Food or Fuel? (Lesson Plan)

(The Chemistry and Efficiency of Producing Biodiesel)

Suggested Grade Level 9-12

Overview

After taking a virtual tour of Penn State's Combustion Lab with Professor André Boehman, students will be introduced to the idea of renewable, homegrown fuels. Students will investigate the relationship between fuel properties and chemical structure by making their own batch of biodiesel from virgin olive oil. The suggested time frame for this lesson is three to four (3-4) 50-minute class periods.

Standard Statements

- 3.4.10 A Explain concepts about the structure and properties of matter.
- 3.4.12 A Apply concepts about the structure and properties of matter.
- 3.6.10 A Apply biotechnologies that relate to related technologies of propagating, growing, maintaining, adapting, treating and converting.
- 3.6.12 A Analyze biotechnologies that relate to related technologies of propagating, growing, maintaining, adapting, treating and converting.
- 3.8.12 A Analyze the relationship between societal demands and scientific and technological enterprises.
- 4.8.10 B Analyze the relationship between the use of natural resources and sustaining our society.
- 4.8.10 C Analyze how human activities may cause changes in an ecosystem.

Content Objectives

Students will know that

- 1. Biodiesel is a clean, renewable, domestically produced fuel source that could lower United States dependence on imported oil.
- 2. The properties of compounds are determined by their chemical structure.
- 2. Biodiesel is derived from three reactants: glyceride (oil), alcohol and a catalyst.
- 3. The chemical structure of biodiesel is described as a mono alkyl ester of long chain fatty acids derived from natural oils.

Process Objectives

Students will be able to

- 1. Describe the process of transesterification utilizing chemical formulas.
- 2. Identify fuel properties and resulting issues. For example, cold weather usability of biodiesel as a function of its chemical structure.
- 3. Diagram methods of biodiesel production from raw materials for virgin oils.
- 4. Construct and compare models of chemical structure of the substances involved in biodiesel production such as: alcohols, alkenes, alkanes, alkyls, acids, esters, etc.

Assessment Strategies

- 1. Completion of *Food or Fuel?* laboratory handout and related calculations.
- 2. Completion of small group analysis of biodiesel as a viable fuel source for transportation.
- 3. Informal evaluation of participation in group discussion.

4. Performance assessment of modeling and comparison of chemical compounds involved in biodiesel production.

Materials

- Laboratory Set-up and special materials part of Student Handout and Teacher Notes
 - Note: Several chemicals included to be used with caution: lye and methanol. Sodium methoxide is formed in the transesterification of vegetable oil to make biodiesel and should be performed under a hood.
- Ball-and-stick modeling sets or virtual simulation for each pair of students (not required)
- Teacher computer with internet access
- Computers with internet access:1 per student group
- Projection equipment

Multimedia Resources

- PSU Combustion Lab video series: Segments 1-7 [QuickTime movies]
- Virtual Gasoline engine simulation: http://auto.howstuffworks.com/engine1.htm
- Virtual Diesel engine simulation: http://auto.howstuffworks.com/diesel1.htm
- UNH Biodiesel Group PowerPoint presentation (optional)
- "Biodiesel-O-Matic" Excel spreadsheet (optional)

Procedures

Part 1: <u>Take a virtual tour of PSU's Combustion Lab</u>

(30 minutes)

- 1. Share selected segments [1-3] of the PSU Combustion Lab tour to introduce the idea of what engineers researching renewable, homegrown fuels do.
- 2. Allow student pairs or small groups to explore how internal combustion engines work and compare that to how a diesel engine works using *How Stuff Works?* http://auto.howstuffworks.com/engine3.htm & http://auto.howstuffworks.com/diesel1.htm.
- 3. Follow up with a discussion to reveal students thoughts on the differences between the two types of engines. Also, make the connection that rotational motion is produced by the crankshaft.

Part 2: Grease Monkeys: Form and Function in a Fuel

(1-50 min Class Period)

- 1. Revisit the video collection to view segments 4-7 for specific background on fuel properties and Professor Andre Boehman's "State of Biodiesel" comments.
- 2. Introduce students to the process of transesterification and the chemical structures of the components of biodiesel (glyceride, alcohol and catalyst).
- 3. Discuss the role of chemical structure in determining physical properties of a substance. For example, the solvent properties of biodiesel in an engine.
- 4. Allow students to work in small groups to reflect on the video series by asking: How does biodiesel measure up to today's fuels? Challenge students to make models of the components of the biodiesel production reaction using ball-and-stick sets, Styrofoam and pipe cleaners, or a virtual modeling simulation like ChemStudio if you have access to it. Students should submit their modeling efforts and a journal or written record of their discussion of the video as homework if it cannot be completed in class. A helpful website to direct students to for schematics is:
 - http://www.me.iastate.edu/biodiesel/Pages/biodiesel1.html. (Chemistry is discussed on Pages 2 and 3 of this site).

Part 3: Homework and lab prep

(Homework)

- 1. Prompt students to the idea that many individuals are making and using biodiesel to power their vehicles and that energy security relies on a diverse energy portfolio. Explain to students that they will be producing some biodiesel as a lab exercise and that they need to be aware of the process.
- 2. Give each student a lab handout to review and assign the National Biodiesel Board's article, "Biodiesel and Energy Security," for supplementary reading. The article is available online at: http://www.biodiesel.org/pdf files/fuelfactsheets/Energy Security0604.pdf.

Part 4: Lab time! Make some biodiesel...

(2-50 min Class Period)

- 1. **Important:** Run through an oral quizzing activity to review lab safety for the group.
- 2. Allow students to work through the lab handout with a partner to answer the questions and produce biodiesel from virgin olive oil (students are prompted to get your initials after calculating how much NaOH they need to catalyze the reaction for the volume of oil specified).
- 3. Give students instructions on the next steps for further investigation and storage of their biodiesel.
- 4. Debrief lab activity and address any questions generated from the reading assignment.
- 5. Allow students to complete lab handouts and Questions to Consider. Questions to Consider may be assigned for homework if not finished in class.

Extension

- 1. Visit the Energy Efficiency and Renewable Energy website to compare the fuel properties and trade-offs for biodiesel to other alternative fuels: http://www.eere.energy.gov/afdc/altfuel/fuel_properties.html.
- 2. Wash and test your biodiesel products for quality and compare to the American Society for Testing & Materials (ASTM) standards.
- 3. Use the EERE Alternative Fuels locator website: http://afdcmap.nrel.gov/locator/LocatePane.asp to find the closest refueling station and if possible, visit a local vendor and/or find out how to donate your product for use in a diesel engine.
- 4. Titrate waste vegetable oil (WVO) and try a round of processing with fryolator feedstock (see Mike Pelly's biodiesel method for more information, http://journeytoforever.org/biodiesel_mike.html). The titration procedure is included on the next page.

Titration Procedure:

Approximately 3.5g of NaOH per L of oil is needed when using virgin oil. However when using waste oils (which therefore have free fatty acid (FFA) content), the pH must first be determined (by titration) to calculate the additional amount of NaOH needed. The *titration* (for used oils, or older oils that could have degraded some, producing FFAs) is done as follows:

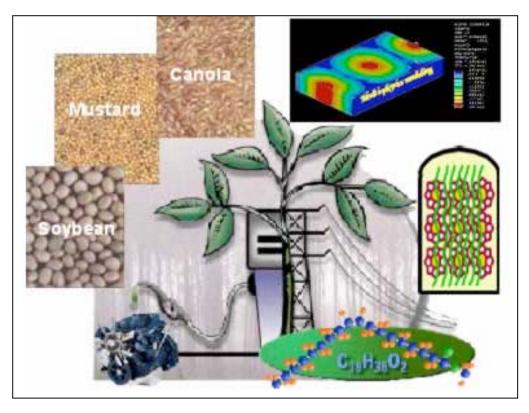
Dissolve one gram of NaOH into 1 liter of distilled water. Dissolve 1ml of the filtered oil into 10ml of isopropyl alcohol and add two drops of phenolphthalein, an acid base indicator. Now slowly add, by calibrated dropper or pipette, the NaOH(aq) solution to the oil solution, mix intermittently. When the oil solution turns pink and stays pink for ten seconds the titration is complete. The volume of NaOH(aq) solution in milliliters necessary to neutralize the free fatty acids corresponds directly with the number of additional grams per liter of NaOH needed for transesterification.

5. Use the "Biodiesel-O-Matic" Excel spreadsheet to compute costs for the biodiesel students have produced.

Food or Fuel? (Student Handout)

(The Chemistry and Efficiency of Producing Biodiesel)

Name:



Source: http://www.cmu.edu/cmnews/extra/050527 biodiesel.html

Our lab research goal is simple: To learn how to make biodiesel from household ingredients and materials.

In order to build a more balanced energy portfolio for our transportation wants and needs, it is important for you to understand the pros and cons of an alternative fuel like biodiesel. Since the Department of Energy's Office of Biomass Program has discontinued its biodiesel research due to other priorities, we are recruiting you! In the enclosed investigation, your team will synthesize a batch of fuel (that could be used in a diesel engine of a VW Jetta TDI) from oil that can be purchased in the local grocery store. The procedure is relatively simple, but there are some important safety issues to keep in mind. If you are prepared to take responsibility for minding the precautions involved and operate in a SAFETY FIRST fashion, sign below and welcome the potential for a piece of a whole new world of energy security and independence from foreign Oil.

Signed:	 	 	
Dated:			

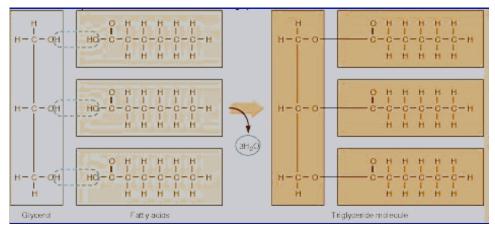
Background (Adapted from the *Biodiesel Handout for the 2005 New Hampshire Science Teacher's Association Workshop*¹ and Kitchen Biodiesel's step-by-step guide² to brewing small batches of biodiesel.)

What is Biodiesel Made From?

Biodiesel consists of three principal feed stocks.

1. Oil:

Glycerides are commonly known as oils or fats, chemically speaking these are long chain fatty acids joined by a glycerin backbone. They appear most often with three fatty acid chains connected to the glycerin (named glycerol in the diagram below), making them *trigylcerides*.



Source: www.unh.edu/p2/biodiesel/media/NHSTA05.ppt

In the US, the primary triglycerides used currently for biodiesel production are soybean oil and waste vegetable oil (which is often used soybean oil). Other vegetable oils like corn oil, canola (and edible version of rapeseed) oil, cottonseed oil, mustard and palm oil, etc³. To get more information on the chemical structure of different oils you can purchase in the grocery store, visit: http://www.me.iastate.edu/biodiesel/Pages/biodiesel2.html.

During the process of being used in fryolators, some of the triglycerides are broken apart into mono or diglycerides, leaving free fatty acids (FFAs) in the oil. To counter this, additional catalyst must be added according to the acidity of the specific oil, since the FFAs will bond with and neutralize some of the alkali catalyst.

2. Alcohol:

Although a variety of alcohols can be used to produce Biodiesel, such as, ethanol or butanol, this experiment will focus on methanol as it is most readily available, and most frequently used. Therefore, the Biodiesel produced is referred to as *methyl esters*. Methanol is one of the most common industrial alcohols; because of its abundant supply it's most often the least expensive alcohol as well. Most methanol comes from fossil fuels (though it can also be made from biomass, such as wood), while most ethanol is plant-based (though it's also made from petroleum).

¹ UNH Biodiesel Group. *Biodiesel Handout for the 2005 New Hampshire Science Teacher's Association Workshop*. http://www.unh.edu/p2/biodiesel, accessed 15July 2005.

² Kitchen Biodiesel. "Step by step guide to making one liter batch of Biodiesel in a two liter soda bottle." http://www.kitchen-biodiesel.com, accessed 22 December 2005.

³ Biodiesel Education. "What is biodiesel?" http://wwww.me.iastate.edu/bidiesel/Pages/biodiesel1.html, accessed 4 January 2006.

METHANOL



POISON! Causes eye and skin irritation. May be absorbed through intact skin. This substance has caused adverse reproductive and fetal effects in animals. **Danger! Flammable liquid and vapor.** Harmful if inhaled. May be **FATAL** or cause **BLINDNESS** if swallowed. May cause central nervous system depression. May cause digestive tract irritation with nausea, vomiting, and diarrhea. MSDS

3. Catalyst:

The third reactant needed is a catalyst that initiates the reaction and allows the esters to detach. The strong base solutions typically used are commonly found in stores as lye or chemically know as sodium hydroxide (NaOH) and potassium hydroxide (KOH). The **lye** catalyst can be either sodium hydroxide (caustic soda, NaOH) or potassium hydroxide (KOH); however, NaOH is often easier to get and it's cheaper to use. With KOH, the process is the same, but you need to use 1.4 times as much (1.4025). KOH can also provide potash fertilizer as a by-product of the biodiesel process.

Once again since this classroom experiment will be using NaOH... CAUTION: Lye (both NaOH and KOH) is dangerous -- don't get it on your skin or in your eyes, don't breathe any fumes, keep the whole process away from food. Lye reacts with aluminum, tin and zinc. Use HDPE (High-Density Polyethylene), glass, enamel or stainless steel containers for methoxide (The result of mixing NaOH and methanol).

Sodium Hydroxide

POISON! DANGER! CORROSIVE. MAY BE **FATAL** IF SWALLOWED. HARMFUL IF INHALED. CAUSES BURNS TO ANY AREA OF CONTACT. REACTS WITH WATER, ACIDS AND OTHER MATERIALS. MSDS

How is Biodiesel Made?

(Here I would put the step by step equations showing the reactants that you just described above, and the mechanism of the reactions. I will help you with this if you need me!) Vegetable oils and animal fats are triglycerides, containing glycerin. The biodiesel process turns the oils into esters, separating out the glycerin. The glycerin sinks to the bottom and the biodiesel floats on top and can be siphoned off.

The process is called **transesterification**, which substitutes alcohol for the glycerin in a chemical reaction, using lye as a catalyst.

You will be working with DANGEROUS/POISONOUS chemicals. Common sense MUST be used. You are responsible for your actions and the safety of yourself and everyone/everything around you!



Recommended Safety Gear:

The minimum is chemical proof gloves, apron, eye protection and dust mask.

Do NOT inhale any vapors.

Always have running water available to wash off any splashes.

Making your Own Biodiesel

Materials for each lab group **All equipment should be clean and dry**

- Safety goggles (for all lab inhabitants!)
- Nitrile gloves-2 pairs
- Long pants and sleeves and lab aprons
- Ventilating masks
- 200 mL of new vegetable oil: We are using ______ oil
- 4 mL of methanol
- Lye catalyst, ____ mg NaOH

!!! Get your teacher's initials & approval

- Scales accurate to 0.01 grams
- 400 mL beaker (for oil)
- glass graduated cylinder or comparable glass measuring device (for methanol)
- 1 canning jar with secure lid and sealing ring
- 2 funnels
- 1 plastic bottle with cap (water or soft-drink bottle clean and dry) for settling

Pre-Lab Preparation:

- 1. Read over the lab handout.
- 2. Calculate the amount of NaOH you must use in your transesterification reaction if 3.5 g are needed to process 1 L of new vegetable oil.
- 3. Read "Biodiesel and Energy Security" (a separate handout) and make note of any questions you may develop about the lab. Take care to look at the procedure carefully...you may have a quiz before beginning the lab activity...

PROCEDURE:

Step 1: **MAKING THE METHOXIDE:**



WARNING: METHOXIDE is a POISON! MSDS

DO NOT BREATH VAPORS. WASH OFF ANY SPLASHES.



!!DO NOT MIX THE METHOXIDE IN A PLASTIC SOFT DRINK BOTTLE AS THE NaOH ATTACKS THE PLASTIC AND YOU WILL QUICKLY BE SHAKING A BOTTLE FULL OF HOLES WITH METHOXIDE GOING EVERYWHERE!!

- 1. In a well ventilated area, measure 4 mL of room temperature methanol using a glass graduated cylinder and pour it into a glass mason jar.
- 2. Measure out the quantity of NaOH (lye) you calculated earlier and add to the methanol in the jar replacing the lid tightly to prevent any leaks.

3. Shake/swirl until all the lye is dissolved. **As you mix the temperature will increase substantially. This is normal and may take 10 minutes or more to dissolve all of the lye—be patient and swirl until ALL lye is dissolved**

Step 2: MAKING THE BIODIESEL With NEW OIL

- 1. Using a funnel, pour the oil into a **DRY** 16 oz soda/water bottle.
- 2. In a well ventilated area, pour the mixture of methanol/NaOH (methoxide) on top of the oil using the same funnel.
- 3. Remove funnel.
- 4. Screw the top down TIGHT onto the bottle.
- 5. Shake vigorously for about ten seconds/ 40 good shakes.
 *NO appreciable pressure is generated during this mixing.
- 6. Now place the bottle on a table and begin work on the *Questions to Consider* section of the lab handout.
- 7. After 10 minutes, take a look at your product and shake vigorously for about ten seconds again.
- 8. After another 10 minutes, catalog (diagram with labels) what has happened in the box below or in your lab journal.

<u>Diagram of biodiesel product after two shakings</u> :					

9. Follow your teacher's instructions for next steps with your biodiesel product and complete the Questions to Consider for homework.

Questions to Consider

1.	Which substance in this reaction is the catalyst?				
2.	What kind of reaction is this? generates heat]	[Hint: it			
3.	What might the "glop" that is settling out be?	_			
4.	Why is this reaction called <i>transesterification</i> ?				
-					
5.	What does biodiesel look like? (at the molecular level, using chemistry notation)				
6.	How might our processing technique be improved to make a better biodiese	l product?			
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-					
7.	How can biodiesel contribute (if you feel it can at all) to our future?				
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_					

Food or Fuel? Student Lab Handout

6

Analyzing Your Product:

Possible errors:

- 1. Soap formation. You can get this if using waste vegetable oil that is heavily used, or using too much catalyst. Any Free Fatty Acids (FFAs) will combine with the alkaline catalyst (NaOH or KOH) to make soap (which is why you need to use extra NaOH when dealing with used oils with FFAs). These soaps need to be removed from the biodiesel by "washing" it (discussed later). A large amount of FFAs can result in enough soap forming to turn the entire reaction into a bunch of "glop" (non-scientific term). If using waste vegetable oil (WVO) with high levels of FFAs (determined via titration), a slightly different process can be used, known as an "acid-base" process (whereas this process discussed is strictly a base catalyzed process). In the acid-base process, you first add some acid (hydrochloric typically) and a small amount of methanol to esterify the FFAs into biodiesel, and then do the normal base catalyzed process to convert the triglycerides into biodiesel. With high FFA oils, the normal base process doesn't yield as much biodiesel, since the FFAs are being turned into soap (and removed through washing). The acid-base process allows you to turn those FFAs into biodiesel.
- 2. Not enough lye, resulting in unreacted oil.
- 3. Not enough alcohol (reaction does not proceed to completion), can also get more soap formation.
- 4. Water in oil (results in catalyst being broken apart, and more soap formation)
- 5. Not enough reaction time (a common problem with demonstration batches like this. Ideally you want to mix for an hour or so, at a slightly elevated temperature (120-130° F). The reaction does not proceed instantly from triglycerides (oils) to 3 biodiesel molecules (per triglyceride). Instead, the methoxide first cleaves off one fatty acid (making one biodiesel molecule from it, by combining with the methanol), leaving a diglyceride (DG). If there is further agitation, the DG is broken apart by the methoxide to make another biodiesel molecule and leaving a monoglyceride (MG). Further agitation breaks that MG apart, making the third biodiesel molecule and leaving free glycerin. If the agitation does not continue long enough (or is not repeated multiple times), you will likely not see full conversion of triglycerides to biodiesel, instead being left with some MGs and DGs, which will often show up as white "chunks" when the fuel cools down to room temperature.

Additional analysis notes	

Food or Fuel? (Teacher Notes)

(The Chemistry and Efficiency of Producing Biodiesel)

Background on Biodiesel Production

(It is strongly suggested that the materials adapted for this background: http://www.unh.edu/p2/biodiesel/media/NHSTA-handout.doc be consulted as a one-stop resource for further detail in gathering lecture material).

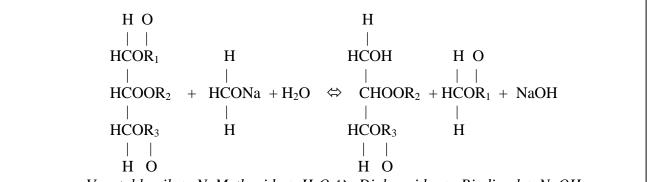
Vegetable oils and animal fats are triglycerides, containing glycerin. The biodiesel process turns the oils into esters, separating out the glycerin. The glycerin sinks to the bottom and the biodiesel floats on top and can be poured off.

The process is called transesterification, is show below. The reaction substitutes alcohol for the glycerin in a chemical reaction, using lye as a catalyst.

$$\begin{array}{|c|c|c|c|}\hline CH_2OOR_1 & catalyst & CH_2OH\\ | & & & & |\\ CHOOR_2 & + & 3CH_3OH & \Leftrightarrow & 3CH_3OOR_x & + & CHOH\\ | & & & & |\\ CH_2OOR_3 & & & & CH_2OH\\ \hline \textit{Vegetable oil} & \textit{3 Methanols} & \textit{Biodiesel} & \textit{Glycerin}\\ \hline \end{array}$$

 R_x is used since the biodiesel produced will consist of different types of mono-alkyl esters, because of the various fatty acids (R_1 , R_2 , R_3) in the vegetable oil. The reaction can proceed both ways, so it is necessary to add an excess of methanol to force the reaction to the right. Since it is not desirable to have free methanol in the biodiesel fuel, it is then necessary to recover the methanol either by water washing, or a pressure-condensing method. But, the glycerin must be removed first (and actually, most of the excess alcohol stays with the glycerin). If you remove the surplus methanol while the glycerin is still present with the biodiesel, the process will start gradually reversing – biodiesel and glycerin combining to re-make vegetable oil and methanol. The glycerin is more dense than the biodiesel, so it will gradually settle to the bottom in the reactor, simplifying separation.

Now, since we first react the catalyst with the methanol to form a methoxide (potassium or sodium methoxide), the reaction doesn't actually proceed exactly as shown above. If we use NaOH as our catalyst, it combines with methanol (CH₃OH) to form sodium methoxide (NaO-CH₃) and a water: NaOH + CH₃OH \rightarrow NaOCH₃ + H₂O. Sodium Methoxide is a quite hazardous material, so it is extremely important to handle it with care – it is explosive and toxic.



Vegetable oil + NaMethoxide + $H_2O \Leftrightarrow Diglyceride + Biodiesel + NaOH$ Step 1 – triglyceride turns to diglyceride, one methoxide joins freed fatty acid to make biodiesel

1. Notes on chemicals needed

The alcohol used can be either methanol, which makes methyl esters, or ethanol (ethyl esters). Most methanol comes from fossil fuels (though it can also be made from biomass, such as wood), while most ethanol is plant-based (though it's also made from petroleum). But the biodiesel process using ethanol is much more difficult than with methanol, strictly not for novices.

Methanol is also called methyl alcohol, wood alcohol, wood naphtha, wood spirits, methyl hydrate (or "stove fuel"), carbinol, colonial spirits, Columbian spirits, Manhattan spirits, methylol, methyl hydroxide, hydroxymethane, monohydroxymethane, pyroxylic spirit, or MeOH (CH3OH or CH4O) -- all the same thing. (But, confusingly, "methylcarbinol" or "methyl carbinol" is used for both methanol and ethanol.)

You can usually get **methanol** from bulk liquid fuels distributors (for large quantities). For small amounts, you can use DriGas fuel line antifreeze, one type is methanol (e.g. "HEET" in the yellow container), another is isopropyl alcohol (isopropanol, rubbing alcohol)→Per the advice on the Kitchen Biodiesel website, make sure to get the methanol type of DriGas because isopropanol does not work for making biodiesel. Methanol is also sold in supermarkets and chain stores as "stove fuel" for barbecues and fondues, but check the contents -- not all "stove fuel" is methanol, it could also be "white gas", basically gasoline. It must be *pure* methanol or it won't work for making biodiesel.

The **lye** catalyst can be either sodium hydroxide (caustic soda, NaOH) or potassium hydroxide (KOH). This lesson chooses to use NaOH because it is often easier to get and it's cheaper to use. With KOH, the process is the same, but you need to use 1.4 times as much (1.4025). Note: Experienced biodieselers making top-quality fuel usually use KOH, and so do the commercial producers. KOH can also provide potash fertilizer as a by-product of the biodiesel process.

You can get both NaOH and KOH from soapmakers' suppliers and from chemicals suppliers. NaOH is used as drain-cleaner and you can get it from hardware stores, but it has to be pure NaOH (not Drano or equivalent, no colored granules).

CAUTION:

Lye (both NaOH and KOH) is dangerous -- don't get it on your skin or in your eyes, don't breathe any fumes, keep the whole process away from food.. Lye reacts with aluminum, tin and zinc. Use HDPE (High-Density Polyethylene), glass, enamel or stainless steel containers for methoxide.

2. Lye

You need to be quick when measuring out the lye because it very rapidly absorbs water from the atmosphere and water interferes with the biodiesel reaction.

How much to use: For a 1L batch of biodiesel, NaOH must be at least 96% pure. Use exactly 3.5 grams. If you're using KOH it depends on the strength. If it's 99% pure (rare) use exactly 4.9 grams (4.90875). If it's 92% pure (more common) use 5.3 grams (5.33). If it's 85% pure (also common) use 5.8 grams (5.775). Any strength of KOH from 85% or stronger will do the job.

3. Mixing the methoxide

Methanol also absorbs water from the atmosphere so do it quickly and replace the lid of the methanol container tightly. Don't be too frightened of methanol, if you're working at ordinary room temperature and you keep it at arm's length, you won't be exposed to dangerous fumes.

Shake the container a few times -- swirl it round rather than shaking it up and down. The mixture gets hot from the reaction. If you swirl it thoroughly for a minute or so five or six times over a period of time the lye will completely dissolve in the methanol, forming sodium methoxide or potassium methoxide. As soon as the liquid is clear with no undissolved particles you can begin the process.

[Tip: The more you swirl the container the faster the lye will dissolve]. Experienced biodieselers comment that with NaOH it can take from overnight to a few hours to as little as half-an-hour with lots of swirling; but the micro-scaled version used in this lab will take 10-15 minutes. (Remind students not to be impatient and that it may take 5 or 6 swirls to get it to dissolve, wait for ALL the lye to dissolve). Mixing KOH is much faster, it dissolves in the methanol more easily than NaOH and can be ready for use in 10 minutes].

Some additional notes on materials and safety:

A few cautions: DON'T mix the methanol and NaOH (lye) in a plastic bottle (the student lab handout is suggesting pint-sized or smaller canning or jelly jars) as NaOH attacks some types of plastic. Once the methoxide is mixed, though, it is quite acceptable to mix your biodiesel in a plastic bottle.

DO NOT allow any WATER into any steps of this procedure.

Again, Do NOT store unused methoxide in plastic bottles. Some plastic will degrade over time when in contact with methoxide.

Methanol boils at about 65°C/148°F. DO NOT mix when the oil is above 60°C/140°F.

The process revisited:

If you would like to use a blender, here is a sample process. (The student lab handout processes by shaking.)



Use a spare blender you don't need or get a cheap secondhand one -- cheap because it might not last very long, but it will get you going until you build something better.

Check that the blender seals are in good order. Make sure all parts of the blender are clean and dry and that the blender components are tightly fitted.

Pre-heat the oil to 55 deg C (130 deg F) and pour it into the blender.

With the blender still switched off, carefully pour the prepared methoxide from the High density polyethylene (HDPE) container into the oil.

Secure the blender lid tightly and switch on. Lower speeds should be enough. Blend for at least 20 minutes.

4. Transfer

As soon as the process is completed, pour the mixture from the blender or the mini-processor into the 2-litre PET bottle for settling and screw on the lid tightly. (As the mixture cools it will contract and you might have to let some more air into the bottle later.)

5. Settling

Allow to settle for 12-24 hours.

Darker-colored glycerin by-product will collect in a distinct layer at the bottom of the bottle, with a clear line of separation from the pale liquid above, which is the biodiesel]. The biodiesel varies somewhat in color according to the oil used (and so does the by-product layer at the bottom) but usually it's pale and yellowish (used-oil biodiesel can be darker and more amber). The biodiesel might be clear or it might still be cloudy, which is not a problem. It will clear eventually but there's no need to wait.

Carefully decant the top layer of biodiesel into a clean jar or PET bottle, taking care not to get any of the glycerin layer mixed up with the biodiesel. If you do, re-settle and try again.

6. Analyzing the Product: (this section relates to Q#4 in the student handout)

After allowing the solution to sit for about ten minutes, the beginning of separation can be observed. However, after about eight hours the glycerin molecules will have mostly settled to the bottom of the container and the methyl esters (biodiesel) will be on top.

Student products should now have a bottle containing lighter colored biodiesel on top of a layer of darker "glop." The biodiesel will be very cloudy, and it will take a day or two more for it to clear.

Typically the "glop" layer is about the same or a bit more than the amount of methanol used. Methanol makes up approximately 10% of the Biodiesel, but to force the reaction, an excess is generally added – usually totaling 20% of the volume of the oil (at a larger scale, well designed processors can use methanol recovery systems to recover this surplus methanol after the reaction, after removal of the glycerin).

For a reaction with one liter of oil; 200 ml of methanol is first mixed with 3.5 grams of NaOH, plus any additional NaOH in regards to the free fatty acid titration (only necessary if using used/waste vegetable oil).

Additional Resources

Websites:

- Biomass Glossary from the U.S. DOE's Energy Efficiency and Renewable Energy Information Portal
 - http://www.eere.energy.gov/biomass/student_glossary.html
- The U.S. DOE's Alternative Fuels Data Center http://www.eere.energy.gov/afdc/altfuel/biodiesel.html
- The National Biodiesel Board http://www.biodiesel.org/
- Howstuffworks segment on "How Biodiesel Works" http://auto.howstuffworks.com/biodiesel.htm
- BECON Biodiesel Education http://www.me.iastate.edu/biodiesel/Pages/biodiesel1.html
- Colorado University's Biodiesel Chemistry presentation http://www.cu-biodiesel.org/webPRES/evan.htm

Interactive Excel Spreadsheet:

Biodiesel-O-Matic.xls

In-depth information on washing fuel and further quality testing:

• Wash test:

This is the most useful all-round test, and it's very simple: Put 150 ml of unwashed biodiesel (settled for 12 hours or more, with the glycerin layer removed) in a half-liter glass jar. Add 150 ml of water, screw the lid on tight and shake it up and down violently for 10 seconds or more. Then let it settle. The biodiesel should separate from the water in half an hour or less, with amber biodiesel on top and milky water below. This is quality fuel, a completed product with minimal contaminants. Wash it, dry it and use it with confidence.

But if it turns into something that looks like mayonnaise and won't separate, or if it only separates very slowly, with a creamy white layer sandwiched between water and biodiesel, it's not quality fuel and your process needs improvement. Either you've used too much catalyst and made soap (better titration), or a poor conversion has left you with half-processed mono- and diglycerides, fuel contaminants that act as emulsifiers (better titration, try more methanol, better agitation, longer processing time, better temperature control), or both too much catalyst and poor conversion.



Wash-test with unwashed biodiesel -- left, after a violent 10-second shaking; right, biodiesel and water separated cleanly within minutes. The biodiesel will be cloudy, and the water can be milkier than this, but as long as it separates quickly and cleanly, it passes the test.

Whichever, you're headed for washing problems. Super-gentle washing techniques might avoid the problems, but you'll still be left with poor-quality fuel laced with contaminants that can cause injector coking and engine damage and they can't be washed out.

If you have an emulsion any thicker than the normal "paper thin" interface layer between oil and water, the batch should be retreated. Retreat as with virgin oil, with the standard 3.5 g of lye per liter of oil but using only 100 ml methanol per liter of oil.

Quality testing, continued.

Aleks Kac, a contributor to the "Journey to Forever" web forum, has provided the following information and some additional quality tests. An extension activity may be coordinated utilizing the following text to spur discussion about fuel quality and subsequent viability.

"Diesel engines require fuel of a certain quality. You just can't pour poor-quality biodiesel into the tank and expect the engine to go on and on without problems. You have three very dangerous enemies: free glycerin, poorly converted oils/fats and sodium lye. Free glycerin and mono-, di- and triglycerides (poor ester conversion) will form gum-like deposits around injector tips and valve heads, sodium lye can damage the injector pump. The key to good fuel is to just do right and finish it! Use pure chemicals (sulfuric acid, sodium lye and methanol) and measure them accurately -- this will take care of poor conversion. A proper wash will get rid of any glycerin and neutralize the remaining lye.

7. Washing

If you would like to extend the lesson to complete a wash and pass through your own version of standards testing, proceed to www.biodieselcommunity.org/washingasmallbatch, for more information.