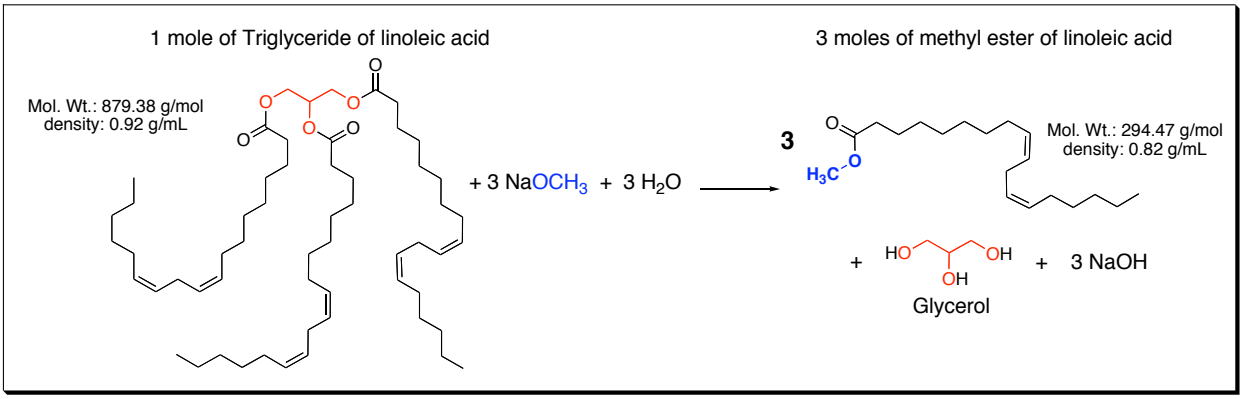
**Experiment 4 - A Small Scale Synthesis of Biodiesel**

Biodiesel has gained a lot of attention over the past decade because of its use as an alternative to fossil fuels for automobiles and trucks. Biodiesel consists of the methyl esters of fatty acids found in vegetable oil, obtained through a process called transesterification (see **Scheme 1** below).[1] The combustion of biodiesel releases carbon dioxide gas into the atmosphere similar to the combustion of fossil fuels. However, biodiesel is viewed as being more carbon-neutral than fuels obtained from petroleum products because the carbon in plant-derived biodiesel originated from carbon dioxide in the atmosphere.




**Scheme 1.** Synthesis of biodiesel from corn oil *via* "transesterification."

There are several cars and light duty pick-up trucks on the market today engineered to operate using multiple fuel sources, including biodiesel. More commonly, people purchase kits that enable them to convert their own gasoline-powered cars to run on biodiesel. Many of these people make their own biodiesel at home, procuring the vegetable oil from restaurants in their community. This re-purposing of used cooking oil converts an industrial waste product into a usable commodity, providing additional validation for the use of biodiesel as an alternative to fossil fuels.

In this experiment, students will synthesize biodiesel from a commercially available vegetable oil.[2,3] Products will be analyzed through visual inspection as well as $^{1}$H NMR.

**Notebook Preparation -** This experiment is performed individually.

**Students were provided with a 20-mL scintillation vial to fill (~3/4 full) with vegetable oil from home, such as corn, sesame, peanut, or soybean oil. Don't forget to label the vial.**

- *Purpose:* One-sentence description of the experimental and learning objectives, in addition to the reaction scheme (**Scheme 1**).

- *Reagent table*: List the amounts (mg or mL and mmol), molar equivalents ("equiv."), and physical properties (MW, bp or mp, density, one-word hazard) of each chemical in the reaction scheme.

- *Hand-written procedure*: step-by-step instructions in your own words, not directly copied. Organize using numbered steps or bullet points, including diagram of reaction set up. A flow chart for the procedure is recommended, but not strictly required.

- *Safety & Clean*-up*:* create your own table after the procedure using the specific clean up and safety notes given within the procedure itself

## EXPERIMENTAL PROCEDURE[2,3]

### Step A: Preparation of sodium methoxide ($NaOCH_3$) solution (Eq 1)

$$NaOH + CH_3OH \rightarrow NaOCH_3 + H_2O \quad (1)$$

This solution should be prepared in a fume hood. Weigh out 1 pellet (approximately 0.10 g) of NaOH and transfer it into a mortar. Use a pestle to grind the NaOH pellet into a fine powder. NaOH is hygroscopic, caustic, and will cause burns on contact so use caution. To facilitate the transfer of the NaOH powder, pour 1.5 mL of methanol into the mortar. Methanol is flammable and poisonous and should be handled with care. Use the pestle and/or a glass stir rod to break the NaOH powder free from the interior of the mortar so that it can dissolve into the methanol. Transfer as much of the contents as possible to a 50 mL round-bottom flask using a glass funnel and stir rod to control the flow of liquid. *Remember this is a very basic and caustic solution!* Clean up any spills immediately with a spill mat. Add 1.5 mL more methanol into the mortar to dissolve any remaining NaOH and transfer it to the RBF (for a total of 3 mL MeOH). Add a magnetic stir bar to the flask and use a stirring hot plate to gently stir the mixture at room temperature for 5-10 minutes (or until the NaOH has dissolved) as you begin Step B.

### Step B: Transesterification reaction (Eq 2)

$$1\ (\text{Triglyceride}) + 3\ NaOCH_3 \rightarrow 3\ (\text{methyl ester of fatty acids}) + 3\ NaOH \quad (2)$$

Use a graduated cylinder to measure 12 mL of your vegetable oil and add it to the $NaOCH_3$ solution prepared in step A. Attach a water-jacketed condenser and reflux the solution in a boiling water bath for 20 minutes. Since there is not ample hood space for the reflux, it is extra important that ice cold water is running through the condenser at all times. The solution must be stirred vigorously so that the mixture of methanol and vegetable oil does not separate into two layers. Once the reflux is complete, remove the heat but allow the solution to cool before handling the flask.

**Step C: Isolation & Analysis**

Transfer the crude reaction mixture to a large test tube while the solution is still a little bit warm but not hot. Allow the test tube to sit for 5-10 minutes undisturbed. Record observations regarding the appearance of the products in comparison to the starting materials (color, viscosity, etc.). Analysis will be primarily through visualization. Exact yield will not be determined, but volumes should be estimated by eye-balling similar amounts of water in another test tube.

Each student will prepare a sample for NMR analysis! Add 2 drops of biodiesel to 800 uL $CDCl_3$ directly in the NMR tube per TA demo. Use a long-stem pipet to mix the solution. Do not invert the NMR tubes, as the caps are not spill proof! Your sample must contain a label with the following, or it will not be analyzed: "Last Name, First Initial, Section Day/Time, TA, Vegetable Oil ID." Spectra will be posted online. The methyl ester protons, alpha protons, and any vinylic protons will be integrated.

**Step D: Waste disposal & Clean up Procedure**

*Put into table format along with safety notes from the procedure…*

Dispose of the products in liquid waste. Wash all glassware at least twice with hot water (if you can get it) and lots of soap. Thoroughly clean all countertops with the cleaner provided (spray bottle) and dry with a paper towel. Do not leave greasy counter-tops!

**References**

1. Leung, D. Y. C.; Wu, X.; Leung, M. K. H. *Appl. Energy* **2010**, *87*, 1083−1095.

2. Meyer, S. A.; Morgenstern, M. A. *Chem. Educator*, **2005**, *10 (2)*, 130-132.

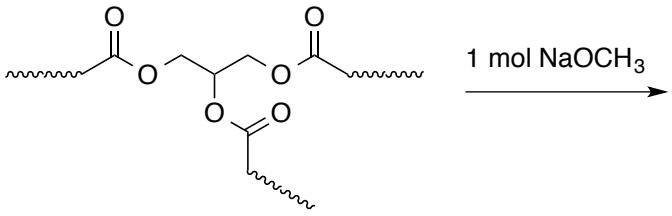
3. Yang, J.; Chunli, X.; Li, Baozin, L.; Ren, G.; Wang, L. *J. Chem. Ed.* **2013**, *90*, 1362-1364.

## INTRODUCTION: PRE-LAB QUESTIONS

1) What is the difference between a saturated fat and an unsaturated fat? How is an unsaturated fat different from a poly-unsaturated fat? Draw an example of each, including the common name.

2) Use specific IMFs to explain why methanol and vegetable oil would separate into two layers if the reaction were not stirred sufficiently.

3) Draw the arrow-pushing mechanism and products of the reaction of a triacylglycerol with just one mole of $NaOCH_3$.




4) Calculate the mmols of methanol and the vegetable oil you will be using. You will need to look up the constituent fatty acids in that particular triglyceride and estimate the molecular weight. If you cannot find the metrics of your oil, use the ones provided in **Scheme 1**. Show your work. Determine the limiting reagent and the molar equivalents of the reagent in excess. Briefly comment on the mole ratios, taking into account your response to pre-lab #3.

5) Calculate the theoretical yield of biodiesel in milliliters. Show your work.

## RESULTS: POST-LAB QUESTIONS

1) Report the approximate yield (mL) of biodiesel and calculate the percent yield.

2) The analysis of the $^1H$ NMR spectrum will not be very detailed, as the long chains cause many peaks to overlap. Instead, create a table with the observed peaks and assign the distinctive peaks (anything not in the alkyl region) to the best of your ability. Include the structure of the methyl ester and label accordingly.

3) Report your observations of the products in comparison to the starting materials (methanol and vegetable oil) and to each other (glycerine and biodiesel). Why is biodiesel immiscible with methanol, glycerol, and water (discuss the IMFs of each)?

4) Briefly describe one method that could have been performed in the lab to purify the biodiesel product (hint: see reference 1).

**Exp 4 – Synthesis of Biodiesel** Name _____

Section Day _____ Time _____ TA Name_____

CHEM 110L GRADING RUBRIC – Use as cover page for report

| SECTION | INSTRUCTOR COMMENTS | POINTS ASSIGNED |
|---|---|---|
| **IN-LAB QUIZ** | | **/ 5** |
| **INTRODUCTION**<br>Original responses to pre-lab questions with TA initials | | **/ 50** |
| **RESULTS**<br>The main results are stated, as outlined in the in-lab questions, using complete sentences | | **/ 50** |
| **NOTEBOOK PAGES**<br>Proper format: purpose, reaction scheme, chemical info table, procedure, waste & clean-up notes | | **/ 20** |
| **NEATNESS, ORGANIZATION, & LAB TECHNIQUE**<br>Proper order and format. Safety rules followed, equipment used properly | | **/ 25** |
| **LAB REPORT TOTAL** | | **/ 150** |