

# What's "Green" About Biodiesel?

## Research Experiences for Teachers Participants, 2006

Jo G. McCormick, [jomccor@kckps.org](mailto:jomccor@kckps.org)

Washington High School

Kansas City, KS

Jeremy J. Way, [jway@usd232.org](mailto:jway@usd232.org)

Mill Valley High School

Shawnee, KS

## Graduate Student Mentors

April French and Danielle Barker Department of Chemistry

Carrie Hohl, Department of Environmental Engineering

## Faculty Mentors

Dr. Joseph A. Heppert, Department of Chemistry, [jheppert@ku.edu](mailto:jheppert@ku.edu)

Dr. Susan Williams, Department of Chemical and Petroleum Engineering, [smwilliams@ku.edu](mailto:smwilliams@ku.edu)

## Education Program Coordinator

Dr. Claudia Bode, Center for Environmentally Beneficial Catalysis, [bode@ku.edu](mailto:bode@ku.edu)

785-864-1647, 1501 Wakarusa Dr., Lawrence, KS 66045

This module was developed during the summer of 2006 in the Research Experience for Teachers Program. This program was sponsored by the Center for Environmentally Beneficial Catalysis (<http://www.cebc.ku.edu/>) at the University of Kansas (<http://www.ku.edu/>) with funding from the National Science Foundation (<http://www.nsf.gov/>).



## Table of Contents

<b>Major Topics</b>	<b>Page Number</b>
What's Green About Biodiesel? – Teacher's Guide	3-19
Introduction	3-4
Timeline	5
Biodiesel background information	6-7
Resources	7
Skills and topics	8-9
Science standards	9-10
Variables for investigation	10-11
Biodiesel preparation method	11-14
Testing procedures	14-18
References	19
Appendix 1: Student Prompt	20-23
Appendix 2: National NEED Project materials	24

# What's "Green" About Biodiesel? – Teacher's Guide

## Introduction

The purpose of this module is to provide teachers with a resource for instruction on the topics of "green" chemistry and engineering using an inquiry involving the preparation and testing of biodiesel fuels. The authors placed special emphasis on developing preparation techniques and testing protocols that utilizes readily available and relatively inexpensive supplies. Furthermore, the protocols for testing the products were developed based on the assumption that most high school teachers have limited resources and limited access to technical equipment.

The high school science curriculum has started to focus more on human impact on the environment. Students are now taught about the green house effect, acid rain, and pollution. However, textbooks typically do not cover the topic of green chemistry. Green chemistry can be applied in all branches of chemistry. It is more a way of thinking, rather than an individual field of science. The 12 tenets of green chemistry as described by Anastas and Warner (1998) are to:

1. Prevent waste, rather than have to treat it
2. Maximize atom economy, using as much of each reactant in the product as possible
3. Use less hazardous chemical syntheses
4. Use safer chemicals
5. Use safer solvents
6. Increase energy efficiency, using as little energy as possible in the synthesis
7. Use renewable feedstocks
8. Reduce derivatives, unwanted products from side reactions
9. Use catalysts
10. Design products which will degrade naturally
11. Analyze for pollution prevention
12. Minimize the potential for chemical accidents

*(From: Anastas, P. T.; Warner, J. C. Green Chemistry: Theory and Practice, Oxford University Press: New York, 1998, p.3)*

While there is a large quantity of information on the internet and in the literature about green chemistry, there is little on the implementation of it as a topic of high school classroom study. This unit was developed to help secondary teachers introduce green chemistry to their classes using biodiesel as an example of how green chemistry can be applied in both the laboratory and daily life. While the emphasis is on introducing green chemistry, this is also a true inquiry-based project for first-year chemistry students.

Although the primary focus of this activity is to introduce the principles of green chemistry to high school students, teachers may find it beneficial to emphasize the importance of "green" engineering during this activity as well. Green engineering is defined by the US Environmental Protection Agency as "the design, commercialization, and use of processes and products, which are feasible and economical while minimizing 1) generation of pollution at the source and 2) risks to human health and the environment." Thus, the principles of green engineering will be important for the design of environmentally benign biodiesel production processes. The twelve principles of green engineering are as follows:

1. Make all material and energy inputs and outputs as inherently non-hazardous as possible.
2. Prevent waste, rather than treat or clean up waste after it is formed.
3. Design separation and purification operations to minimize energy consumption and materials use.
4. Maximize mass, energy, space, and time efficiency when designing products, processes, and systems.
5. Products, processes, and systems should be "output pulled" rather than "input pushed" through the use of energy and materials.
6. View embedded entropy and complexity as an investment when making design choices on recycle, reuse, or beneficial disposition.
7. Targeted durability, not immortality, should be a design goal.
8. Design for unnecessary capacity or capability (e.g., "one size fits all") solutions should be considered a design flaw.
9. Minimize material diversity in multi-component products to promote disassembly and value retention.
10. Design of products, processes and systems must include integration and interconnectivity with available energy and materials flows.
11. Design products, processes, and systems for performance in a commercial "afterlife".
12. Material and energy inputs should be renewable rather than depleting.

From: [Anastas, P. and Zimmerman, J. "Design Through the 12 Principles of Green Engineering," \*Environmental Science and Technology\*. March 1, 2003, ACS Publishing.](#)

Described in this document are background information on green chemistry and biodiesel, a method for preparation of biodiesel in the classroom, suggested methods for testing the product, and clear indicators of the chemistry skills learned in this activity.

This module could be used (1) at the start of the year to teach scientific method, introductory topics, and basic lab skills found in the traditional first chapters of most chemistry texts; (2) as a cumulative inquiry associated with organic chemistry; or (3) as a capstone laboratory. We suggest the following outline for implementing this module in a first year, high school chemistry course.

## Proposed Timeline for Module

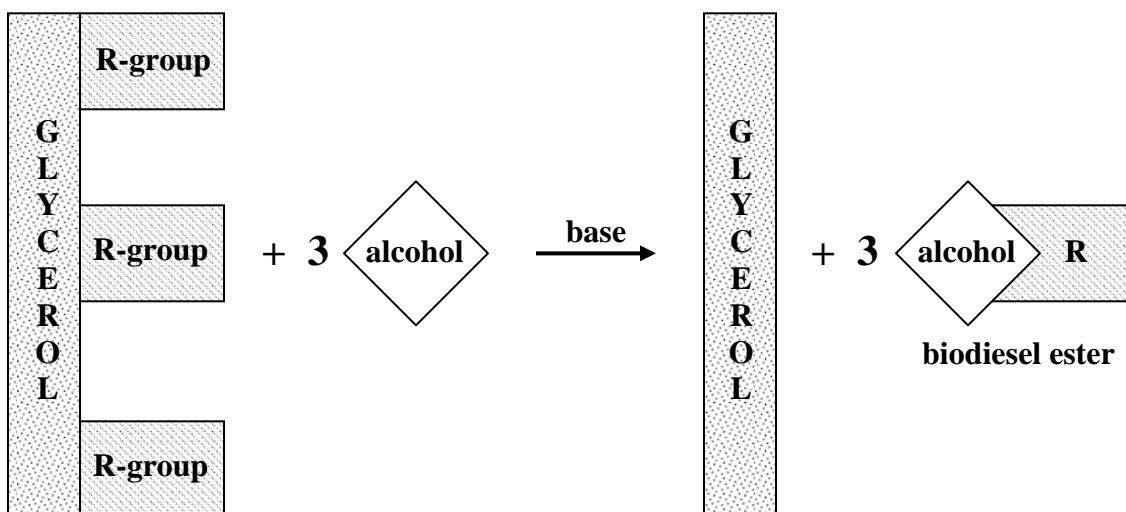
- Day 1      What is green chemistry?
- 12 tenets
  - Green Chemistry: Stopping Pollution Before it Starts (see Resources)
  - Introduce the topic of biodiesel
- Day 2      Researching biodiesel
- Biodiesel—A Domestic, Renewable Fuel (excerpt from The Need Project’s “Transportation Fuels” handout; see Appendix)
  - Begin researching biodiesel
- Day 3      Researching biodiesel
- Student selection of variable(s) to be tested
- Day 4      Prepare biodiesel
- Discuss MSDS
  - Review/Introduce lab procedures
  - Prepare biodiesel sample(s)
- Day 5      Separate biodiesel/Wash if desired (see note below)
- Day 6      Discuss and design lab techniques and testing protocols
- pH
  - density
  - cloud point
  - rate of flow
  - heat of combustion
  - measurements
  - reproducibility/multiple trials
  - controlled variables
- Day 7      Testing biodiesel products
- Day 8      Testing biodiesel products
- Day 9      Work on laboratory report
- address why biodiesel is a green chemistry application
  - discuss results of tests
- Day 10     Laboratory reports due

## What is Biodiesel?

Most diesels currently available are distilled from petroleum. The benefits of diesel over gasoline are that diesel engines traditionally get better mileage, are less prone to breakdown and until recently, were cheaper to run. However, diesel engines produce large amounts of small particulate matter that contaminate the atmosphere. A recent trend has been to prepare a fuel to replace petroleum-based diesel from vegetable oils. This fuel is commonly called biodiesel and can be used either straight or in varying mixtures with petroleum diesel. The advantages to biodiesel are that the source of the oil is renewable, it burns more cleanly, and is less hazardous to the environment. Although engines running on biodiesel produce less particulate matter, the engines do produce more nitrogen oxides. It is probably not possible to supply sufficient biodiesel to replace diesel in all uses because of the large amount of land necessary to produce enough oil. Research to find more land-efficient ways to produce plant-based oils includes farming oil-producing algae.

Biodiesel is produced from a variety of plant oils. The oils are reacted with an alcohol in the presence of a base catalyst. The oils contain triglycerides, which have a glycerin backbone and three attached alkyl groups. The reaction to produce biodiesel breaks the bond between the glycerin (also called glycerol) and the alkyl groups, which react with an alcohol to produce an ester. Glycerol is a byproduct of the reaction.

There are many different sources of vegetable oil that are used to manufacture biodiesel and many of them can be purchased at your local grocery. Variation in the procedure of manufacture, the oil used, the alcohol used or the catalyst used all could affect the final biodiesel product. The reaction to produce biodiesel is shown below.



Most biodiesel is produced from either soy or canola oil, although any plant-based oil can be used. Typical large-scale production uses a strong base, such as potassium or sodium hydroxide,

and methanol for the alcohol. Other alcohols may be used, but the reaction is much more difficult due to the size of the alcohols.

Because this reaction occurs as a step-wise replacement of the R-groups, incomplete reactions may occur, leaving some mono- and diglycerides in the final mixture. These can be removed during the washing step, however, it is not necessary to do so to perform the tests included in this module.

## **Suggested Resources for Green Chemistry and Biodiesel**

The internet is an excellent place to begin researching green chemistry and biodiesel. Many of the sites below were found using Google with such search phrases as 'green chemistry' and 'preparation of biodiesel'. Among the more useful sites found were:

### Green Chemistry Links

<http://www.acs.org/portal/a/c/s/1/resources?id=c373e9f7186084f98f6a4fd8fe800100> – Green chemistry article from the American Chemical Society's ChemMatters, April 2003.

<http://www.cebc.ku.edu/about/green.html> – Lists the tenets of green chemistry and green engineering.

<http://www.chemistry.org/portal/a/c/s/1/resources?id=fa4f7d66a3e711d6e8254fd8fe800100> – A PowerPoint presentation from the American Chemical Society suitable for introducing green chemistry in the classroom.

### Biodiesel Links

[www.journeytoforever.org](http://www.journeytoforever.org) – At first glance, this site appeared to be written by amateurs, however, it is a complete reference guide for the preparation of biodiesels.

[http://www.greeningschools.org/resources/view\\_cat\\_teacher.cfm?id=42](http://www.greeningschools.org/resources/view_cat_teacher.cfm?id=42) – Green chemistry and biodiesel resources assembled by the state of Illinois.

[http://www.biodiesel.org/pdf\\_files/Biodiesel\\_Curriculum\\_Schools.pdf](http://www.biodiesel.org/pdf_files/Biodiesel_Curriculum_Schools.pdf) – Biodiesel activities for use in class. *Transportation Fuels – Biodiesel* by the NEED Project ([www.NEED.org](http://www.NEED.org)) 2004-2005. "Secondary Level: Biodiesel – A Domestic, Renewable Fuel," pp12-13. (Included in this module's appendix).

<http://www.epa.gov/otaq/consumer/fuels/altfuels/420f00032.pdf> – An easy-to-read EPA document on biodiesel.

<http://www.unh.edu/p2/biodiesel/media/NHSTA-handout.doc> – Provides background information for teachers wanting to use biodiesel in lessons in the high school classroom, including preparation and lesson ideas.

<http://www.unh.edu/p2/biodiesel/media/NHSTA05.ppt> – PowerPoint presentation that accompanies the previous link.

## **Skills and Topics Addressed in Module**

If this module is used in its entirety, students will be applying the following skills and techniques:

### Laboratory design and analysis skills

- recognize green chemistry applications in experiments and processes
- determine controlled variables in an experiment
- developing procedures for determining density, cloud point, heat of combustion and rate of flow from background information
- learn to read and interpret MSDS
- analyze experiments for errors

### Laboratory skills

- use an electronic balance
- use a graduated cylinder and volumetric or graduated pipette for volume measurements
- write a research question and identify the variables involved
- separate a mixture according to density and immiscibility
- use a drying agent
- find the pH of a liquid
- prepare an ice bath
- make comparisons to standards
- use a thermometer
- use a ruler, protractor and timer
- follow a written procedure
- properly dispose of waste

### Data manipulation skills

- use significant figures in calculations
- determine mass efficiency of a reaction
- graphing data
- use formulas to find experimental values

### Depending on the focus chosen, the following chemistry topics can be integrated

- measurement
- significant figures
- units
- density
- scientific method
- mixtures
- pH
- reaction types (double displacement)
- balancing equations
- catalysis
- physical and chemical properties



- physical and chemical changes
- chemical activity as related to catalysis
- phase changes
- likes dissolve likes
- solubility/miscibility
- limiting and excess reactants
- freeze point depression
- endothermic and exothermic reactions
- heat capacity
- calorimetry
- heat of combustion
- viscosity
- renewable and non-renewable resources
- waste
- greenhouse gases/global warming
- qualitative vs. quantitative data
- hydrocarbons
- functional groups (alcohol, ester, carboxylic acid)

## Science Standards

### Kansas State Science Standards addressed in this module:

Standard 1: Science as Inquiry

Standard 2A: Chemistry. (2A.2.1, 2A.2.3, 2A.3.1, 2A.3.2)

Standard 2B: Physics. (2B.2.2)

Standard 5: Science and Technology. (5.1.1, 5.1.2, 5.1.3)

Standard 6: Science in Personal and Environmental Perspectives. (6.3.1, 6.3.2, 6.4.2, 6.5.1)

Standard 7: History and Nature of Science. (7.1.2)

### National Science Education Standards for grades 9-12 addressed in this module:

Content Standard A: Science as Inquiry

Content Standard B: Physical Science

1. Chemical Reactions.

e. Catalysts accelerate chemical reactions.

Content Standard F: Science in Personal and Social Perspectives.

1. Natural Resources

b. Human populations use resources in the environment in order to maintain and improve their existence. Natural resources have been and will continue to be used to maintain human populations.

c. The earth does not have infinite resources; increasing human consumption places severe stress on the natural processes that renew some resources, and it depletes those resources that cannot be renewed.

d. Humans use many natural systems as resources. Natural systems have the capacity to reuse waste, but that capacity is limited. Natural systems can change

to an extent that exceeds the limits of organisms to adapt naturally or humans to adapt technologically.

2. Environmental Quality
  - c. Many factors influence environmental quality. Factors that students might investigate include population growth, resource use, population distribution, overconsumption, the capacity of technology to solve problems, poverty, the role of economic, political, and religious views, and different ways humans view the earth.
3. Natural and Human-Induced Hazards
  - b. Human activities can enhance potential for hazards. Acquisition of resources, urban growth, and waste disposal can accelerate rates of natural change.
  - d. Natural and human-induced hazards present the need for humans to assess potential danger and risk. Many changes in the environment designed by humans bring benefits to society, as well as cause risks. Students should understand the costs and trade-offs of various hazards--ranging from those with minor risk to a few people to major catastrophes with major risk to many people. The scale of events and the accuracy with which scientists and engineers can (and cannot) predict events are important considerations.
4. Science and Technology in Local, Global and National Challenges
  - b. Understanding basic concepts and principles of science and technology should precede active debate about the economics, policies, politics, and ethics of various science- and technology-related challenges. However, understanding science alone will not resolve local, national, or global challenges.

## **Variables for Investigation**

- Plant-derived oil used
  - Soybean oil
  - Corn oil
  - Canola (rapeseed) oil
  - Peanut oil
  - Olive oil
  - Used fryer oil
  - Others
- Esterification alcohol used
  - Methyl alcohol
  - Ethyl alcohol (reaction is difficult)
- Base catalyst used
  - Sodium hydroxide
  - Potassium hydroxide
- Preparation conditions
  - Agitation
  - Temperature
  - Time of reaction
- Post-production treatment of biodiesel

- Washing/not washing
- Use of drying agent
- Comparisons
  - Commercial biodiesel
  - Petroleum diesel
  - Feedstock oils

## **Method for Biodiesel Preparation**

### Materials

vegetable oil

9 M base solution (NaOH or KOH)

methanol or ethanol

lab balance

2 250 mL jars with lids (canning jars work well)

1 25 mL graduated cylinder

1 100 mL graduated cylinder

5 Beral pipettes

distilled water

test tubes

Additional materials will be required depending on the tests completed on the biodiesel.

### Procedure to make laboratory samples of biodiesel

The following procedure can be used to prepare lab samples of biodiesel. These samples should not be used as a fuel in any engine due to impurities caused by incomplete reaction of the starting oil and the alcohol. While the samples can be washed according to the procedure given below, in most cases, it is not necessary to wash the samples to obtain reasonable results from the tests. If washing is to be used as a variable, an extra lab period may be required as there are settling times required after each step.

1. Add 20 mL methanol to a container which can be sealed.
2. Pipette 1 mL of either 9 M KOH or 9 M NaOH into the methanol.
3. Seal the container and mix for 2 minutes.
4. Add 100 mL (approximately 80 g) of vegetable oil to the container. Record the mass added.
5. Seal the container and shake vigorously for 10 minutes.
6. Allow the biodiesel to settle overnight. The top layer is the ester (biodiesel) and the bottom layer is glycerol (byproduct).
7. Separate the layers by pouring off the top layer, leaving the bottom layer behind in the container. It will not be possible to completely separate the layers in this manner. To completely separate the layers, pour the remaining glycerol into a test tube and use a pipette to separate the last of the biodiesel.
8. The glycerol layer should be neutralized and can be disposed of down the drain.



shaken samples before settling



sample after settling overnight

### To remove cloudiness from the biodiesel sample without washing

1. Put the biodiesel sample in a container which can be sealed.
2. Add a 'scoop' of anhydrous magnesium sulfate to the sample, seal, and shake.
3. Filter the sample to remove the magnesium sulfate.
4. This procedure removes emulsified water and base catalyst, but does not remove soap or incompletely-reacted triglycerides.



samples drying with  $\text{MgSO}_4$

### To spray wash biodiesel samples to remove most impurities

1. Using distilled water in an ordinary spray bottle, gently spray the surface of the biodiesel sample using approximately 10 mL of water. Agitation of the sample at this point may form a non-breakable emulsion.
2. Allow the sample to separate into the washed biodiesel layer (top) and water layer (bottom).
3. Carefully pour off the biodiesel into another container and repeat steps 1 and 2 two more times.
4. After the third wash, add a 'scoop' of anhydrous magnesium sulfate to the sample, seal, and shake.

5. Filter the sample to remove the magnesium sulfate.



spray washing technique



sample settling after spray washing

It is possible to prepare larger, more completely-reacted biodiesel samples in the classroom lab. A suggested method can be found at [www.journeytoforever.org](http://www.journeytoforever.org). Methods are also available for making biodiesel from used vegetable oil.

## Disposal Procedures

Dispose of excess alcohol by allowing it to evaporate in the hood.

Dispose of excess hydroxides by neutralizing with an acid and flushing it down the drain with excess water.

Prepared biodiesels in these quantities can be disposed of by neutralizing and then mixing them with cat litter until completely absorbed (no marks on a paper towel). The glycerin layer should also be neutralized, but is then safe to be poured down the drain with plenty of water.

## Safety Precautions

Goggles and an apron should be worn during this experiment.

The Material Safety Data Sheets (MSDS) for all chemicals in this experiment are included in the Appendix of this module.

Alcohols are flammable and poisonous. Methanol can be absorbed through the skin or inhalation and could cause blindness or death.

The methoxide formed when the base and methanol are shaken is poisonous and flammable and should only be prepared if it will be used soon after preparation.

Hydroxides are corrosive. Gloves and goggles are required for handling.

Oils, biodiesels, alcohols, and petroleum fuels are all combustible. The procedures for testing for heat of combustion and particulate matter produced during combustion involve burning liquid fuels. Store all fuels in approved containers. Have fire extinguishers available. Demonstrate how to smother a small fire before starting the lab.

## **Testing Procedures for Biodiesel Samples**

### pH

Determine using traditional methods of pH paper, universal indicator, or a pH probe.

### Density

Determine using traditional methods with a graduated cylinder and lab balance.

### Cloud Point

Cloud point represents the temperature at which a liquid becomes turbid or cloudy. Because biodiesels are a mixture of a variety of esters, no clearly defined freezing point can be determined. The cloud point is an indication of the temperature when solid fractions appear in the solution. This is important because fuels must remain liquid to pass through fuel filters and to function in a diesel engine even at cold winter temperatures.

#### Procedure:

1. Prepare an ice bath (using salt in the bath will allow lower temperatures to be tested)
2. Add approximately 15 mL biodiesel each to 2 large test tubes
3. Place a thermometer in 1 of the above test tubes and place tube in the ice bath.
4. Remove the test tube from the bath every few seconds to check the solution for cloudiness using the second test tube as a reference sample. Since the temperatures may be cold enough to cause condensation on the surface of the test tube, check for cloudiness immediately after removing the tube from the ice bath. Continue this process until the biodiesel becomes cloudy. Record the temperature of the biodiesel.
5. Warm the sample back to room temperature and repeat step 4 a minimum of 2 times.



cloud point sample in ice bath



cloud point reached as compared to room temperature reference

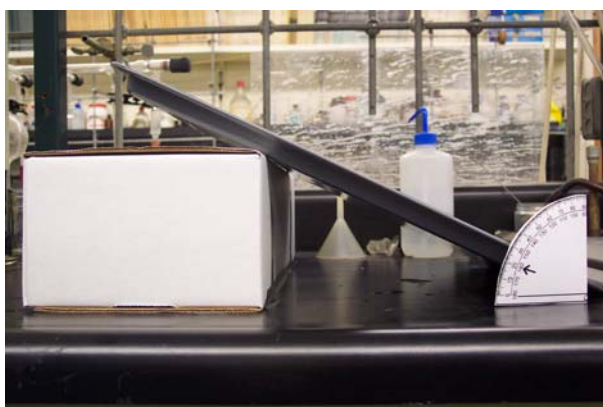
## Viscosity

The viscosity of a liquid is a measure of its resistance to flow. A liquid with a high viscosity, such as molasses, pours very slowly, while a liquid with low viscosity, such as water, pours easily. Fuels must have a low enough viscosity to be used in an engine. Viscosity typically decreases as temperature of a liquid increases. There are instruments to measure the viscosity of fluids and the force applied to the fluid, but these instruments are not commonly found in a high school laboratory. Previous methods for determining viscosity suggested for high school use have included timing the rate of descent of a marble through a column of fluid or measuring the flow of the liquid through a funnel. Both of these methods require fairly large samples and long clean-up times. The following method uses small sample sizes, allows multiple samples to be run simultaneously and is easy to clean-up. While no absolute viscosities can be calculated, comparative viscosities can be determined by determining the rate of flow of a sample of biodiesel on an incline as viscosity is inversely related to the rate of flow.

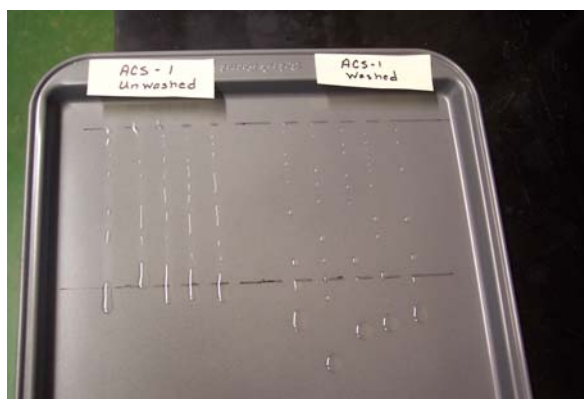
### Procedure:

1. Prepare a Teflon-coated baking sheet by marking a 'start' line using a permanent marker.
2. Set up a lower 'stop' for the bottom edge of the baking sheet by securing a ruler, dowel, or similar object to a lab bench so the lower edge of the baking sheet ends in the same location for each run.
3. Set up something to hold the upper end of the baking sheet at the same height and location for each run. If the object used is large enough, such as a box, the baking sheet can be loaded on it and then the lower edge dropped quickly to the 'stop' on the lab bench so that the same angle is reproduced with each run.

4. On the level baking sheet, place 5 drops of a sample of biodiesel on the start line to form one larger drop. Multiple runs of the same sample may be completed by making additional starting drops on the start line. It is suggested to use 5 runs of the same sample to find the average distance traveled.
5. After the baking sheet is loaded with samples, tip the free (lower) edge of the baking sheet down to the 'stop' and let the samples flow for 10 seconds. At 10 seconds, return the baking sheet to a horizontal position and determine the distance traveled for each sample on the sheet.
6. Wipe the samples off of the baking sheet and then wash twice with warm soapy water, rinse until it is 'squeaky clean' and dry the sheet when finished. If the sheet has unusual reflection and streaks, rewash until the surface appears uniform.



set up for testing rate of flow



results of rate of flow test

### Heat of Combustion

The heat of combustion is a measure of the amount of energy released when a substance is burned. When trying to get heat out of a substance, the more heat released, the better. Since the amount of heat will depend on the size of the sample, the heat of combustion is usually calculated per gram or per mole of the substance so that easy comparisons can be made between substances.

Heat of combustion is calculated by knowing the mass of a sample absorbing the heat ( $m$ ), its specific heat ( $C_p$ ), and the change in its temperature during the combustion ( $\Delta T$ ). The heat of combustion is then divided by the mass of the biodiesel sample burned. The following equation is used to calculate the heat of combustion:

$$\Delta H_c = \frac{m_{\text{water}} \cdot C_p \cdot \Delta T_{\text{water}}}{\text{mass burned}}$$

where  $C_p$  is the specific heat of water, 4.184 J/g·°C.



The following method will not produce absolute heats of combustion, but will provide relative data for the samples tested.

Procedure:

1. Put 50.0 mL water in an aluminum pan (such as for pot-pies). Put this pan in an iron ring supported on a ring stand at a height such that the bottom of this pan is about 1 cm above the sample described below. Record the initial temperature of the water in °C.
2. Fill a small metal, glass or ceramic dish with a sample of biodiesel about 2/3 of the way full. Metal condiment cups work well for this.
3. Cut a piece of aluminum foil large enough to cover the opening of the dish. When covering it, make the center of the foil just touch the top of the biodiesel sample.
4. Prepare a wick for the 'candle' by putting a staple through a wick so that it doesn't fall through the aluminum foil in to the biodiesel sample.
5. GENTLY push the wick through the foil so the lower part of it is in the biodiesels and the upper part looks like a candle-wick above the aluminum foil covering the dish.
6. Determine the initial mass of the biodiesel candle.
7. Light the biodiesel 'candle' wick and put it under the aluminum pan containing the water and burn for 5-10 minutes.
8. At the end of the burn time, extinguish the candle and determine the final temperature of the water and the final mass of the candle.
9. Observations should also be made about the smoke and soot produced by the biodiesel sample.
10. The heated water can be disposed of down the drain. The aluminum pan can be cleaned and reused. The candle biodiesel should be returned to your sample and the wick and foil thrown out in the trash. Wash the dish after completing the test.



wick with staple



biodiesel candle



heat of combustion set up

### **Expected Trends in Results**

- pH is significantly lower in washed and dried samples, regardless of oil or base used
- density is consistent regardless of variable changed
- cloud point varies with feedstock oil, based used, and with washing and drying
- cloud point for biodiesels is higher than that for petroleum diesel
- rate of flow (a measure of relative viscosities) varies with feedstock oil, based used, and with washing and drying
- rate of flow is significantly less than commercial biodiesel and petroleum diesel
- heat of combustion appears similar regardless of variable changed within the precision of the method used for testing

### **Acknowledgments**

The authors would like to thank Dr. Claudia Bode, Dr. Joseph A. Heppert, Dr. Susan Williams, Carrie Hohl, April French, and Danielle Barker for their invaluable input in the development of this module.

## References

What is Green Engineering? <http://www.cebc.ku.edu/about/green.html> (accessed July 2006).

LaMerrill, M.; Kirchhoff, M. *ChemMatters* April, 2003, p. 7, 9.

Transportation Fuels: Biodiesel. [http://www.biodiesel.org/pdf\\_files/Biodiesel\\_Curriculum\\_Schools.pdf](http://www.biodiesel.org/pdf_files/Biodiesel_Curriculum_Schools.pdf) (accessed July 2006).

Journey to Forever. <http://www.journeytoforever.org> (accessed July 2006).

Greening Schools. [http://www.greeningschools.org/resources/view\\_cat\\_teacher.cfm?id=42](http://www.greeningschools.org/resources/view_cat_teacher.cfm?id=42) (accessed July 2006).

Clean Alternative Fuels: Biodiesel. <http://www.epa.gov/otaq/consumer/fuels/altfuels/420f00032.pdf> (accessed July 2006).

Biodiesel Handout for 2005 New Hampshire Science Teacher's Association Workshop. <http://www.unh.edu/p2/biodiesel/media/NHSTA-handout.doc> (accessed July 2006).

Biodiesel. <http://www.biodiesel.org> (accessed July 2006).

*Introduction to Green Chemistry*; Ryan, M. A.; Tinnesand, M. J., Eds.; American Chemical Society: Washington, DC, 2002; pp. 13-22.

## Appendix

1. Student prompt for the biodiesel inquiry project (pp 20 – 23).
2. “SECONDARY: Biodiesel - A Domestic, Renewable Fuel” excerpt from the NEED Project ([http://www.biodiesel.org/pdf\\_files/Biodiesel\\_Curriculum\\_Schools.pdf](http://www.biodiesel.org/pdf_files/Biodiesel_Curriculum_Schools.pdf)).  
The materials from the National Energy Education Development (NEED) Project are reprinted with permission. Funding for the NEED Project came from U.S. Department of Agriculture and National Biodiesel Board.

## Student Science Assessment Prompt

### **Introduction** – *(Include this statement on all prompts)*

Below you will find some information that will help you get started with a research project. Read the information carefully and think of a related research question that you would like to investigate. Your job will be to design and conduct an experimental research project to answer this question and write a report of your findings. There are six parts to your report.

1. Research Question / Hypothesis – A statement of what question you will investigate and your “educated prediction” about what you will find out through an experiment.
2. Literature Review – A description of what information you have found out about the research question through reading books, magazines, personal interviews or web-sites.
3. Experimental Procedure – A step by step, repeatable description of how you will conduct your experiment to answer your research questions.
4. Results / Data – Tables that contain the data you collect.
5. Analysis – Graphs or statistical calculations you have created to make your data more meaningful.
6. Conclusions – A description of what you found out by doing your experiment. Include whether your hypothesis was found to be correct or incorrect, and what new directions you would investigate next to improve your experiment or answer new questions that arise.

The six parts of your research paper and how your responses to them will be evaluated are described in more detail in the rubric that your teacher will share with you.

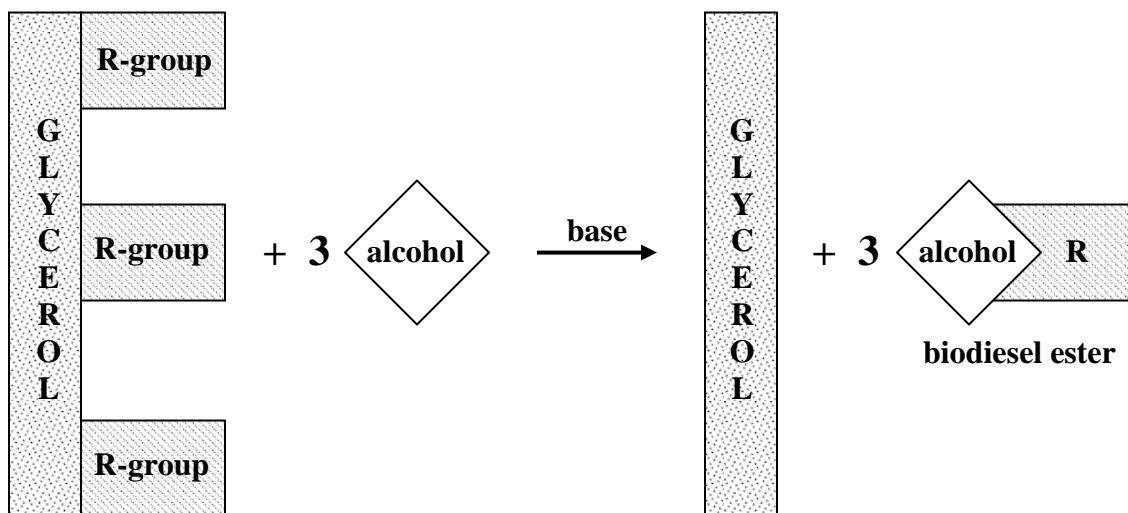
### **Title** – Biofuel Synthesis

**Basic Information for Literature Review** – Will the car of the future run on vegetables? With limited reserves of petroleum and ever increasing prices for gasoline and diesel, the search is on for a replacement fuel. Research is occurring at universities and in basements to find the best method of converting vegetable oils into biodiesel fuel. The reaction can be completed with simple lab equipment and supplies available at the local grocery or even your local McDonalds.

This is your chance to become a biofuels chemist. Changing a vegetable oil into a biodiesel fuel that can be used in a diesel engine just takes a few simple steps. Vegetable oils are mostly composed of chemicals called fatty acids. These fatty acids can be reacted with an alcohol using a base as a catalyst (a chemical added to speed up a reaction) to form an ester (the fuel) and glycerol (the byproduct). It sounds complicated but the reaction is easy. The American Chemical Society has published a method to make laboratory samples of biodiesel using soy oil. A modified version of this method is described in the procedure section.

There are other reasons that using biodiesel might be a good idea for our environment. Research has shown that biodiesel is a cleaner burning fuel than diesel. Information about how biodiesel is manufactured and used can be found at [www.biodiesel.org](http://www.biodiesel.org). There are many other resources on the internet. Use a search engine and key words such as biodiesel, particulate matter from diesel and biodiesel, or manufacture of biodiesel.

There are many different sources of vegetable oil that can be used to manufacture biodiesel and many of them can be purchased at your local grocery. Variation in the procedure of manufacture, the oil used, the alcohol used or the catalyst used all could affect the final biodiesel product. Another concern in any chemical procedure is how efficient the chemical process is. In other words, how much mass of biodiesel is there compared to the mass of glycerin made in the reaction. The reaction to produce biodiesel is shown below.



#### Standards, Benchmarks and Indicators –

Standard 1: Science as Inquiry

Standard 2A: Chemistry. (2A.2.1, 2A.2.3, 2A.3.1, 2A.3.2)

Standard 2B: Physics. (2B.2.2)

Standard 5: Science and Technology. (5.1.1, 5.1.2, 5.1.3)

Standard 6: Science in Personal and Environmental Perspectives. (6.3.1, 6.3.2, 6.4.2, 6.5.1)

Standard 7: History and Nature of Science. (7.1.2)

#### Material Requirements –

vegetable oil

9 M base solution (NaOH or KOH)

methanol or ethanol

lab balance

2 250 mL jars with lids (canning jars work well)

1 25 mL graduated cylinder

1 100 mL graduated cylinder

5 Beral pipettes

distilled water

white gas, kerosene or diesel

test tubes

Additional materials will be required depending on the tests completed on the biodiesel.

### **Starting Points for Experimental Procedure –**

Procedure to make laboratory samples of biodiesel:

1. Add 20 mL methanol to a container which can be sealed.
2. Pipette 1 mL of either 9 M KOH or 9 M NaOH into the methanol.
3. Seal the container and mix for 2 minutes.
4. Add 100 mL (approximately 80 g) of vegetable oil to the container. Record the mass added.
5. Seal the container and shake vigorously for 10 minutes.
6. Allow the biodiesel to settle overnight. The top layer is the ester (biodiesel) and the bottom layer is glycerol (byproduct).
7. Separate the layers by pouring off the top layer, leaving the bottom layer behind in the container. It will not be possible to completely separate the layers in this manner. To completely separate the layers, pour the remaining glycerol into a test tube and use a pipette to separate the last of the biodiesel.

If different biodiesels have been prepared from the chosen oils, properties of the biodiesel can be determined and compared to the properties of white gas, kerosene or diesel. Important properties of biodiesel include density, pH, cloud point, viscosity, heat of combustion, and production of particulate matter when burned. Any one of these properties could be compared for this inquiry. Also of interest is the production efficiency, the ratio of grams of biodiesel produced to the grams of oil used.

Other possibilities for investigation might include changing the alcohol used in the reaction or the base used for the catalyst. How do those changes affect the properties of the biodiesel produced in the reaction or the production efficiency? Be warned that using another alcohol, such as ethanol, makes the reaction much more difficult. You should search the internet for a procedure designed to work for other alcohols.

Cloud point can be determined with an ice water bath, a thermometer and a small test tube filled with the substance tested. The cloud point is the temperature at which the substance becomes cloudy. A cloud point is an indication of how the fuel might perform in an engine in cold weather. It can be hard to see when the solution becomes cloudy. A room temperature sample in a test tube can be used as a reference point.

Viscosity is a measure of how easily a fluid flows under a set condition. One way to get comparative viscosities of the samples is to determine the rate of flow of a small sample (approximately 5 drops) of biodiesel down an inclined plane. A Teflon-coated cookie sheet works well. Think carefully about the variables that need to be controlled in this test.

Heat of combustion for fuels can be determined by burning a small sample of biodiesel and using the heat of the reaction to heat 50 g of water contained in an aluminum tray. This method gives relative values which can be used for comparison purposes only. Use a candle wick and a piece of aluminum foil to make a biodiesel candle. This controls the combustion reaction so the biodiesel burns safely. (See picture below.) The temperature change of the water is recorded. The formula to calculate the heat of combustion per gram of fuel determined in this manner is

$$H = \frac{(\text{mass of water}) \cdot C_p \cdot (\Delta T \text{ of water in } ^\circ\text{C})}{\text{mass of biodiesel consumed}}$$

The  $C_p$  for water is 4.184 J/g  $\cdot$ °C

The amount of soot (particulate matter) produced during combustion can also be studied. Biodiesel is reported to be cleaner burning than petroleum diesel.

**Cautions / Laboratory Safety.**

Goggles and an apron should be worn during this experiment.

Alcohols are flammable and poisonous. Methanol can be absorbed through the skin or inhalation and could cause blindness or death. Dispose of excess alcohol by allowing it to evaporate in the hood.

The methoxide formed when the base and methanol are shaken is poisonous and flammable and should only be prepared if it will be used soon after preparation.

Hydroxides are corrosive. Dispose of excess by neutralizing with an acid and flushing it down the drain with excess water.

Oils, biodiesels, alcohols, and petroleum fuels are all combustible. The procedures for testing for heat of combustion and particulate matter produced during combustion involve burning liquid fuels. Store all fuels in approved containers. Have fire extinguishers available. Demonstrate how to smother a small fire.

Prepared biodiesels in these quantities can be disposed of by neutralizing and then mixing them with cat litter until completely absorbed (no marks on a paper towel). The glycerin layer should also be neutralized, but is then safe to be poured down the drain with plenty of water.

## Appendix 2

“SECONDARY: Biodiesel - A Domestic, Renewable Fuel” excerpt from the NEED Project ([http://www.biodiesel.org/pdf\\_files/Biodiesel\\_Curriculum\\_Schools.pdf](http://www.biodiesel.org/pdf_files/Biodiesel_Curriculum_Schools.pdf)).

Funding for the NEED Project came from U.S. Department of Agriculture and National Biodiesel Board.

Use the secondary level materials described in this pdf file, including:

1. “SECONDARY: Biodiesel - A Domestic, Renewable Fuel”, pp 12-13.
2. “SECONDARY: Biodiesel Math – Graphing Emissions,” p 16.
3. “Secondary crossword,” p 19.