**Synthesis and Analysis of Biodiesel**

**HIGH SCHOOL**

**Green Chemistry & Sustainable Science**

**Teacher Background:**

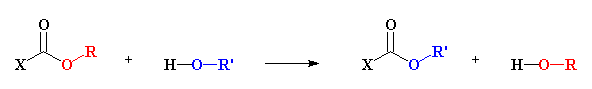
Fossil fuels (natural gas, coal, oil) are non-sustainable and non-renewable resources that our current society has become heavily dependent on. Petroleum (literally rock oil) is the source of gasoline and diesel gas, two common fuels that keep our automobiles running. Prices of these petroleum based fuel are rising due to their high demand and scarcity. In addition, the burning of these fuels increase society’s ecological footprint and add to the green house effect, which underlies global warming. This lesson demonstrates to students the sustainability of a renewable resource, biodiesel.

Biodiesel is a fuel made from vegetable oil through a reaction called *transesterification*. Transesterification is a reaction between an ester and an alcohol in which the -O-R group of the ester and the -O-R' group of the alcohol trade places.

Alcohol

Ester

transesterification



**Figure 1**: A general transesterification reaction.

Source: <http://pslc.ws/macrog/kidsmac/glossary.htm>

The main component of vegetable oil is a molecule called triglyceride (also known as triacylglycerol). Triglyceride is composed of a glycerol group linked by an ester bond to 3 fatty acid molecules. Figures 2, 3 and 4 show the chemical structures of triglyceride, glycerol and fatty acids.

**Figure 2:**

Triglyceride structure

**Figure 3:**

Glycerol structure



R, R’ and R” are long alkyl (hydrocarbon) chains ranging from 12-20 carbons in length

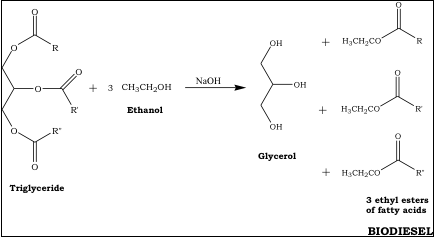
**Figure 4:**

Fatty acid structures. Fatty acids contain a polar carboxylic acid group (-COOH) and a nonpolar alkyl group (-R, -R’, -R”). The conversion of a polar carboxylic acid to a nonpolar methyl or ethyl ester converts the properties the of fatty acids to be similar to fossil fuel diesel.



The ester group in triglyceride will exchange places with the alcohol group in methanol or ethanol\* to form biodiesel. Biodiesel is a methyl or ethyl ester (depending on whether methanol or ethanol is used) of a long chain fatty acid hydrolyzed from triglyceride.

**Figure 5:** The transesterification reaction in the synthesis of biodiesel



The chemical composition of diesel is about 75% saturated hydrocarbons (primarily paraffins including n, iso, and cycloparaffins), and 25% aromatic hydrocarbons (including naphthalenes and alkylbenzenes). The average chemical formula for common diesel fuel is C12H23, ranging from approx. C10H20 to C15H28. The heat of combustion of diesel = 10,700 cal/g.

**Goal:**

The goal of this unit is for the students to understand that our current practices of burning gasoline and diesel gas for automotive transportation is not sustainable and has a big ecological footprint. Students will learn about a renewable resource that may be able to replace gasoline and diesel fuel one day. By synthesizing biodiesel, students will learn how it is made and where it comes from. By analyzing biodiesel, students will be able to determine how likely biodiesel is to eventually replace gasoline and diesel gas as our primary transportation fuels.

**Objectives:** Students will …

* Determine the properties of various cooking oils
* Synthesize biodiesel
* Determine the properties of their biodiesel
* Analyze biodiesel’s heat of combustion
* Assess whether biodiesel can be used in transportation vehicles or not

**Materials:**

* See the materials section for each lesson plan

**Time Required:**

* Lesson 1: Properties of various cooking oils

(1 x 45-60 minute class period)

* Lesson 2: Synthesis of biodiesel (1 x 90 minute class period)
* Lesson 3: Analysis of biodiesel (1 x 90 minute class period)

**MA State Science Standards Met (MA Department of Education Science and Technology/Engineering Frameworks):**

***Properties of Matter***

Broad Concept: Physical and chemical properties can be used to classify and describe matter.

**1.1** Identify and explain some of the physical properties that are used to classify matter, e.g., density, melting point, and boiling point.\*

1.2 Explain the difference between mixtures and pure substances.\*

***Chemical Bonding***

Central Concept: Atoms bond with each other by transferring or sharing valence electrons to form compounds.

* 1. Identify how hydrogen bonding in water affects a variety of physical, chemical, and biological phenomena (e.g., surface tension, capillary action, density, boiling point).

***5. Chemical Reactions and Stoichiometry***

Central Concepts: In a chemical reaction, one or more reactants are transformed into one or more new products. Chemical equations represent the reaction and must be balanced. The conservation of atoms in a chemical reaction leads to the ability to calculate the amount of products formed and reactants used (stoichiometry).

**5.1** Balance chemical equations by applying the laws of conservation of mass and constant composition (definite proportions).

**5.2** Classify chemical reactions as synthesis (combination), decomposition, single displacement (replacement), double displacement, and combustion.

**5.3** Use the mole concept to determine number of particles and molar mass for elements and compounds.

**5.5** Calculate the mass-to-mass stoichiometry for a chemical reaction.

**5.6** Calculate percent yield in a chemical reaction.

***6. States of Matter, Kinetic Molecular Theory, and Thermochemistry***

Central Concepts: Gas particles move independently of each other and are far apart. The behavior of gas particles can be modeled by the kinetic molecular theory. In liquids and solids, unlike gases, particles are close to each other. The driving forces of chemical reactions are energy and entropy. The reorganization of atoms in chemical reactions results in the release or absorption of heat energy.

**6.4** Describe the law of conservation of energy. Explain the difference between an endothermic process and an exothermic process.

**Green chemistry principles met:**

1. Use of renewable feedstocks
2. Use of safer chemicals
3. Real-time monitoring
4. Inherently safer chemistry for accident prevention
5. Catalysis

**Procedure:**

PREP *(if needed)*

* 32 copies of the student sheets for lessons 1-3
* Obtain materials needed prior to each lesson

IN CLASS

* Supervise the students working

**Assessment:**

* Compare the students’ biodiesel’s heat of combustion versus literature heat of combustion values of diesel

**References:**

* <http://www.doe.mass.edu/frameworks/current.html>
* Fisher Science Education; Beyond Benign. Think Green. *http://www.fishersci.com/ wps/ portal/ CMSTATIC?href=/ScienceEducation/ scienceEduStandard/ Features/ Think\_Green/ se\_std\_ThinkGreen\_040708\_1435.jsp &store=ScienceEducation &segment=scienceEduStandard* (accessed August 2008).
* [www.fisheredu.com](http://www.fisheredu.com)
* <http://greenchem.uoregon.edu/gems.html>
* Thompson, J. E. *Biodiesel Synthesis*, Science Division, Lane Community College, 2006
* <http://en.wikipedia.org/wiki/Diesel>
* <http://en.wikipedia.org/wiki/Heat_of_combustion>
* Smith, J. G., Organic Chemistry, 2nd ed. McGraw-Hill Science/Engineering/Math; February 23, 2007.
* Freeman, W.H., Chemistry in the Community: (ChemCom),; 5th edition, American Chemical Society (ACS), January 31, 2006.

**Lesson 1: Properties of Various Cooking Oils**

**Student Sheet**

This lesson contains 2 parts:

* Part 1: Density of various cooking oils
* Part 2: Freezing point and melting point of various cooking oils

**Background:**

Which vegetable oils are the best for use as biodiesel? What property do you think a vegetable oil should have in order to operate properly in a diesel engine? To think about this, we will first take a closer look at vegetable oils that we find in the kitchen. Although these oils are similar to those used in biodiesel, they cannot be used directly as fuel. These oils need to be chemically converted to biodiesel fuel.

**Objective:**

* Determine the density of various cooking oils
* Determine the freezing point and melting point of various cooking oils

**Part 1: Density of various oils**

**Materials:**

* Scale or triple beam balance
* 50 mL graduated cylinder
* 4 x various cooking oils (i.e. vegetable, canola, corn, peanut oils)

**Procedure:**

1. You will work in pairs.
2. Wear safety goggles, gloves and apron.
3. Weigh an empty 50 mL graduated cylinder. Record the mass in grams (g).
4. Pour 20 mL of vegetable oil into the 50 mL graduated cylinder. Record the exact volume of the vegetable oil in milliliters (mL).
5. Weigh the graduated cylinder and vegetable oil on a scale. Record the mass in grams (g).
6. Repeat steps 3-5 for all of the various oils. Use clean glassware for each oil.
7. Calculate the density of each oil by filling in the data sheet on the next pages.

**Data for part 1 (density of various cooking oils):**

***Vegetable oil***

1. Mass of an empty 50 mL graduated cylinder (in g) \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

2. Mass of a 50 mL graduated cylinder + 20 mL vegetable oil (in g) \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

3. Mass of 20 mL of vegetable oil (in g) *line 2 – line 1* \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

4. Volume of 20 mL vegetable oil as determined from the graduated cylinder

(in mL)

\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

5. Density (d) of the vegetable oil (g/mL) *line 3 ÷ line 4*

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(show calculations including units)

***Canola oil***

1. Mass of an empty 50 mL graduated cylinder (in g) \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

2. Mass of a 50 mL graduated cylinder + 20 mL canola oil (in g) \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

3. Mass of 20 mL of canola oil (in g) *line 2 – line 1* \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

4. Volume of 20 mL canola oil as determined from the graduated cylinder

(in mL)

\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

5. Density (d) of the canola oil (g/mL) *line 3 ÷ line 4*

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(show calculations including units)

***Corn oil***

1. Mass of an empty 50 mL graduated cylinder (in g) \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

2. Mass of a 50 mL graduated cylinder + 20 mL corn oil (in g) \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

3. Mass of 20 mL of corn oil (in g) *line 2 – line 1* \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

4. Volume of 20 mL corn oil as determined from the graduated cylinder

(in mL)

\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

5. Density (d) of the corn oil (g/mL) *line 3 ÷ line 4*

\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

(show calculations including units)

***Peanut oil***

1. Mass of an empty 50 mL graduated cylinder (in g) \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

2. Mass of a 50 mL graduated cylinder + 20 mL peanut oil (in g) \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

3. Mass of 20 mL of peanut oil (in g) *line 2 – line 1* \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

4. Volume of 20 mL peanut oil as determined from the graduated cylinder

(in mL)

\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

5. Density (d) of the peanut oil (g/mL) *line 3 ÷ line 4* \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

(show calculations including units)

**Part 2: Freezing point and melting point of various cooking oils**

**Materials:**

* 4 x various types of oils (i.e. vegetable, canola, corn, peanut oils)
* 4 x graduated plastic transfer pipettes
* 2 x thermometers (wide-range)
* 1 x 500 mL beaker
* 1 x glass stirring rod
* 4 x test tubes (12 x 75 mm test tubes are a good size)
* 1 test tube rack
* Rock salt
* Ice
* Dry ice (optional)
* Water

**Procedure:**

1. Label a test tube with the name of the first oil.
2. Using the graduated plastic transfer pipette, transfer 2 mL of this oil into the correspondingly labeled test tube.
3. Place a thermometer into the test tube.
4. Set the test tube in the test tube rack.
5. Add rock salt, ice and water to a 50 mL beaker. Stir the water with a glass stirring rod to dissolve the salt.
6. Optional step: add dry ice instead of ice to the salt water mix after salt has fully dissolved.
7. After the salt has dissolved (or after 5 minutes if dry ice was added in step 5), record the temperature of the water in the beaker in degrees Celsius (°C).
8. Transfer the test tube from the test tube rack into the beaker, making sure not to get any water into the test tube.
9. Observe the oil in the test tube. Record the temperature on the thermometer inside the test tube when the oil solidifies. This is the freezing temperature of the oil.
10. Remove the test tube from the beaker and place it in the test tube rack.
11. Observe the oil in the test tube and record the temperature at which it begins to liquify. This is the melting point of the oil.
12. Repeat steps 2-11 for each oil, using a new pipette and test tube for each oil.
13. Fill in the data table on the next page.

**Data for part 2 (freezing point and melting point of various cooking oils):**

|  |  |  |
| --- | --- | --- |
| **Oil name** | **Freezing point (°C)** | **Melting point (°C)** |
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|  |  |  |
|  |  |  |
|  |  |  |

Temperature of water, rock salt and ice: ˚C

If dry ice was used instead of ice,

Temperature of water, rock salt and dry ice: ˚C

**Questions:**

1. Which oil has the lowest freezing point?

TBD

1. Is the freezing point of the oils the same as the melting point of the oils?

Yes

1. What is the definition of freezing point? Of melting point?

The freezing point of a compound is the temperature at which the compound converts from its liquid phase to its solid phase. The melting point of a compound is the temperature at which the compound converts from its solid phase to its liquid phase. The freezing point and melting point of a substance is the same temperature.

1. What happens during the freezing/melting process in terms of molecular dynamics?

During the freezing process, the molecules in the liquid slow down and reduce their kinetic energy. The substance becomes more dense as the molecules slow down, and eventually the substance converts from a liquid form to a solid form. Alternatively during the melting process, the molecules speed up in their motions, which may be due to an increase in temperature in the surrounding environment. Eventually, the solid converts to a liquid during the melting process of a substance.

1. Besides density, freezing points and melting points, what other properties might help you differentiate the oils?

Color, viscosity, odor, physical state at room temperature

1. If you are investigating the use of these oils for biodiesel engine, which oil will you choose? Why?

Choose the oil with the lowest freezing point, so that it will freeze at lower temperatures and can be used in more locations, including those that get really cold in the winter.

1. What benefit do you think biodiesel has over the traditional diesel gas?

Biodiesel can be synthesized from vegetable oils, and can be done without the use of fossil fuels. Traditional diesel gas is made using the fossil fuels, which is a depleting resource. Because of this, traditional diesel gas is not sustainable.

1. What are some examples of chemical change? How are they different from a physical change?

Some examples of a chemical change include combining carbon dioxide and water to make sugar (photosynthesis), mixing an acid with a base to make a salt and water, and burning wood to create ashes. A chemical change occurs when something new has been formed, or if something has decomposed. A physical change does not require the formation of something new or decomposition of a substance. Instead, a physical change is one in which the state of matter changes. i.e. ice melts (solid changes to liquid), dry ice subliming (solid changes to gas), boiling water, or crumbling a post-it note.

**Further thinking:**

1. What is the freezing point of water?

0˚C

1. What was the temperature of the water with rock salt and ice in it?

TBD

1. If dry ice was used, what was the temperature of the water with rock salt and dry ice in it?

TBD

1. How low can the temperature of water and rock salt go before ice forms (water solidifies)?

TBD

1. Explain why the freezing point of water is lowered with the addition of rock salt.

The rock salt lowers the freezing point of water because its presence does not allow water to create its pure crystalline lattice structure. By incorporating itself within the water crystals, the salt is an impurity and more energy from the surrounding environment is required to form solid ice.

**Lesson 2: Synthesis of Biodiesel**

**Student Sheet**

This lesson contains 2 parts:

* Part 1: Synthesis of biodiesel
* Part 2: Separation and purification of biodiesel

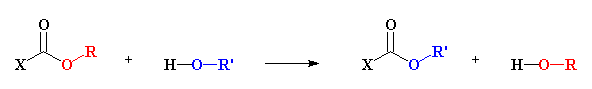
**Background:**

Biodiesel is a fuel made from vegetable oil through a reaction called *transesterification*. Transesterification is a reaction between an ester and an alcohol in which the -O-R group of the ester and the -O-R' group of the alcohol trade places.

Alcohol

Ester

transesterification



**Figure 1**: A general transesterification reaction.

Source: <http://pslc.ws/macrog/kidsmac/glossary.htm>

The main component of vegetable oil is a molecule called triglyceride (also known as triacylglycerol). Triglyceride is composed of a glycerol group linked by an ester bond to 3 fatty acid molecules. Figures 2, 3 and 4 show the chemical structures of triglyceride, glycerol and fatty acids.

**Figure 2:**

Triglyceride structure

**Figure 3:**

Glycerol structure



R, R’ and R” are long alkyl (hydrocarbon) chains ranging from 12-20 carbons in length

**Figure 4:** Fatty acid structures. Fatty acids contain a polar carboxylic acid group (-COOH) and a nonpolar alkyl group (-R, -R’, -R”). The conversion of a polar carboxylic acid to a nonpolar methyl or ethyl ester changes the properties of fatty acids to be similar to properties of fossil fuel diesel.



The ester group in triglyceride will exchange places with the alcohol group in methanol or ethanol\* to form biodiesel. Biodiesel is a methyl or ethyl ester (depending on whether methanol or ethanol is used) of a long chain fatty acid hydrolyzed from triglyceride.

**Figure 5:** The transesterification reaction in the synthesis of biodiesel



The chemical composition of diesel is about 75% saturated hydrocarbons (primarily paraffins including n, iso, and cycloparaffins), and 25% aromatic hydrocarbons (including naphthalenes and alkylbenzenes). The average chemical formula for common diesel fuel is C12H23, ranging from approx. C10H20 to C15H28. The heat of combustion of diesel = 10,700 cal/g.

*\* In this lesson, ethanol is used because its material safety data sheet (MSDS) indicates that it is safer than methanol. Although ethanol is the type of alcohol found in alcoholic drinks such as beer and wine, the type of ethanol used in chemistry labs is different and will be toxic if ingested. Ingesting methanol is also toxic, and can cause blindness.*

**Questions:**

1. What is the name of the reaction used to make biodiesel?

Transesterification

1. What two chemical groups make up a fatty acid? Draw the structures of these chemical groups

Polar carboxylic group (-COOH) and

nonpolar alkyl group (-R, -R’, -R”)

1. What molecule in vegetable oil takes place in the reaction to form biodiesel? Draw the structure of this molecule.

Triacylglyerol (or triglyceride)



**Part 1: Synthesis of biodiesel**

**Materials:**

* 20 mL of ethanol (CH3CH2OH)
* 0.35 g sodium hydroxide (NaOH)
* 20 mL of vegetable oil
* 1 x 125 mL Erlenmeyer flask
* 1 x magnetic stir bar
* 1 x stir/hot combination plate
* 1 x balance or scale
* 1 x weigh paper or weigh boat
* 1 x spatula or scoopula
* 1 x 50 mL graduated cylinder
* 1 x 25 x150 mm test tubes (or multiple smaller tubes)
* 1 x test tube rack
* 1 x thermometer capable of measuring 40˚C

**Procedure:**

1. You will work in pairs.
2. Wear safety goggles, gloves and apron.
3. Measure 20 mL ethanol in the 50 mL graduated cylinder.
4. Transfer the ethanol into the 125 mL Erlenmeyer flask.
5. Add a magnetic stir bar into the flask.
6. Place the flask on a stir/hot plate and turn on the stir function on the stir/hot plate. Stir the ethanol at a medium speed.
7. Using weigh paper (or a weigh boat) and a spatula (or a scoopula), measure 0.35 g of NaOH on a balance.
8. Transfer the NaOH into the flask. Allow the solution to stir until all of the NaOH has dissolved. *Sodium ethoxide has formed inside the flask.*
9. Measure 20 mL of vegetable oil using the graduated cylinder.
10. Transfer the vegetable oil into the flask.
11. Turn on the heat function of the stir/hot plate and allow the vegetable oil to warm to 40ºC.
12. Heat and stir the mixture at 40˚C for 30 minutes.
13. Turn off the stir/hot plate and remove the flask from the top of it.
14. Pour the mixture in the flask into one 25 x 150 mm test tube (or split the mixture into multiple smaller tubes) and place the test tube(s) in the test tube rack.
15. Allow the mixture to sit in the test tube rack overnight to separate into 2 layers: biodiesel and glycerol.

**Part 2: Separation and purification of biodiesel**

**Note:**

After allowing the mixture from part 1 to sit overnight, the test tubes will show 2 layers of materials. The top layer is biodiesel and the bottom layer is glycerol. In this part, the biodiesel will be separated from glycerol and neutralized using a weak acid, 0.1 M acetic acid. Acetic acid is a weak organic acid that you may have already had contact with at home. Vinegar (commonly used for cooking) is 5% acetic acid. Although the acetic acid is a weak acid, do not allow direct contact with skin nor ingest the 0.1 M acetic acid.

**Materials:**

* Biodiesel mixture synthesized in part 1 of lesson 1
* 1 x 25 mL graduated cylinder
* 1 x 50 mL beaker
* 2 x plastic transfer pipettes
* 0.1 M Acetic acid
* Drying agent (i.e. calcium chloride)
* pH indicator strips or pH meter

**Procedure:**

1. Pipette the top biodiesel layer from the mixture and transfer it into the graduated cylinder. Record the volume of the biodiesel in milliliters (mL).
2. Transfer the biodiesel into the beaker.
3. Using a pH strip or a pH meter, measure the pH of the biodiesel.
4. Neutralize the biodiesel.
   1. Add 20 drops of 0.1 M acetic acid into the beaker, or until a neutral pH is obtained. **KEEP TRACK OF HOW MANY DROPS WERE ADDED.**
   2. Mix the solution thoroughly after each addition of the 0.1 M acetic acid.
   3. Use pH indicator strips (or a pH meter) to determine the pH of the biodiesel.
   4. Record the final pH of the biodiesel.
5. Add a few pellets of a drying agent into the beaker. Mix the contents.
6. Label the biodiesel test tubes with your initials, the date and “biodiesel”
7. Store the biodiesel in a dry location at room temperature.

**Data:**

1. Volume of biodiesel in step 1: mL
2. Number of drops of 0.1 M acetic acid

added into order to obtain a neutral pH: drops

1. Assuming 20 drops = 1 mL, how many mL of

0.1 M acetic acid were added to the biodiesel? mL

1. What was the starting pH of the biodiesel (from step 3)? ˚C
2. What is the final pH of the biodiesel (from step 4)? ˚C

**Questions:**

1. If methanol (CH3OH) was used instead of ethanol (CH3CH2OH), sodium ethoxide (CH3CH2O-Na+) would not be formed. What WOULD be formed instead? Draw the structure of this molecule.

Sodium methoxide would be formed. (CH3O-Na+)

1. Why is 0.1 M acetic acid used to neutralize the biodiesel mix?

Because the biodiesel mix is basic from the NaOH

1. How many drops or mLs of 0.1 M acetic acid were required to neutralize your biodiesel mix?

To be determined

**Lesson 3: Analysis of Biodiesel**

**Student Sheet**

This lesson contains 2 parts:

* Part 1: Density and freezing/melting point of biodiesel
* Part 2: Heat of combustion of biodiesel

**Part 1: Density and freezing/melting point of biodiesel**

**Materials:**

* Biodiesel
* 1 x 50 mL graduated cylinder
* 1 x 100 mL beaker
* 1 x triple beam balance or scale
* Ice and rock salt OR dry ice
* Water
* Test tubes
* Thermometer
* Calculator

**Procedure:**

1. You will work in pairs
2. Wear safety goggles, gloves and apron
3. Weigh the empty 50 mL graduated cylinder. Record the mass in grams (g).
4. Transfer 20 mL of biodiesel into the 50 mL graduated cylinder. Record the volume of the biodiesel in milliliters (mL).
5. Weigh the graduated cylinder and biodiesel on a scale. Record the mass in grams (g).
6. Determine the mass of the biodiesel
7. Calculate the density of the biodiesel
8. Transfer the biodiesel evenly into 3 test tubes
9. Set up an ice bath by dissolving some rock salt in water and ice in a 100 mL beaker.
10. Place the test tubes of biodiesel into the beaker, making sure not to get any water into the test tubes
11. Place a thermometer into each test tube
12. At every 1 minute interval, record the temperature of the biodiesel and the physical state of the biodiesel (solid, liquid, etc.)
13. If the biodiesel solidifies (freezes), record the temperature on the thermometer
14. If the biodiesel does not freeze and the temperature inside the test tubes is below -20˚C, the monitoring can stop
15. Remove the biodiesel test tubes from the beaker and allow it to sit at room temperature
16. Monitor the temperature at which the biodiesel melts (the solid or gelled biodiesel is back to liquid state, and seems to clarify in appearance)

**Data for part 1** (Density and freezing/melting point of biodiesel):

***Density of biodiesel***

1. Mass of an empty 50 mL graduated cylinder (in g) \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

2. Volume of biodiesel measured in graduated cylinder (in mL) \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

3. Mass of biodiesel and graduated cylinder (in g) \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

4. Mass of biodiesel (in g) *line 3 – line 1*

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5. Density of biodiesel (in g/mL) *line 4/line 2*

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**Questions:**

1. Given the following information: glycerol’s density is 1.26 g/mL and your biodiesel density from the above section in g/mL, would you be able to identify which layer was which when the biodiesel mix separated? How so?

Yes, I would be able to identify which layer was glycerol and which layer was biodiesel. The density of glyercol (1.26 g/mL) is larger than the density of biodiesel (biodiesel’s density ranges from 0.5-0.8 g/mL depending on the starting oil. It is around 0.6-0.7 g/mL if vegetable oil is used). The material with the larger density will sink to the bottom of the beaker, so the lower layer must be glycerol.

1. What were some possible mistakes that you can make?
   1. Getting water in the biodiesel test tubes, which affects the melting/freezing point of the biodiesel
   2. Using a 10 mL graduated cylinder twice to measure out the biodiesel instead of a graduated cylinder that has >20 mL capacity

***Freezing point of biodiesel***

|  |  |  |
| --- | --- | --- |
| **Time**  **(in minutes)** | **Temperature of biodiesel (˚C)** | **State of biodiesel**  **(solid or liquid)** |
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At what temperature did the biodiesel freeze? ˚C

***Melting point of biodiesel***

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| --- | --- | --- |
| **Time**  **(in minutes)** | **Temperature of biodiesel (˚C)** | **State of biodiesel**  **(solid or liquid)** |
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At what temperature did the biodiesel melt? ˚C

**Part 2: Heat of combustion of biodiesel**

**Materials:**

* Wad of steel wool (with the diameter of a quarter)
* Metallic sample cup
* Plastic transfer pipettes
* Biodiesel
* Triple beam balance or scale
* Empty, dry soft drink can
* Glass stirring rod
* Ring stand with ring holder
* Match sticks
* Deionized water
* 100 mL graduated cylinder
* 200 mL beaker
* Thermometer
* Hotplate
* Tongs
* Calculator

**Procedure:**

* 1. Clamp the ring holder onto the ring stand.
  2. Obtain the dry empty soft drink can and adjust the flip top so that the flip top is vertical and perpendicular to the can opening.
  3. Take the glass stirring rod and stick it through the hole in the flip top of the can. Slide the can so that it hangs from the middle of the glass stirring rod.
  4. Place the glass stirring rod over the ring holder on the ring stand. The soft drink can should hang from the rod, and be below the opening of the ring holder.
  5. Measure 100 mL of deionized water using a 100 mL graduated cylinder
  6. Pour the deionized water into the soda can
  7. Measure the initial temperature of the water in the can using the thermometer
  8. Place the wad of steel wool in the metal sample cup.
  9. Weigh the metal sample cup with the wool in it using the triple beam balance. Record the mass of the metal sample cup.
  10. Pipette 5 mL of biodiesel into the metal sample cup.
  11. Measure the metal sample cup with the biodiesel in it. Record the mass of both and determine the mass of the biodiesel.
  12. Place the metal sample cup with the biodiesel on top of a hot plate. Turn on the hot plate.
  13. Allow the metal cup to get hot, then ignite the biodiesel will inside by using a match. **Exercise extreme caution**.
  14. Using tongs, transfer the metal cup from the hot plate and place it 2 inches below the soft drink can on the ring stand.
  15. Allow the heat from the burning biodiesel to warm the water in the soft drink can. If the biodiesel stops burning but there is still biodiesel left in the metal cup, reignite the biodiesel using a match.
  16. Once the biodiesel has completely burned so that no more is left, record the final temperature of the water in the soft drink can.
  17. Allow the metal cup to cool to room temperature, then weigh it on the scale to determine the mass of any residual biodiesel.
  18. Calculate the heat of combustion of the biodiesel per gram of biodiesel.
  19. Compare the biodiesel’s heat of combustion with that of commercial diesel fuel.

**Data for part 2 (heat of combustion of biodiesel):**

***Heat of combustion***

1. Mass of an empty metal sample cup (in g) \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

2. Mass of a metal sample cup with biodiesel prior to combustion (in g) \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

3. Mass of biodiesel prior to combustion (in g) *line 2 – line 1* \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

4. Mass of metal cup after biodiesel combustion (in g) \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

5. Mass change of metal cup (in g) *line 3 – line 4*

\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

6. Amount of water added into empty soft drink can (in mL)

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7. Density (d) of water (g/mL)

\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

8. Mass of water in soft drink can (in g)

\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

9. Initial temperature of water (in ˚C)

\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

10. Final temperature of water (in ˚C)

\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

11. Temperature change of water (in ˚C) *line 10 – line 9*

\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

12. Heat of combustion of water (in cal/g) \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

Heat of combustion equation: Q=cmΔT

Where Q = heat of combustion (cal/g)

c = specific heat (cal/g ˚C)

*specific heat of water = 1 cal/g ˚C*

m = mass (g)

ΔT = change of temperature change (˚C)

13. Heat of combustion of biodiesel (in cal/g) *line 12 ÷ line 5* \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

14. Is the heat of combustion of the biodiesel you synthesized similar to the heat of combustion of the fossil fuel diesel?

\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

Heat of combustion of diesel = 10,700 cal/g

**Teacher Notes**

**Lesson 1, Part 1: Properties of Various Cooking Oils - Density**

Ten different oils were analyzed by Beyond Benign. Their densities (averaged over 5 trials) are included in the table below.

|  |  |
| --- | --- |
| **Oil** | **Density (g/mL)** |
| Basil | 0.9613 |
| Canola | 0.9346 |
| Corn | 0.9054 |
| Flax | 0.8990 |
| Peanut | 0.8932 |
| Safflower | 0.8660 |
| Sesame | 0.8775 |
| Soybean | 0.9550 |
| Vegetable | 0.9200 |
| Walnut | 0.9225 |

**Lesson 2, Part 1: Synthesis of Biodiesel**

1. The use of a combination stir/hot plate makes this synthesis much easier and efficient than using a hot plate alone.

If hot plates are used (no stir function), it is recommended that you replace solid NaOH with liquid NaOH, and swirl the flask to mix the NaOH with the ethanol. You will need 0.00875 moles of NaOH (see equation 1 below to see how this was determined). Equation 2 shows the calculation for how to prepare a liquid NaOH solution at a 1.0 M NaOH concentration. Equation 3 shows the calculation for how many milliliters of the 1.0 M NaOH solution should be added in order to achieve an equivalent of 0.00875 moles.

*If 8.75 mL of the 1.0 M NaOH stock solution is added instead of 0.35 g NaOH, add 11.25 mL ethanol in step (20.00 - 8.75 mL = 11.25) to achieve a total of 20 mL ethanol in the reaction mix.*

**Equation 1: Determine how many moles of NaOH is in 0.35 grams NaOH**

0.35 grams NaOH × 1 mole NaOH = 0.00875 moles NaOH

40 grams NaOH

*0.00875 moles of NaOH are present in 0.35 grams NaOH.*

**Equation 2: Prepare 50 mL of a 1.0 M NaOH stock solution**

50 mL × 1 L × 1 mol × 40 g = 2 grams NaOH

1000 mL 1 L 1 mol

*Dissolve 2 grams of NaOH in 50 mL ethanol.*

**Equation 3: Determine how many milliliters of 1.0 M NaOH is needed to have 0.00875 moles NaOH**

0.00875 mol × 1 L × 1000 mL = 8.75 mL NaOH

1 mol 1 L

*If 8.75 mL of the 1.0 M NaOH stock solution is added instead of 0.35 g NaOH, add 11.25 mL ethanol instead of 20 mL ethanol (step 3, page 16) to achieve a total of 20 mL ethanol in the reaction mix. 20.00 mL - 8.75 mL = 11.25 mL*

1. If you have enough 50 mL graduated cylinders for each student group, the students can transfer their biodiesel mix into the graduated cylinders instead of test tubes in step 14 (page 16).

**Lesson 2, Part 2: Separation and Purification of Biodiesel**

1. Instruct the students to mix their biodiesel thoroughly after each addition of 0.1 M acetic acid in order to obtain the most accurate pH measurements.
2. Students should aim for obtaining a final pH of 7.
3. Conversion of # of drops to mL (assume 1 mL is approx. 20 drops):

x # of drops × 1 mL = # of mL

20 drops