122nd ASEE Annual Conference & Exposition
June 14 - 17, 2015
Seattle, WA
Making Value for Society
Paper ID #11540

BYOE: Using the Biodiesel Process as a Lab Activity to Reinforce Chemistry Concepts

Mr. Roger A Beardsley PE, Central Washington University

Roger Beardsley is an associate professor of Mechanical Engineering Technology at Central Washington University, Ellensburg WA. He teaches classes in Thermodynamics, Fluids and Heat Transfer, among others. His professional interests include renewable energy, including biofuels.

BYOE: Using The Biodiesel Process As A Lab Activity To Reinforce Chemistry Concepts And Process Design

This BYOE demonstration presents a simple process converting vegetable oil into biodiesel (or Fatty Acid Methyl Ester, FAME). The conversion of vegetable oil into biodiesel is a spontaneous and relatively robust catalyzed chemical process that can be easily adapted into a lab experiment using inexpensive equipment and supplies. The basic chemistry of the biodiesel conversion process is outlined in a worksheet format to allow students to calculate reactant and catalyst amounts. Students then measure out the chemicals and process their own individual batch in a discarded PET soda bottle. An instructor can also perform this lab activity as a simplified lab activity or an in-class demonstration. The pedagogical context can range from a high school or summer science camp presentation to a lab activity in a senior level engineering course. While the biodiesel reaction has been outlined in other sources, this paper describes a remarkably cost effective way to bring the process into the classroom as a lab activity. As a result it supports an educational outcome of incorporating experimental techniques and procedures into the curriculum. Based on student responses, the lab activity succeeds in demonstrating practical application of chemical principles that achieve the conversion of deep fryer oil waste into biodiesel fuel right before student's eyes.

Introduction

Many students are enthusiastic about working with and learning about biofuels, and the experiment that has been outlined can spark student interest and help them better understand the many related scientific and engineering principles that are embodied in the conversion of vegetable oil into biodiesel. The biodiesel process can give context to chemical concepts, such as calculating molar values and converting them to mass and volume measurements. The fluids involved have a wide range of density and viscosity values that can also be explored further and discussed in a lab or lecture setting. The effect of these properties on process design and equipment selection can also be a point for discussion. The student experience with biodiesel processing can lead to discussions about renewable fuels and related climate change topics.

The process outlined in this paper can be presented three ways, depending on the pedagogical context:

1) Process Demonstration
2) Lab Activity with predetermined process parameters
3) Lab Activity with process parameters developed in worksheet

Biodiesel Reaction Parameters

Most vegetable oils and fats (triglyceride) can be easily converted into fatty acid methyl ester (FAME = biodiesel) with a catalyzed process that can occur at room temperature and pressure. The ratios of reactant and products are outlined in Table 1. A reaction using 400 ml of oil will produce nearly 400 ml of biodiesel. Typically excess methanol is used to push the reaction closer to completion(1). The demo process outlined in this paper will use 4.5 gram-mol of methanol(2) (50% excess). The catalyst amount can also vary, though too much catalyst will contribute to soap formation and may cause gelling while too little catalyst will affect the reaction rate. There is latitude for variability in the measurements, making this process robust and relatively foolproof for a classroom setting.

Reactants	1 gram-mol Triglyceride (Vegetable oils, fats) 3 gram-mols Methanol 0.12 gram-mol NaOH
Products	3 gram-mols FAME (Biodiesel) 1 gram-mol Glycerin

Table 1: Biodiesel Reaction Reactants and Products Outlined

Figure 1: Process Reactants and Products

Basic Experimental Process Outline

1) A Single Step Biodiesel Process Demonstration

The most basic process demonstration is performed in a discarded PET plastic soda bottle. This catalyzed reaction will proceed at room temperature and pressure, a fairly robust and spontaneous process. A simple reaction performed in a 650 ml / 20 oz soda bottle can convert 400 ml of vegetable oil (new or used), 74 ml of methanol, and 1.95 grams NaOH beads into about 400 ml of biodiesel with about 55 ml of glycerin byproduct.

The discarded soda bottle should be rinsed clean and completely dried before adding 400 ml of vegetable oil. The methanol and NaOH can be measured out well ahead of time and stored in their separate containers. The separate catalyst solution is prepared by adding the 1.95 g NaOH to 74 ml methanol in a separate container, but don't combine them until just before the demonstration as the sodium methoxide can degrade over time. This process takes time and agitation (ie, swirling the bottle), and should be performed at least 10 minutes before it is needed. The catalyst bottle will heat up due to the exothermic enthalpy of solution for these compounds.

Undissolved NaOH and other insoluble particles will sink to the bottom of the catalyst solution bottle if it is left to stand undisturbed for a minute or two. Any undissolved NaOH will combine directly with the oil to form excess soaps, so it is advised to avoid pouring the undissolved particles in the catalyst solution into the reaction bottle containing the oil.

The prepared catalyst solution is then carefully added to the oil in the soda bottle in front of the demonstration audience and the reaction bottle is resealed. The reaction should be started very soon after adding the catalyst solution by shaking the bottle vigorously to cause the oil and catalyst solution to blend. When the reaction bottle is first shaken it will change from relatively clear amber vegetable oil to a creamy tan emulsion as the catalyst unzips the triglyceride molecules. As the shaking continues the appearance will change from creamy tan to a cloudy amber color, and the speed with which trapped air bubbles rise in the liquid will visibly increase as the viscosity decreases. Once the reaction is underway the bottle can be passed to members of a demonstration audience to shake, thus engaging them in the process.

The reaction will be mostly done after 10 minutes of shaking. The glycerin byproduct will start to form a visible layer on the bottom within the hour. Overnight the glycerin will settle, leaving a transparent amber layer of biodiesel floating on a layer of glycerin. The glycerin layer will range in color from nearly clear (when using new vegetable oil) to a deep dark maroon red (when using waste deep fryer oil). If the feedstock oil is warmed up (approx. 110 - 130 F) before starting the reaction, the reaction and settling process will proceed faster, and the glycerin layer will form more rapidly.

Plastic soda bottle material (PET) may weaken from exposure to the reactants and/or products of the reaction. In most cases this only becomes apparent in the tendency for the bottleneck to fracture where the cap stresses the threads. Applying excess torque to the cap may result in stress fractures causing the bottle to leak from the thread root. For that reason it is wise to pass a small towel with the bottle if the audience participates in passing and shaking the bottle. Also warning the audience of this issue is wise as some audience members may remove the cap to smell the biodiesel, and then crank the top back on with all their strength. The leakage is mostly a risk of oil stains of clothing, and is not a significant safety risk. Because of the material weakening, a bottle should be used for a reaction one time only and then recycled. It is also recommended that a carbonated drink bottle be used. Uncarbonated drinking water bottles often have a significantly thinner wall thickness and less strength.

2) Lab Activity with Predetermined Process Parameters

For this lab experiment, the same process outlined in the demonstration is followed but students do their own reactant measurements and processing. As in the demonstration, the proportions are 400 ml oil, 74 ml methanol, and 1.95 grams NaOH. The lab requires the same equipment and materials as the demonstration, but with enough reactants and containers to supply each individual or group. Depending on the number of reactions being performed in this lab, extra measuring equipment may be needed (100 gram scale, graduated beakers, graduated cylinders) to set up measuring stations that are shared.

This version of the lab activity is appropriate to save time or if students don't have basic chemistry experience. If class time is very limited, the reactants can be premeasured for each group to save time.

3) Lab Activity with Process Parameter Worksheet

In this version of the lab, students are given a worksheet with higher-level process parameters, and they must calculate the measurements for the reaction. Typically the students are assigned an oil volume based on their bottle size (approximately 75% of total volume). Using oil density and molecular weight data, they work out the mass and gram-mols of oil in their given volume. The worksheet uses that data to determine the gram-mols of the other reactants (4.5 gram mols of methanol and 0.12 gram-mol NaOH per gram-mol of oil). The mass of NaOH catalyst can then be calculated. For methanol, the required molar quantity is calculated, converted into mass, and further converted into a volume measurement. Data of reactant properties used in these calculations is presented in Table 2 [3].

Compound	Molecular Weight, grams per gram-mol	Density, kg/Liter @ 20 C
Sodium Hydroxide NaOH	40.09	-
Methanol CH_3OH	32.04	.7914
Vegetable Oil (Triglyceride of Oleic Acid)	885.46	.8988
Biodiesel (Oleic Acid Methyl Ester)	296.5	.8739
Glycerol	92.10	1.2613
Water	18.02	1.000

Table 2: Selected Properties of Biodiesel Process Reactants [3]

Before students proceed to measure out reactants, it is wise to check their calculated measurements for errors to prevent process problems and reactant overconsumption. The introduction, worksheet, measurement and processing can be completed in a two-hour lab period. The glycerin layer will form and settle out within an hour, and be mostly complete overnight.

Beyond this worksheet version of the lab activity, there are many ways to increase the rigor of the pedagogy [9]. Additional topics could include acid esterification processes, determining acid number to optimize catalyst amount (ie, predicting catalyst consumption into formation of soaps), and experimentally determining reaction kinetics are among possible ways to expand the lab activity, depending on available equipment and expertise.

Basic Equipment and Supplies for the Process

Measuring Equipment (can be shared with multiple lab groups):

- 600 ml graduated Polyethylene beaker (to measure oil)
- 100 ml graduated cylinder, glass or polyethylene (to measure methanol)
- 100-gram electronic scale, .01-gram sensitivity (to measure NaOH)

Process Vessels (for each experiment; reusable):

- Plastic soda bottle, approx. 20 oz PET bottle, rinsed out and dried (for reaction)
- 125 ml Nalgene HDPE wide mouth bottle (for methanol & catalyst preparation)
- Standard pill bottle (for measuring NaOH beads)

Reactants (per reaction, approximate):

- 400 ml Vegetable oil (used deep fryer oil, strained and water free)
- 80 ml methanol
- 2.0 grams NaOH beads

Item	Cost	Example Source
600 ml Beaker with graduations	$ 3.11 ea	US Plastics #70049
100 ml Graduated Cylinder	$ 4.08 ea	US Plastics #70049
100 gram x 0.01 g Scale	$24.95 ea	AC100, American Weigh Systems
PET Soda Bottle, 20 oz	$ 0	Recycle bin or convenience store
125ml Nalgene PP Wide mouth bottle	$ 1.90	US Plastics #70042
Pill container	$ 0.10 (?)	Local pharmacy
Reactants		
400 ml vegetable oil	$ 0	Food service waste
80 ml methanol	$ 0.75	Utah Biodiesel Supply
1.95 g NaOH beads	$ 0.10	Utah Biodiesel Supply

Table 3: Itemized Cost of Equipment, Process Vessels, and Reactants

Note that the chemicals required usually come in larger quantities than those required for a single reaction, depending on the source. For example, Utah Biodiesel Supply sells sodium hydroxide and methanol. As of January 2015 their delivered price for 2 lb of NaOH is $18.50, providing enough NaOH for over 400 experiments. Likewise their delivered price for 4 – 950 ml bottles of methanol is $33.50, providing enough methanol for around 45 reactions. These reactants may also be available through a school chemistry department.

Based on the costs listed in Table 3, in 2015 the initial cost of all equipment and supplies to perform a demonstration would be around $100. This would supply enough reactant chemicals for about 45 reactions. For a class sized laboratory activity with multiple lab groups, an additional scale, two additional 100 ml graduated cylinders, two additional 600 ml beakers, and at least five 125 ml wide mouth catalyst bottles should be obtained. The total for this lab set up would be around $150, with enough supplies for about 45 reactions. Of this total, $95 is nonrecurring equipment expense. The cost of consumable supplies is around $0.85 per reaction (based on January 2015 prices), assuming the oil supply is at no cost.

Further Processing

Second Reaction: Typically the initial reaction does not reach the 99.7% completion rate implied by ASTM6751 due to the reaction equilibrium caused by the buildup of glycerin byproduct[1]. While the initial process looks complete, after removing the glycerin byproduct a similar second reaction can be performed and further glycerin will be created. The glycerin formed by the second reaction gives a visual indication that the first reaction was incomplete. For the second reaction another catalyst mixture with about 20% of the initial methanol & NaOH proportions is prepared and added to the raw biodiesel from which the initial glycerin layer has been removed. The bottle is then agitated for 10 – 20 minutes and left to continue reacting and separate overnight. If student batches have been combined into a larger container (ie, 5 gallon bucket), the agitation may be performed with a paint mixing stirrer and a power drill.

Soap Removal: The biodiesel process has similarities to soap making. Any free fatty acids in the oil will be converted directly into soap, depleting the catalyst in the process. Water will facilitate the soap formation process. Water is formed in mixing the catalyst solution (one gram-mol H_20 formed per gram-mol of NaOH dissolved), and some water may be present in the used oil. To remove soaps, water is added to the finished biodiesel to rinse soap out. The water is mixed into the biodiesel with gentle agitation for the first wash, more agitation in the second wash, and more vigorous in the third wash. Due to its polar nature and higher density, water will attract the soap and fall to the bottom within approximately 10 minutes. The soapy water is then drained off and the process repeated until the water comes out relatively clear (about 3 washes using around 1/3 the volume of the biodiesel each time, or more if needed).

Drying: Biodiesel will absorb water (up to 1500 ppm), but the ASTM D6751 specification limits water content to .050% by volume (500 ppm) [4]. Removing the dissolved water can be accomplished at lab scale by heating up the washed and drained biodiesel in a pot or beaker on a hot plate to approximately 185 F and stirring to help drive out the dissolved moisture. This temperature is high enough to vaporize the dissolved moisture as the fluid is stirred (like a steaming cup of hot coffee), but low enough to prevent boiling of any water pooling in the vessel bottom which might form a large steam bubble that could rise to the surface and eject the fluid from the pot (ie, known in chemistry as ‘bumping’). This temperature is also much lower than the biodiesel boiling point of approximately 660F [10], so risk of fuel combustion is very low.

Safety Precautions

Precautions to be considered for the demonstration should include safety glasses or face shield, a towel for spills, and a covered container large enough to hold the volume of the batch in the event of bottle material failure. When much larger batches are being made, proper ventilation may be an additional concern, along with gloves and protective clothing. Vegetable oil and biodiesel can stain clothing. Biodiesel and its process chemicals can soften and degrade many common safety gloves made with natural rubber, nitrile, neoprene, latex, and butyl rubber. Chemical resistant safety gloves made with polyethylene, PVC, or Viton should be unaffected.

Finished biodiesel itself has low levels of toxicity and flammability hazard. It has been proposed for cleaning petroleum spills [5], where it will dissolve spilled petroleum on shorelines. The biodiesel-petroleum solution can be skimmed out of the water and recovered for use as a fuel. Due to the low vapor pressure of biodiesel [10] a lighted match dropped onto an open surface of finished biodiesel will go out, though unwashed biodiesel may have excess methanol with low vapor pressure that could sustain a flame.

While the vegetable oil and finished biodiesel do not pose significant safety hazards, keeping large quantities of methanol on hand does pose a flammability risk. This has been the cause of most serious biodiesel related accidents. Methanol is also toxic in large amounts and can be absorbed through the skin and inhaled vapors. A majority of the excess methanol and the catalyst end up in the glycerin layer.

The lye-based catalyst is corrosive, and when added to methanol the catalyst solution becomes toxic, corrosive, and flammable. This sodium methoxide solution is the most dangerous compound in the process, a combination of two toxic materials, and is reactive and flammable. Exposure to this compound in the demonstration is limited to the act of pouring the catalyst solution into the oil and capping the reaction container. A small funnel can help limit exposure due to spills, if desired.

Reactant Sources

The least expensive source for feedstock oil is a restaurant or cafeteria with a deep fryer. Each deep fryer contains about 4 gallons of oil and is typically changed every week or two. Most restaurants have at least one and more commonly two or more deep fryers, and disposing of the used grease can be an expense for the restaurant. It is best to arrange for the oil direct from the deep fryer as opposed to collecting it from an outside grease dumpster, where it may be contaminated with water from condensation or rain.

For small quantities, commercially pure methanol is available in auto parts stores as a water absorber for fuel tanks. HEET brand fuel moisture absorber in the yellow bottle is 99% commercial grade methanol with 1% “proprietary additive” (HEET in a red bottle is primarily isopropanol). Larger quantities of methanol may be available from motor sports competitors or fuel distributors. School chemistry labs may also have access to methanol. Alcohol sold as paint thinner has also been used, though may be a blend of ethanol and isopropanol and requires appropriate adjustment to the measured quantity though the molar amount is the same.

Isopropanol sold as rubbing alcohol does not work since it is significantly diluted with water.

A commercial grade Sodium Hydroxide powder may be commonly available in grocery stores, sold as Red Devil Lye drain opener (though it has been removed from many store shelves due to its use in making methamphetamines). Some sources for sodium hydroxide powder include soap making craft suppliers, lab chemical suppliers, or biodiesel supply companies[6].

Process Issues

Forming Soaps: If there is a high concentration of NaOH, soap will be formed. Typical cold process soap making involves vegetable oil, NaOH, and water. Any available moisture in the process will help facilitate soap formation, as will free fatty acids broken off of the triglycerides from use in the deep fryer. Undissolved NaOH in the catalyst solution may immediately convert into soap. Every soap molecule formed also depletes the NaOH catalyst. If a low proportion of soap molecules are formed, they can be easily removed in the water wash. If enough soap is formed the biodiesel can gel, and there is no practical way to recover the demonstration batch after it has gelled.

Material Compatibility: The demo process works well using PET, HDPE or Polypropylene containers, though these materials may lose some strength and be affected in minor ways due to contact with reactants or products of the reaction. Polycarbonate has been observed to craze and crack during processing and should not be used. Many common elastomers (Buna-N, natural rubber, etc) are attacked and swell with biodiesel contact, but Teflon and Viton elastomers are compatible. Stainless steel and carbon steel metals are considered to be compatible with biodiesel but copper, brass, bronze, lead, and tin (and galvanized metals) can be attacked and should not be used [7]. Aluminum is compatible with biodiesel (in context of engine components) [7], but it is not compatible with NaOH and should be avoided for processing vessels.

NaOH Water Absorption: If NaOH is left open to the air, it will absorb moisture from the air and clump together. You can easily see this happening by pouring some dry beads into a pill containers and observing the individual beads clumping together as they are exposed to air. Always make sure there is a tight moisture proof seal on your NaOH container. For purposes of the class lab you should open your main container only long enough to remove enough NaOH for the lab into a secondary container. Gloves and safety glasses or face shield are recommended when handling NaOH.

Other Documented Biodiesel Procedures

Other educators have recognized the biodiesel process as an easily accessible process for teaching process parameters and chemical reactions. Most papers do not address the process as lab activity. One paper reviewed did include a lab procedure for making small batches in a lab setting [8]. The procedure outlined there is more complex and more expensive, using hot plates, glass beakers, and blenders with total set up expense of over a thousand dollars. While it is true that the commercial processes use elevated temperatures and mechanical agitation to increase the reaction rate, the added expense and complexity does not enhance student comprehension of the basic process. Experience has demonstrated that poor temperature control of the hotplate can vaporize the methanol and result in gelling of the oil, ruining the reaction.

A second paper utilized the biodiesel process as a lab activity for organic chemistry. In that paper, the procedure is much more in depth and complex, spread over multiple lab sessions[9]. This procedure includes determining acid number for the oil (free fatty acid levels), an acid-catalyzed esterification process, followed by a base-catalyzed esterification process and testing for glycerol levels (reaction completeness). The lab procedure described in that paper provides context for a number of evaluation processes for organic chemistry and is a higher-level organic chemistry approach to the biodiesel process. The added complexity requires more significant student chemistry background, additional specialized lab equipment and supplies. The principles being taught in that paper focus on more complex chemical principles, in contrast to a focus on the more basic process as outlined in this paper.

Student Survey Responses

The lab activity has been used in a senior level MET Energy Systems (thermodynamic systems) class, though it can be adapted for use at many levels from high school science classes through upper level undergraduate classes. The Energy Systems class included topics such as calculating adiabatic flame temperature and higher heating value preceding the biodiesel lab. These topics required balancing combustion chemistry equations that help remind students of the chemistry calculations in this lab. A survey of students in the class has been conducted over the last three years, with a total of 48 responses recorded thus far. A survey with five statements was given to students with a 5 point Likert scale (1 = strong disagreement and 5 = strong agreement with the statement). General comments were also encouraged. Table 4 summarizes the questions and collective responses. About half of the respondents also included comments, a selection of which are included below.

Statement	Response	Std Dev
1. I knew about the biodiesel process before participating in this lab	2.36	1.06
2. This lab activity was **not** relevant to course topics	1.56	0.89
3. The worksheet helped reinforce my ability to manipulate chemical and physical properties to develop a process recipe	4.35	0.62
4. This Lab Activity helped me gain an understanding of the relative sensitivity of the different process design parameters in the biodiesel conversion process	4.42	0.67
5. This biodiesel lab should **not** be continued in future years	1.17	0.42

Table 4: Student Survey Result Data

Some selected student comments:

"It is a great lab. This helps you apply the chemistry of Energy Systems"
"Very fun and neat. You get to use the chemistry equations to actually produce something instead of just calculating it"
"I learned a lot about the process with this lab. I would continue it, perhaps even have the students perform the entire process."

"It would be interesting to complete more of the processing in concurrent labs."
"I wish we could do the washing etc. in a future class period instead of the lab just ending."
"It's a good lab activity to perform and this gives students a better understanding about biodiesel and the process in making it"
" It would have been interesting to see the wrong mixture turning it into soap, to know the tolerance in the chemistry"
"No direct feedback of reaction success or consequences of calc or process error (other than jelling)"
"It was a fun lab that allowed for collaboration"
"Interesting lab, fun process mixing chemicals"
"Good practical lab"
"Best Lab ever!"

Based on student responses, the lab activity has succeeded in incorporating the learning objective of including experimental techniques and procedures into the curriculum. Efforts have been made to involve the students in further processing (second reaction, washing, drying, evaluation). Due to time constraints of the current Energy Systems course, further processing is done outside of lab time. The individual reactions are combined into one batch and the initial glycerin layer is separated outside of class time. The second reaction can be prepared and performed as a demo, though if the batch is large enough (i.e., 2 – 5 gallons) it may require agitation with a paint stirrer attachment on a power drill. Then the second glycerin layer is removed (typically by siphoning from the bottom with a vinyl hose), and water added for the washes. These processes are generally performed outside of class, and the results are displayed in the back of the classroom with progress monitored over the next week or so by students. While there is limited access to analytical tests available to us for testing the product quality in the lab, there is a qualitative biodiesel test that has been used called the pHLip test that can be used to identify common fuel faults (http://www.phliptest.com/what.html, accessed January 2, 2015).

Conclusion

Biodiesel is an important renewable fuel. It can be produced by converting the waste stream from a restaurant into usable fuel for diesel engines. Performing the basic conversion of vegetable oil into biodiesel helps to make the related chemistry calculations more relevant to students. The conversion process outlined in this paper is inexpensive, robust, and suitable for classroom demonstrations and lab activities. The paper has outlined a process that can be used as a lecture demonstration, a lab activity with predetermined reactants, or as a lab activity determining the basic process recipe starting from a chemical equation. Each version of the experiment gives students a different learning experience. The calculations required for the lab activity can open a window into some basic factors involved in a simple process design. Furthermore, the lab activity or demonstration can expand into a discussion of energy sources, renewable fuels, or climate change. Based on student responses, the lab activity succeeds in demonstrating practical application of chemical principles that achieve the conversion of deep fryer oil waste into biodiesel fuel right before student's eyes.

References

(1) Biodiesel Production Technology
Van Gerpen, J., Shanks, B., Pruszko, R., Clements, D., Knothe, G.
NREL/SR Publication No. 510-36244, July 2004.
National Renewable Energy Laboratory, Boulder CO.

(2) Process Optimization Of Biodiesel Production Using Alkaline Catalysts
A. Singh, B. He, J. Thompson, J. Van Gerpen
Applied Engineering in Agriculture Vol. 22(4): 597-600
American Society of Agricultural and Biological Engineers, 2006

(3) AC 2008-570: Biofuels In The Classroom: Using The Biodiesel Process To Demonstrate Chemical And Physical Principles
Beardsley, R; ASEE Annual Conference Proceedings, 2008
American Society for Engineering Education, Washington, D.C.

(4) Biodiesel Handling and Use Guide, Fourth Edition
NREL Technical Publication TP-540-43672, December 2009
National Renewable Energy Laboratory, Golden, CO

(5) Cytoculture Inc Website http://www.cytoculture.com/process.html, accessed

(6) Utah Biodiesel Supply website, accessed January 10, 2015
https://utahbiodieselsupply.com/biodieselchemicals.php

(7) Compatibility of Metals and Elastomers in Biodiesel: A Review
Bhardwaj, M; Gupta, P; Kumar, N
International Journal of Research (IJR) Vol-1, Issue-7, August 2014

(8) AC 2008-701: Energizing An Introductory Chemical Engineering Course With Biodiesel
Taconi, K; Banish, R.M. ASEE Annual Conference Proceedings, 2008
American Society for Engineering Education

(9) Biodiesel Synthesis and Evaluation: An Organic Chemistry Experiment
Bucholtz, E.C.; Journal of Chemical Education • Vol. 84 No. 2 February 2007
American Chemical Society

(10) Vapor Pressure And Normal Boiling Point Predictions For Pure Methyl Esters And Biodiesel Fuels Yuan, W; Hansen, A.C.; Zhang, Q;
Fuel 84 (2005) 943–950

Appendix: Biodiesel Lab Activity Worksheet

Biodiesel Processing Lab

Objectives: To determine process parameters for a chemical conversion process: converting waste deep fryer oil (triglyceride (TG) source) into fatty acid methyl ester (FAME = biodiesel) using a homogeneous alkaline catalyst based process.

Tasks:

1. Listen to process outline presentation:

   Methanol amount: 4.5 mol MeOH per mol TG
   Catalyst amount: 0.12 mol per mol TG

2. Determine approximate batch size: (1) ________________ **ml Oil**

   (Note: Use PET soda bottle; oil fill should not exceed 75% of bottle capacity)

3. Determine approximate mols of TG (ie, vegetable oil) in oil volume assigned

   Triglyceride (Oleic acid TG) = 885.46 g/mol (2) ________________ mol Oil
   Specific Gravity of Oil: .8988

4. Determine required catalyst mol & mass (3) ______________ mol NaOH

   NaOH = _____________ g/mol
   (4) ____________ **grams NaOH**

5. Determine mols & volume of methanol required (5) _____________ mol CH_3OH

   CH_3OH = ______________ g/mol
   S.G of Methanol @ room temp = .7914 (6) ______________ **ml CH_3OH**

6. Based on (2), predict mass and volume of Glycerin (7)______________ mol

   Glycerin = 92.10 g/mol
   Specific gravity = 1.2613 Glycerin = (8)______________ ml

8. Measure reactants: (Note: Have amounts checked before proceeding)

   a. Measure oil volume and add to oil bottle. Label bottle with your name or initials
   b. Measure methanol volume and add to catalyst solution container
      Note: **DO NOT add methanol directly to oil** in oil bottle
   c. Measure catalyst mass and add to pill container

9. Create catalyst solution:

   Add NaOH catalyst to methanol and dissolve catalyst completely (5-10 min)
   Note: Notice the heat given off in forming the catalyst solution

10. Perform Reaction: Add catalyst solution to oil in bottle.

    Agitate by shaking to mix reactants for 5- 10 min.
    Let raw Biodiesel settle overnight; observe glycerin layer formed on bottom

Lab Worksheet: Due at end of lab period

Appendix: Biodiesel Lab Activity Worksheet Instructor Notes

Biodiesel Process Lab – Instructor Notes
Sample Calculations for Biodiesel Process Worksheet
For 400 ml oil batch size in 20 oz soda container
(1) 400 **ml oil** (batch size)
(2) 400 ml oil (.8988 g oil/ml oil) (1 mol oil/ 885.45 g oil) = .406 mol oil
(3) .406 mol oil (0.12 mol NaOH / 1 mol oil) = 0.0487 mol NaOH
(4) .0487 mol NaOH (40 g/mol NaOH) = **1.95 g NaOH**
(5) .406 mol oil (4.5 mol Methanol / 1 mol oil) = 1.83 mol Methanol
(6) 1.83 mol Methanol (32 g methanol / 1 mol) (1 ml / .7914 g) = **74 ml Methanol**
(7) (1 mol glycerin / 1 mol oil) (.406 mol oil)(92 g/mol)(1 ml / 1.2613 g) = 29.6 ml
Note: most excess methanol goes to glycerin layer; since 1/3 of methanol is excess, the bottom byproduct layer could be 29.6 ml + 24.7 ml = 54.4 ml

For 1.00 Liter oil batch size (useful reference for scaling different batch sizes)

(1) 1000 **ml oil** (batch size)
(2) 1000 ml oil (.8988 g oil/ml oil) (1 mol oil/ 885.45 g oil) = 1.015 mol oil
(3) .406 mol oil (0.12 mol NaOH / 1 mol oil) = 0.122 mol NaOH
(4) .0487 mol NaOH (40 g/mol NaOH) = **4.87 g NaOH**
(5) .406 mol oil (4.5 mol Methanol / 1 mol oil) = 4.56 mol Methanol
(6) 4.56 mol Methanol (32 g methanol / 1 mol) (1 ml / .7914 g) = **185 ml Methanol**

Compound	Molecular Weight, grams per gram-mol	Density, kg/Liter @ 20 C
Sodium Hydroxide NaOH	40.09	-
Methanol CH_3OH	32.04	.7914
Vegetable Oil (Triglyceride of Oleic Acid)	885.46	.8988
Biodiesel (Oleic Acid Methyl Ester)	296.5	.8739
Glycerol	92.10	1.2613
Water	18.02	0.998

Reference Table of Reactant Properties (from various sources)