the glycerol.

## **Washing the Biodiesel**

### **Step #20**

Mix the biodiesel with about 200 ml of wash-water (regular tap water) and mix very gently (if one mixes the water/biodiesel too vigorously, soap may be created versus biodiesel).

### **Step #21**

Allow the water/biodiesel mixture to separate (this may take a few hours). The wash-water will be at the bottom of the container and the biodiesel will be at the top.

### **Step #22**

Using a 5-ml pipette, withdraw 4 ml of wash-water from the bottom of the container.

### **Step #23**

Check the pH of the wash-water according to the instructions in the pH kit (1 drop of pH indicator for 4 ml of solution). The first sample of wash-water will probably have a pH of 10 or 11 (caustic).

### **Step #24**

Decant (pour off) the biodiesel into another container and properly dispose of the wash-water solution.

### **Step #25**

Repeat Steps 20 through 25 until the wash-water has a pH near 7 (neutral).

## **Using the Biodiesel**

Filter the biodiesel through a standard diesel fuel filter before placing it in the fuel tank of any diesel powered device. Biodiesel should be used with 3 months.