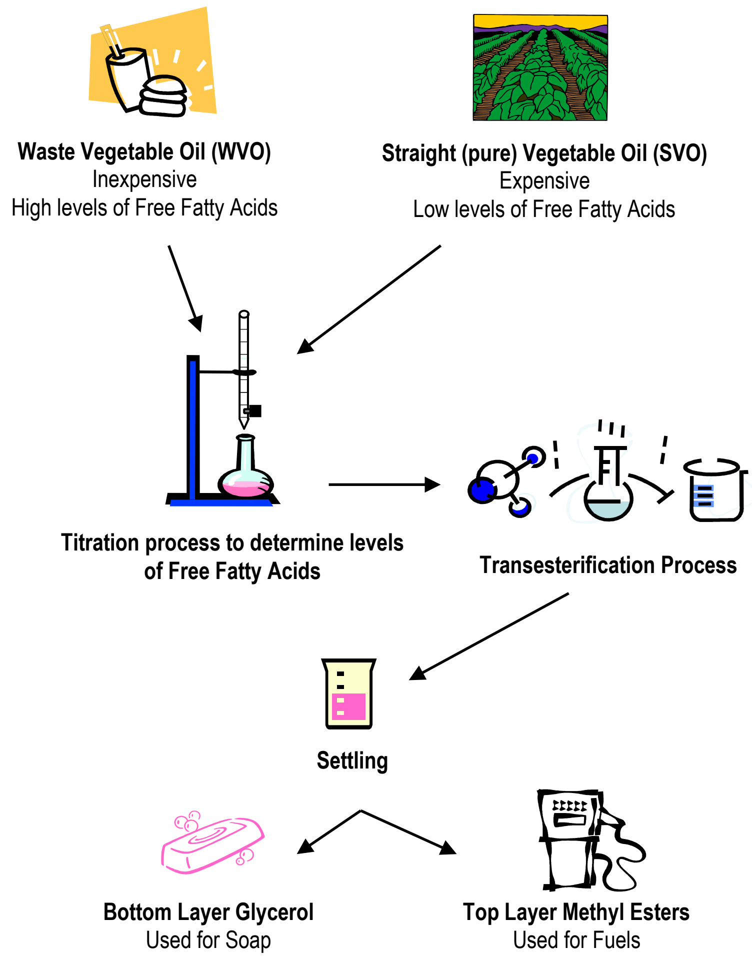
Name:

The Biodiesel Lab

Determination of Free Fatty Acid Concentration Through the Use of Titration

Biodiesel is an alternative to petroleum-based diesel fuel. Biodiesel is a renewable form of fuel manufactured from natural oils, such as soybeans or animal fat. Biodiesel fuel is used in diesel engines (like those found in school buses), and provides a cleaner burning alternative to petroleum diesels. As a renewable resource, another advantage of biodiesel is that it can be manufactured domestically.

Biodiesel is manufactured through a process called **transesterification**. This process begins with vegetable oil. This oil is either pure oil, referred to as straight vegetable oil (SVO), taken directly from plants such as soybeans or corn, or from waste vegetable oil (WVO), the leftover oil after cooking. Restaurants produce a large supply of WVO that can be used to produce biodiesel. The transesterification process separates the oil into two by-products, methyl esters (biodiesel fuel) and glycerol (a valuable by-product used to make soap and other skin care products).



Both gasoline and biodiesel contain hydrocarbons. Hydrocarbons are long chains of hydrogen and carbon atoms. Gasoline and *petroleum-based* diesel are made from fossil fuel, whereas biodiesel is made from recently grown plants. Therefore, when biodiesel is burned as a fuel, its hydrocarbons are also released into the atmosphere as CO_2. However, the carbon released into the environment by burning biodiesel is equal to the amount of carbon taken in by the plants used to make biodiesel fuel. Therefore, the carbon cycle of biodiesel can be described as being "carbon neutral". That is, the amount of CO_2 released into the atmosphere from the burning of biodiesel is equal to the amount of carbon consumed (i.e., taken from the atmosphere) in the production of biodiesel.

Biodiesel Objectives

- Understand the process of titration; perform two (2) titrations
- Analyze the results of the completed titration
- Understand how these results are used to produce biodiesel

Laboratory Activity

As earth-friendly conscious consumers, your class has purchased a new car for the school that will use alternative fuels, such as biodiesel, for power. You can use waste vegetable oil converted to biodiesel, and you've begun collecting samples of waste vegetable oil from local restaurants. Since the vegetable cannot be added directly into the car, you need to actually turn it into biodiesel fuel by a chemical reaction called transesterification. Before starting that chemical reaction, you will need to measure the level of free fatty acids using titration.

Waste vegetable oil (WVO) is converted into biodiesel fuel in a process called transesterification. Transesterification is a reaction that uses a catalyst (NaOH) and an excess of methanol (a type of alcohol) to convert WVO (triglycerides) to biodiesel (methyl esters). However, the transesterification reaction is sensitive to the presence of free fatty acids (FFA). FFA's are the result of oil degradation. As a result, WVO tends to have higher levels of FFA's than pure (unused) vegetable oil. Because FFA's can slow or stop the transesterification reaction, it is important to know the amount of FFA's present in each batch of WVO so adjustments can be made to the transesterification process to neutralize the excess FFA's. The same chemical that is used to catalyze the reaction - NaOH, can neutralize FFA's. Therefore, in order for the transesterification reaction to proceed, enough NaOH will be needed to neutralize any FFA's in the oil in addition to the amount of NaOH that is used as a catalyst.

The amount of FFA varies for each batch of WVO, which is why it is necessary to determine the actual level of FFA in each batch of WVO before beginning the transesterification process. The way to determine the levels of FFA is through a process called titration. A titration is a way of determining the concentration of a substance by adding to it a standard reagent of known concentration until we see an effect (often a color change). In our case the dissolved substance is the FFA, the reagent of known concentration is the sodium hydroxide (written as NaOH), and the effect is color change (from clear to bright pink), which indicates a specific change in pH.

How does a color change relate to pH level and the presence of FFA's? We use a pH indicator in our titration process called turmeric. ***Turmeric is yellow in color, but turns pink in basic conditions***. Thus, when our solution turns pink (through the addition of NaOH), it indicates that we have neutralized the FFA's present in our sample (shifted the pH value from acidic to basic conditions). Previous research has determined that each millilitre (ml) of NaOH added to your titration mixture translates into an extra gram of NaOH that must be added to the transesterification reaction.

In this laboratory, we will be determining the acid value of your batch of WVO. The acid value is the amount of NaOH that is needed to neutralize all of the free fatty acids in your batch of WVO. Each milliliter of NaOH used in your titration reaction corresponds to a gram of NaOH needed to neutralize the free fatty acids in 1 liter of your WVO. For example if you needed 2 ml of NaOH to induce a color change in your titration reaction, your acid value would be 2. This means you would need 2 grams of NaOH to neutralize the free fatty acids in 1 L of waste vegetable oil.

MATERIALS

Unused Vegetable Oil	Micropipette (100-1000 µl range)
Waste Vegetable Oil	Micropipette tips (blue and yellow)
Graduated cylinder	Micropipette (20 - 200 µl range)
0.01% NaOH (a basic solution)	Isopropyl alcohol
Glass beakers	Indicator solution (turmeric)
Clamp Stand	Burette

Which type of oil contains more FFA's? Why? What is your hypothesis?

PART I – Determine the amount of Free Fatty Acids in Straight Vegetable Oil (SVO)

- ☐ 1. Pour 10 mL of isopropyl alcohol into a graduated cylinder. Add the isopropyl alcohol to the small glass beaker labeled "SVO."
- ☐ 2. Add 1000 μL of the straight vegetable oil sample from the tube labeled "SVO" to the small glass beaker labeled "SVO" containing the isopropyl alcohol. **Describe the appearance of the liquid in the beaker.**
- ☐ 3. Gently swirl the contents of the beaker for 10 seconds. **Describe the appearance of the liquid in the beaker.**
- ☐ 4. Add 50 μL of the indicator solution (turmeric) to the oil/isopropyl alcohol mixture in the "SVO" beaker. Gently swirl the contents of the beaker for 10 seconds. **Describe the appearance of the liquid in the beaker.**
- ☐ 5. Read and record the amount of sodium hydroxide (NaOH) in the burette. Be sure to be precise in your measurement and record the data below:

  Starting amount of 0.01% NaOH solution in burette ________ mL

- ☐ 6. Carefully position the burette directly over the SVO beaker. Carefully add one (1) drop of the NaOH solution from the burette into the beaker by turning the stopcock clockwise. **DO THIS SLOWLY and CAREFULLY. Only add ONE DROP at a time.**
- ☐ 7. Swirl the beaker. Record the number of drops and any color changes in **Table 1** immediately after adding the NaOH to the beaker.

Table 1 – Number of 0.01% NaOH added to Straight Vegetable Oil (SVO)

Number of Drops	Color after swirling	Did the color change remain for 15 seconds? Yes or No?

- ☐ 8. Repeat steps six (6) and seven (7) until the solution changes color and **remains** that color for at least 15 seconds.
- ☐ 9. Now that your solution has changed color, record the remaining amount of NaOH in the burette in Table 2 below:

Table 2 – Amount of 0.01% NaOH added to Straight Vegetable Oil (SVO)

Ending amount of 0.01% NaOH solution in burette	**________ ML**
Starting amount of 0.01% NaOH solution in burette	**________ mL**
Total amount of 0.01% NaOH added to the beaker	**________ mL**

PART II – Determine the amount of Free Fatty Acids in Waste Vegetable Oil (WVO)

- ☐ 10. Pour 10 mL of isopropyl alcohol into a graduated cylinder. Add the isopropyl alcohol to the small glass beaker labeled “WVO.”
- ☐ 11. Add 1000 μL of the waste vegetable oil sample from the tube labeled “WVO” to the small glass beaker labeled “WVO” containing the isopropyl alcohol. **Describe the appearance of the liquid in the beaker.**
- ☐ 12. Gently swirl the contents of the beaker for 10 seconds. **Describe the appearance of the liquid in the beaker.**
- ☐ 13. Add 50 μL of the indicator solution (turmeric) to the oil/isopropyl alcohol mixture in the “WVO” beaker. Gently swirl the contents of the beaker for 10 seconds. **Describe the appearance of the liquid in the beaker.**
- ☐ 14. Read and record the amount of sodium hydroxide (NaOH) in the burette. Be sure to be precise in your measurement and record the data below:

  Starting amount of 0.01% NaOH solution in burette _______ mL

- ☐ 15. Carefully position the burette directly over the WVO beaker. Carefully add one (1) drop of the NaOH solution from the burette into the beaker by turning the stopcock clockwise. **DO THIS SLOWLY and CAREFULLY. Only add ONE DROP at a time.**
- ☐ 16. Swirl the beaker. Record the number of drops and any color changes in **Table 3** immediately after adding the NaOH to the beaker.

Table 1 – Number of 0.01% NaOH added to Straight Vegetable Oil (SVO)

Number of Drops	Color after swirling	Did the color change remain for 15 seconds? Yes or No?

- ☐ 17. Repeat steps 15 and 16 until the solution turns **bright pink** and **remains** that color for at least 15 seconds.
- ☐ 18. Now that your solution is bright pink, record the remaining amount of NaOH in the burette in Table 4 below:

Table 4 – Amount of 0.01% NaOH added to Waste Vegetable Oil (WVO)	
Ending amount of 0.01% NaOH solution in burette	**_______ mL**
Starting amount of 0.01% NaOH solution in burette	**_______ mL**
Total amount of 0.01% NaOH added to the beaker	**_______ mL**

PART III – Determine the total amount of NaOH needed to make biodiesel from your sample

Transesterification of virgin vegetable oil (not used for cooking) requires 4.0 grams of NaOH for every liter of oil. However, since your samples were WVO, not straight oil, we know they contained a certain amount of additional free fatty acids. The acid value you calculated for WVO allows us to determine how much additional NaOH we will need in our reaction to neutralize the free fatty acids prior to the transesterification process.

Therefore, to get the *total amount* needed, you will need to add the amount of NaOH needed to neutralize the amount of free fatty acids in your sample (the Acid Value number) to the amount of NaOH needed for the transesterification process (4.0 g NaOH/liter virgin oil).

Total amount of NaOH that will be needed to carry out the neutralization of free fatty acids and the transesterification process:

4.0 g/L	+	____________	=	_______ G NaOH/Liter of oil
(Constant value)		**(Total amount NaOH added to the WVO from number 18)**		**(Amount to add to make biodiesel)**

Review Questions

1. Was your hypothesis supported by your results?

2. Why did you include the sample of pure vegetable oil in your experiment?

3. Is NaOH a basic or acidic substance? Is the pH value low or high?

4. Why did you add the indicator solution (turmeric) to the oil/isopropyl alcohol mixture? Why did it turn pink?

5. Which sample required more of the NaOH solution to cause the color to change to pink? Why?

6. What is the next step in the process of making the biodiesel fuel?