Chemistry 140

Please have the following pages ready **before** class on Wednesday, October 11. For this particular writeup, please write an **abstract** and **paper-clip** it to the front of your individual writeup. The abstract and the carbon-copy pages of the write-up is due in class on **Wednesday, October 25.**

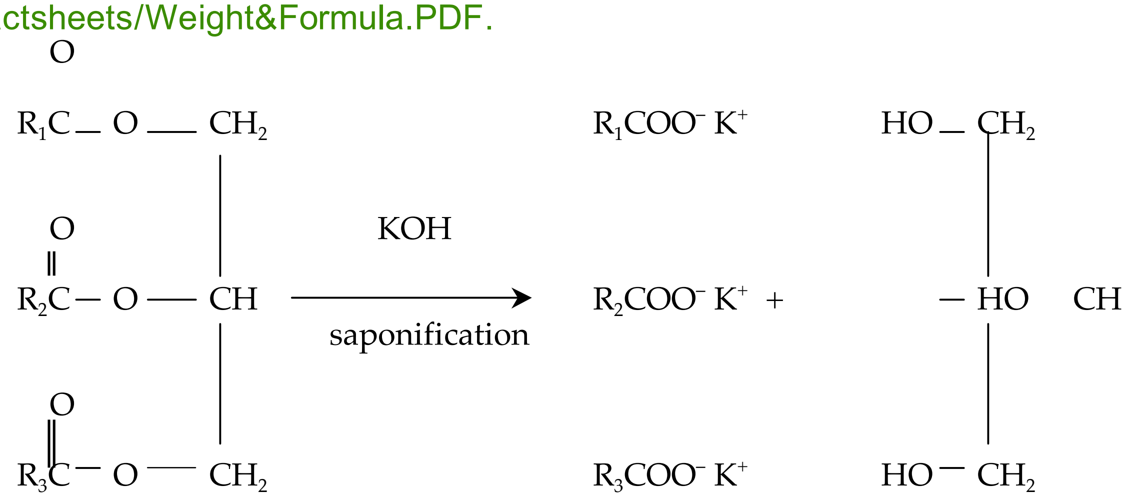
Introduction

Diesel is a common fuel used to power many large vehicles and heavy equipment (such as tractors). It is made from crude oil that was formed millions of years ago by the decomposition of prehistoric plants and animals. In an oil well, crude oil is pumped out of the ground, and is transferred (often by large ocean tankers) to oil refineries. Crude oil contains widely varying organic (carbon-based) chemicals which range in size from small molecules with only 1 carbon atom (C_1) to very large molecules with over 20 carbon atoms ($>C_{20}$). By using a distillation tower, crude oil is broken into various fractions (or components) based on the size of the molecule. Distillation towers operate on the principle that smaller carbon compounds have lower boiling points, while larger ones have higher boiling points. A distillation tower works much like a distillation apparatus you might use in the laboratory. Crude oil is heated as it enters the distillation tower so that it boils. The vapor is then gradually cooled as rises up the tower. Since less-volatile compounds condense at higher temperatures they are separated low down the tower. The more-volatile compounds rise higher in the tower before they start to condense. Thus, fractions of crude oil can be separated by the decreasing temperature as they move up the tower.

Chemists have created a substitute for diesel - biodiesel - by chemically changing various fats and oils. Fats and oils can be burned without any chemical alteration (old whaling ships used to burn the 'blubber' or fat from whales in their oil lamps). By using a chemical technique called transesterification chemists can turn oils from various crops (such as rapeseed and soy) into a viable diesel substitute. One of the major advantages of using biodiesel instead of diesel is that biodiesel is derived from a renewable resource. Diesel comes from crude oil, which takes millions of years to form. While over the course of the next few million years more underground pools of crude oil will be formed, it is consumed at a rate which is drastically faster than the rate at which it is forming. Many experts believe that at current production, crude oil will be economically (and thus essentially) exhausted in the next 40 years. Biodiesel, however, is made from renewable resources; oils derived from farm crops, such as soybeans. One major focus of green chemistry is to develop new chemical processes and products which eliminate the need for using non-renewable starting materials, by replacing them with renewable starting materials. Biodiesel also creates less sulfur emissions when it is burned which helps reduce acid rain and it also breaks down more quickly in the environment; thus lessening the consequences of an accidental spill when compared to crude oil. Finally, while combustion of a hydrocarbon always creates carbon dioxide (CO_2), biodiesel is made from crops that need carbon dioxide to grow. Thus, much of the carbon dioxide released from burning bio-diesel, is absorbed by the crops growing to make the fuel and no excess carbon dioxide is produced to contribute to global warming.

Biodiesel is a mixture of methyl esters of fatty acids (shown below) and can be synthesized from vegetable cooking oil. Enough fuel will be produced from this experiment to burn in a later activity, as well as to donate to the organic chemistry class to analyze for contents and purity. The synthesis is a simple chemical reaction that produces biodiesel and glycerol. Cooking oil is mixed with methanol, while potassium hydroxide is added as a catalyst. The products separate into two layers, with the biodiesel (desired product) on the top. The biodiesel is separated and washed and is then ready for further experimentation.

In this experiment, you will use soybean oil as the starting material. Soybean oil is composed of triglycderides (the "R" groups in the reaction diagram shown below). What are the "R"s made of? They are the fatty acids mentioned above, and soybean oil's triglycerides contain 52% omega-6,9-linoleic acid ($C_{19}H_{34}O_2$), 25% oleic acid ($C_{19}H_{36}O_2$), 12% palmitic acid ($C_{17}H_{34}O_2$), 6% omega-3,6,9,-linoleic acid ($C_{19}H_{32}O_2$), and 5% stearic acid ($C_{19}H_{38}O_2$). This information was obtained from the National Biodiesel Board site, www.biodiesel.org/pdf_files/fuelfactsheets/Weight&Formula.PDF.



Triglycerides carboxylic acid salts glycerol then

$$RCOO^- K^+ + CH_3OH \xrightarrow{\text{basic conditions}} RCOOCH_3$$

methanol biodiesel

For this experiment, assume that soybean oil is made of nothing but triglycerides composed of R = omega-6,9-linoleic acid. Thus its structure is:

$$CH_3CH_2CH_2CH_2CH_2CH{=}CHCH_2CH{=}CHCH_2CH_2CH_2CH_2CH_2CH_2CH_2C({=}O){-}O{-}CH_2$$

$$CH_3CH_2CH_2CH_2CH_2CH{=}CHCH_2CH{=}CHCH_2CH_2CH_2CH_2CH_2CH_2CH_2C({=}O){-}O{-}CH$$

$$CH_3CH_2CH_2CH_2CH_2CH{=}CHCH_2CH{=}CHCH_2CH_2CH_2CH_2CH_2CH_2CH_2C({=}O){-}O{-}CH_2$$

which reacts to become

$$CH_3CH_2CH_2CH_2CH_2CH{=}CHCH_2CH{=}CHCH_2CH_2CH_2CH_2CH_2CH_2CH_2C({=}O){-}O{-}CH_3$$

Introduction adapted from The Advancing Chemical Science Network

Your name, your partner's name, date of experiment

Lab 2: The synthesis and characterization of biodiesel from soybean oil

Part 1. Purpose

Copy the following into your notebook.

To determine the typical percent yield and to characterize the synthesis of biodiesel from soybean oil.

Write the chemical equation that shows the reaction from the triglyceride (you may abbreviate it "triglyceride") to the omega-6,9-linoleic acid methyl ester. Balance the equation, and include any other reactants or products in proper stoichiometric ratio. Include any catalysts under the reaction arrow.

Part 2. Materials and methods

Look over the procedure, and decide what pieces of fluid-holding or fluid-measuring glassware you will be using.

Glassware:

Other equipment:

Chemicals: Distlled water, soybean oil, methanol, 9 M potassium hydroxide

Sketch the setup for a) the stirring step and b) the washing step.

Safety — **Methanol is a strong organic solvent. 9 M potassium hydroxide is caustic. Both should be handled with care.**

Part 3. Procedure

Copy the following into your notebook; you may cut this part out of the handout and tape it in the appropriate area of your notebook.

Synthesis

1. Measure 100. mL of soybean oil. Record its mass.

2. Carefully add 15.0 mL of methanol.

3. Slowly add about 1 mL of 9 M potassium hydroxide.

4. Stir the mixture for 10 minutes. **Be careful not to spill!**

5. Allow the mixture to sit and separate **for at least one hour.**

6. Carefully remove the top layer using a disposable pipet.

7. Wash the product using about 10 mL of distilled water. Mix thoroughly in a separatory funnel.

8. Allow the mixture to sit and separate **for at least one hour.**

9. Carefully remove the top layer using a disposable pipet.

10. Measure, to three significant figures, the volume of your recovered product.

11. Measure, to the milligram, the mass of your recovered product.

Characterization

12. Following your instructor's instructions, obtain an infrared (IR) spectrum of your product.

Waste disposal — Pour the fluid layers you don't use, plus any excess chemicals in the "biodiesel waste" container in the hood.

Part 4. Original data

You don't necessarily need to make a table out of the following measurements, but all of these measurements, with the appropriate units, should be found neatly arranged in this part of the notebook:

Msss of soybean oil, volume of methanol used, length of stirring time, volume of product recovered, mass of product recovered.

This is also the section in which you make any **notes of changes** that occur to the materials as the reaction progresses (i.e., "observations"). You may also note these changes in the "Procedure" section at the steps where the changes occur, but put a box around the observations so they stand out from the procedural text.

This is also the section in which you attach (use tape) your IR spectrum.

Part 5. Calculated results

Calculate the **expected yield** (in grams) of product given your starting conditions.

Calculate your percent yield. The formula for % yield is:

$$\% \ yield = \frac{\exp ected \ \ yield - your \ \ yield}{\exp ected \ \ yield} \times 100\%$$

Note that nothing in the formula precludes a greater than 100% yield.

Part 6. Group results

Write your group's % yield on the overhead projector and record all groups' % yields. Calculate a mean and standard deviation for the class.

Part 7. Questions

1. What physical changes did you note that led you to believe that a chemical reaction took place?

2. What would you suspect had gone wrong, if you'd gotten greater than 100% yield? Name some sources of error in the experimental procedure which might have led to that result.

3. In the group results, were there any outliers? Or, a much better question, is there such a thing as a "typical" percent yield from this experiment?

4. Why isn't a more precise volume needed for the potassium hydroxide?

5. What is the purpose of the "washing" step?

6. In the commercial production of biodiesel, 1200 kg of soybean oil produces 1100 kg of biodiesel. How does your yield compare to this?

7. Examine the IR spectra of pure biodiesel and pure soybean oil. Even though you may not know anything about IR spectroscopy, what should the IR spectrum of your product look like? If it does not, what does this suggest about the purity of your product?

Part 8. Conclusion

Don't worry about this section in this lab writeup.

Abstract

Use the same format as before (you and your partner's name, North Seattle Community College, TITLE OF THE EXPERIMENT, etc.).

Start off with something like "In the synthesis of biodiesel from soybean oil..." and mention the percent yield you achieved. Compare this to what the class got and what can be commercially obtained (question 5). If significantly lower, explain one *specific* major source of error. Finally, state whether the IR spectrum showed whether your product was pure biodiesel or not.