Testing Biodiesel

- **Completion of Reaction:** Dissolve 25 ml of biodiesel in 225 ml of methanol.
- The biodiesel should be fully soluble in the methanol forming a clear bright phase.
- If you observe undissolved material at the bottom of the sample the reaction did not proceed to completion
- Each ml of undissolved material corresponds to 4% by volume.
- **Emulsification:** Combine one part biodiesel with one part water (50/50 mix) and shake vigorously.
- For washed and polished fuel: If the resulting mixture separates quickly and the biodiesel phase on top appears clear and bright and the water phase at the bottom appears clear and free of debris your fuel is clean
- For unwashed fuel: If the resulting mixture does not settle out within a few minutes take care during washing not to agitate too vigorously. The fuel still contains excess soaps.

Testing Biodiesel

- **Specific gravity**: The specific gravity of a substance is a comparison of its density to that of water ($1 g/cm^3$). Imagine three gallon bottles: one filled with water, another filled with feathers, and another filled with lead. All bottles have the same volume but different weights. The feathers have a lower specific gravity than water and the lead is higher. (Specific Gravity = Mass/Volume)

- **Clarity:** Ideally, there will be two distinct layers: an amber (ranging from very light to very dark depending on the oil used) biodiesel layer on top and a darker glycerol layer on the bottom (usually contaminated with catalyst, alcohol, or food particulates). Sometimes, there will be a third or fourth layer between the glycerol and the biodiesel. These layers are soap from too much catalyst or water and often appear milky or yellowish.

- **pH**- When an acid is poured into water, it gives up hydrogen (H) to the water. When a base is poured into water, it gives up hydroxide (OH) to the water. We can measure pH using litmus paper or a digital pH meter. Unwashed biodiesel will have a pH around 9 and washed biodiesel will be closer to 7.

- **Cloud Point** - The cloud point is the temperature at which a cloud of wax crystals first appears in a fuel sample that is cooled. The cloud point is determined by visually inspecting for a haze to become visible as the fuel is cooled (in a refrigerator, for example).

Cold Flow Properties

- At low temperatures, biodiesel will gel or crystallize into a solid mass that cannot be filtered or pumped. The engine cannot run at these temperatures. This is not a new problem for diesel engine operators. Petroleum-based diesel fuel also gels but at temperatures that are lower than for biodiesel.
- The cloud point is the temperature at which crystals first start to form in the fuel
- The pour point is the lowest temperature at which the fuel will still pour from a container.
- The cold filter plugging point (CFPP) is the lowest temperature at which at certain volume of fuel can be drawn through a metal screen filter. It usually correlates well with the lowest temperature that an engine will operate.
- When crystallization starts for biodiesel the entire fuel tends to gel at the same time. In contrast, petroleum diesel fuel is a mixture of hundreds of different compounds that solidify at very different temperatures. So, even if some compounds crystallize at a relatively high temperature, many other compounds will stay liquid to a much lower temperature.

How can you test specific gravity?

- A hydrometer is used to measure the specific gravity
- A hydrometer is a graduated glass tube filled with air and fitted with a weight
- To test specific gravity, obtain a hydrometer & a large graduate cylinder
- Fill the graduated cylinder with biodiesel and drop the hydrometer into the biodiesel
- The point at which the hydrometer floats is the measurement of specific gravity
- Hydrometer readings should taken at temperatures between 60 to 85 degrees Fahrenheit
- **Specific gravity of Biodiesel is between 0.860 and 0.900**
- Specific gravity of vegetable oil is usually 0.920 or higher

Visual Inspection of Biodiesel

- Visual inspection indicates successful biodiesel reaction in 2 distinct layers
- Top layer: 80-90% product, lighter color than the bottom layer, it is biodiesel!
- Bottom Layer: 10-20% of the reaction, darker color, a mixture of glycerin, catalyst/lye, alcohol & possibly food particles
- A milky middle layer in the test batch indicates a production of soap (due to water in vegetable oil or too much lye)
- Biodiesel with this middle layer should undergo a water wash
- Unsuccessful reactions result in no layering or one solid batch of soap

Separating Glycerin from Biodiesel

With a stopcock container:

- drain glycerin into a beaker, shut valve after glycerin is completely drained
- A small amount of biodiesel in glycerin is acceptable

With Mason Jar or Blender:

- Carefully pour the biodiesel out of the container so as not to disturb the glycerol layer at the bottom.
- pump, siphon, or ladle biodiesel off the top layer
- glycerin will settle at the bottom of the blender
- do not mix the biodiesel & glycerin layers

Water Wash

- The purpose of a water wash is to remove un-reacted alcohol, catalyst, or glycerin in the biodiesel
- Un-reacted alcohol decreases the flashpoint of biodiesel
- Biodiesel with 0.2% alcohol does not meet ASTM fuel standards
- Water wash reduces alcohol levels below 0.2%
- Water wash will also remove any soap in the biodiesel

Water Wash Procedure

1. After the glycerin settles to the bottom of the container, measure the pH of biodiesel layer. pH of unwashed biodiesel should be close to 9.
2. Drain the glycerin from the container
3. Using a shower head/lawn sprinkler, slowly sprinkle water into the biodiesel container until there is a equal amount of water & biodiesel. Using acetic or tannic acid in a ratio of 10 ml acid to 1 L of water can increase the effectiveness of the wash.
4. There are two schools of thought on agitation. One says agitate the biodiesel & water gently for 5 minutes. The other says avoid agitation at all costs. Agitating the biodiesel seems to significantly decrease the final yield. The solution will appear as a yellowish or whitish, milky mixture.
5. Allow water to settle out the biodiesel. This will take several days.
6. Once the mixture has settled, the biodiesel will be floating on top of the water. There may be a cloudy middle layer. Drain the water off (ground or wastewater drain).
7. Drain the cloudy layer into another container, it will eventually settle into biodiesel and water.
8. Measure the pH of biodiesel, it should be close to 7.
9. Biodiesel can be washed multiple times to purify the fuel.

Glycerin: How to use it

- For every 100 units of biodiesel fuel produced there are about 11 units of glycerine by-product.
- Glycerin is used in animal feeds, hand creams, toothpaste, soaps, and lubricants.
- The glycerin is biodegradable, non-toxic, contains no PCBs
- Can be composted after recovering excess methanol

Cleaning Up Glycerol

- Glycerol can be purified to about 85% by acidulating with acid (like 85% concentrated phosphoric)

- About 1.5 to 1.7 ml of phosphoric acid for each gram of sodium lye used in the process

- The acid combines with the residual catalyst to form salt and water.
- The acid breaks the soaps into FFA.
- FFA can be recovered and used as boiler fuel or esterified
- Remaining glycerol should be about 85% pure with salt, water making up the majority of the balance.

Reclaiming Methanol from Glycerol

- It is possible to reclaim methanol from crude glycerol by heating it above 148 degrees Farenheit (the boiling point of methanol)
- This is most commonly done in a pressure rated vessel outfitted with a condenser
- If you plan on reusing the methanol to make biodiesel using a distillation column to ensure that it contains no water is important
- Extreme caution should be used during this operation since methanol burns easily and is *extremely* dangerous because it burn invisibly.

Glycerol Stock Pile

- From Tom Leue's operation….

  "Well, this is it, so far. We are storing the glycerol, and the containers are piling up. (Doesn't this picture look like a family portrait?) We plan on cleaning the glycerol and burning it for heat energy. Waste not, want not…"

Links

- www.journeytoforever.org
- www.me.iastate.edu/biodiesel
- www.greenfuels.org
- www.yellowbiodiesel.com
- www.the-sage.com
- www.soapcrafters.com