2018

Experiential Learning Activity: Biodiesel Inquiry Project

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Research based inquiry lab activity: Production of Biofuel from Waste cooking oil and Analysis using Thin Layer Chromatography (TLC) and Gas Chromatography (GC)

Biofuel, renewable and environmentally friendly, is derived from the transesterification of vegetable oils or animal fats. Its properties are similar to petroleum-based diesel and has been used in a diesel engine, the famous "Veggie Van" traveled across the country by biodiesel made by waste cooking oil and/or cooking oil. The high cost of vegetable oil, however, redirects to the interest of converting waste cooking oil into biodiesel. It also makes sense that a large amount of waste cooking oil, generated 4.5-11.3 million liters per year in USA should be converted into biofuel, preventing the environmental hazards from the harmful disposal including illegal dumping.

Pre-lab activity

In the group project, 3-4 students per group, each team member must be assigned responsibilities to work on the project integrating the following questions. Your project report is following APA format, min. 5-paged paper, including proper citations (in-text or works cited), a 10-15 minutes’ group presentation (10 slides, 15 mins talk) will be peer-evaluated.

1. What is Biomass?
2. What is Biofuel?
3. What is transesterification? Propose a chemical reaction.
4. Discuss environmental, social, and economic benefits to using biodiesel?
5. Discuss the sources and threat of microorganism in Biodiesel.
6. interview your family members, friends, or neighbors and find out their awareness of biodiesel production from waste cooking oil
7. Calculate the amount of waste cooking oil produced per week in your house.
8. Visit at least two street vendors, Delis, or restaurants, and find out the disposable methods -how fat and grease leftovers are trapped in their kitchen sink. Please keep fieldnotes and any videos and photos taken, share them in your group presentation.
10. Discuss green chemistry and sustainable science principles within the context of green chemistry.
11. For this lab activity, your group will address a question in which whether waste cooking oil or vegetable oil (soy, palm, olive, or sesame) will be used for making Biodiesel. After the decision made, about 25 ml of waste cooking oil or unused vegetable oil (please ask your parents’ permission) is collected in an empty water bottle, bring it to the class on the day of your presentation.
12. Develop an experimental procedure. Many recipes could be found on the various websites, but I need to approve whether it is a safe procedure – I suggest your group work on this part all together, get approval by your instructor. The reaction will be
carried out using standard laboratory equipment such as a beaker, graduate cylinder, funnel, test tube, stir bar and hotplate. The production of biodiesel will be monitored by Thin Layer Chromatography (TLC) and/or Gas Chromatography (GC). Discuss how these two chromatographic techniques will be used for this laboratory activity.

13. Discuss the major safety concerns for this lab activity. Be specific, some of the chemicals you will use may be toxic, check the Material Safety Data Sheet (SDS) www.msdsonline.com

Students will need three full class sessions to complete the inquiry lab activity. In the first class session, the students will share their research project with classmates, while the second will be used to synthesize the biodiesel, the last will be used for separation, identification, viscosity measurement, and possible flammability test.

Assessment

The overall contribution of this laboratory activity to the final course grade is 20%. The grade is based on the following components:

<table>
<thead>
<tr>
<th>Component</th>
<th>Percentage</th>
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<tbody>
<tr>
<td>Project Report</td>
<td>20%</td>
</tr>
<tr>
<td>Presentation (peer-evaluated)</td>
<td>20%</td>
</tr>
<tr>
<td>Lab work (punctuality, participation, safety rules, and completion)</td>
<td>20%</td>
</tr>
<tr>
<td>Progress report</td>
<td>20%</td>
</tr>
<tr>
<td>Comprehensive project report</td>
<td>20%</td>
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<td></td>
<td>100%</td>
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</tbody>
</table>

Project Timeline for Students and Instructor

Week 1: Due Project Report and Presentation

You will present for 10-15 minutes as if in a town hall meeting.

Pre-lab quiz I will be conducted. Conduct Students’ Survey (Pre-evaluation)

Week 2:

Pre-lab quiz II will be conducted. Production of Biofuel and Soap

Observation, Results (be specific the amounts of chemicals and oil used)

If time is allowed, do separation, and find the Density. Ensure to take photos of before/after reaction.

Week 3: Due Progress Report

Post-lab quiz I will be conducted. Take TLC of your starting material and product. Calibration of GC, run a sample, measure viscosity, and do flammability test.

Week 4: Due Comprehensive Lab Report
Comprehensive Laboratory Report encourages critical and creative thinking because you need to report on what you did, interpret your own data, and reflection how your ideas has been changed. Your comprehensive research paper is following APA format, min. 7-paged paper, including proper citations (in-text or works cited), photos, TLC plate, and supporting data.

1. Title, Abstract (limit 200 words), name and date
2. Introduction
3. Experimental methods/Observations
4. Data: Report the results of the experiment. If you are doing an isolation and purification or a synthesis you should record the actual yield and the percent yield, and any physical constants for that substance such as, density, viscosity, TLC plate, retention times of the gas chromatography
5. Discussion/Conclusion: This is a crucial part of the lab report. Detailed explanations of all your results and observations must be included. Your writing must be concise and easy to understand. The conclusion has to be thorough and you must demonstrate that you understood the principles of the lab.
6. References

Remember: **There is no wrong or right.** If results are different from what is expected DO NOT CHEAT. Instead try to find logical explanations for what happened. This will be taken into account and your grade will not suffer from it as long as you provide a reasonable justification. This is the opportunity to develop your critical thinking abilities and writing skills.
Student Survey: This survey is conducted to gauge the impact of the project, Biodiesel Production on your learning experience. I need your feedback to assess whether the pedagogical goals are achieved.

1. Complete the following survey (for a-f: improved 3: somewhat progressed 2: no change 1: negative experience 0)

   a. My attitude toward the learning of chemistry before this activity ______
   b. My attitude toward the learning of chemistry after this activity ______
   c. My confidence in learning and using chemistry before this activity
   d. My confidence in learning and using chemistry after this activity
   e. My conceptual understanding of course materials before this activity
   f. My conceptual understanding of course materials after this activity

   g. What did you find most valuable about doing the project?
   h. Did the project satisfy your expectation of learning chemistry?
   i. Did the project promote your interest in learning and doing chemistry?
   j. What do you like least about the project?
   k. What was your overall experience working on the project?
CHEM 110

Part A: Making Biodiesel

Biodiesel is an alternative fuel that is made from waste cooking oil, vegetable oil, and animal fat. It can be used directly in diesel vehicles or blended with traditional petroleum diesel. You will discover during this lab how easy it is to make and store biodiesel. The chemical reaction to produce biodiesel is as follows:

\[
\text{Oil (your choice)} + \text{CH}_3\text{OH (methanol)} + \text{NaOH (sodium hydroxide)} \rightarrow \text{Biodiesel} + \text{Glycerine}
\]

Procedure:

**Making Biodiesel:**

1. Measure out 25 mL of oil (your choice) using a graduated cylinder, and pour the oil into one of the Erlenmeyer flasks and add a stir bar. Put this on top of hotplate/stir. Record the volume in your data sheet, table 1.

2. Record in your data sheet, table 2, observations of color, viscosity, clarity, and other aspects of the appearance of the starting material (oil).

3. Check that the temperature of the water bath is between 50 °C and 60 °C. Place the Erlenmeyer flask containing the oil sample into a water bath.

4. While your oil is warming, measure out 5.0 mL of methanol using a 10 mL graduated cylinder and pour it into a second Erlenmeyer flask.

5. To the Erlenmeyer flask containing the methanol, add 0.2 g of NaOH(s). Swirl your mixture of methanol and base gently to mix. This mixture is called methoxide. • If using a solid base, the mixture should be swirled or stirred until the solid base dissolves completely.

6. Pour the methoxide (from step 5) mixture into the warm oil in the Erlenmeyer flask.

7. Stir the mixture for 10 minutes. Record the change in your table 2. Stir additional 10 mins (total 20 mins), record it and consult your reaction process with me. The mixture will become cloudy.

8. If the reaction is successful, you should start seeing two layers developing inside the flask. The heavier glycerin will start to settle to the bottom soon after you stop mixing the reactants. The biodiesel will be in the upper layer. The biodiesel varies in color depending on the oil used. This will take at least an hour, but longer is better. The mixture should sit overnight to completely react. Check it with me whether you collect biodiesel today, or leave it in the hood for a week. If so, skip steps #9-12 today, start making soap with your oil (go to Part B section).
Collecting Biodiesel:

9. Use a disposable pipette to carefully remove the top layer, containing the biodiesel, from the Erlenmeyer flask and move it to a clean graduated cylinder.

10. Record the volume of the biodiesel transferred from the Erlenmeyer flask in Table 1.

11. Record observations of color, viscosity, clarity, and other aspects of the appearance of the starting material (oil) and the product (biodiesel) collected in Table 2.

12. Place the biodiesel into an appropriately labelled container. Label your name and today’s date.

Note: This is crude biodiesel and is NOT of a high enough quality to put directly into a vehicle. In industrial processes, it must go through a process called “washing” to remove excess glycerin, base, and alcohol.
Datasheet I: Biodiesel Production:

**Oil sample type:** __________________________

Table 1: Quantitative Observations of Reaction

<table>
<thead>
<tr>
<th>Volume of Oil used (mL)</th>
<th>_______ mL</th>
</tr>
</thead>
<tbody>
<tr>
<td>Volume of biodiesel (mL)</td>
<td>_______ mL</td>
</tr>
</tbody>
</table>

Percent yield \((\text{product/reactant}) \times 100\) of the reaction % Yield

Table 2: Qualitative Observations of Reaction

Observations of Reactants (e.g., color, smell, viscosity, etc.)

Initial (before stirring):

:  

After 10 mins stirring:

:  

After 20 mins stirring:

:  

Observations of Products (e.g., color, smell, viscosity, etc.)
Analysis:

1. Use the data from table 2 to compare the two products (biodiesel and glycerin) with the starting reactants (methanol and oil).

2. In commercial production of biodiesel, yields are often around 80% biodiesel. How does your yield compare to the commercial yield?

3. Suggest modifications to the procedure and design of the experiment that would either increase the amount of product from the process.
Part B: Making Soap

The art of making soap began in the ancient world several thousand years ago by boiling animal fat with wood ashes containing alkaline material facilitating the cleavage of ester linkages. Currently, soap is made by boiling animal fats or vegetable oils (triglycerides = esters of fatty acid) with an aqueous sodium hydroxide solution (lye) to convert the fatty acid esters into long carboxylate chains, and glycerol. This reaction, called saponification (latin: *saponis* = soap). The nature of a soap depends upon the fats and oils which are used in the saponification as well as on additional materials (e.g. colorants, fragrances, preservatives, exfoliants) which are added during processing. Traditional folk methods use animal fat such as tallow (rendered beef fat) or lard (rendered pork fat) and very few additives to enhance the aesthetic appeal of the soap. Most craft-oriented books favor the use of vegetable fats and oils for numerous reasons ranging from animal rights to cosmetic properties.

Oil + NaOH \(\rightarrow\) Soap + Glycerin

**PROCEDURE:**

1. Place 10.0 mL of vegetable oil in a 125 mL Erlenmeyer flask. Warm on a hot plate to a gentle boil.
2. Slowly add 10.0 mL of 25% aqueous NaOH solution into the oil from step 1. **CAUTION:** Goggles, gloves and fume hood recommended. Use caution to avoid splashing the caustic solution on skin or in eyes. Do not breath vapors from the solution. Stir constantly to prevent a solid mass of sodium hydroxide from clumping on the bottom of the container. The solution will become quite hot. Stir the solution. Record the observation in your data sheet.
3. Heat the mixture on a water bath for at least 20 minutes. Record the observation in your data sheet. Stir frequently and as vigorously as possible without splashing. A paste consisting of soap, glycerol and excess NaOH (caution: caustic) is produced. Using the pH paper, test the pH of crude soap, record it in your data sheet.
4. To precipitate or “salt out” the soap, add (within 5 minutes) 50 mL of saturated NaCl solution while stirring the mixture. The precipitated soap will float to the top while glycerol and NaOH will remain in the lower portion of the flask. Separate the soap by vacuum filtration (Buchner funnel), washing it with several portions of ice cold water.

**CAUTION:** The final cured soap may still be relatively harsh and care should be taken in using the final product, especially in the case of individuals with sensitive skin. The final product may contain unreacted sodium hydroxide which is a severe irritant. Take care to avoid contact with the eyes. It is not recommended to use the final product as hand soap.
Data sheet II: Making soap

1. Record the observation after mixing oil and NaOH (sodium hydroxide solution)

2. Record the observation after stirring 20 mins:

3. Record the pH of your soap:

4. In the “salting out” process, what products are separated from soap?