Introduction

The process of finding a suitable topic for my chemistry IA was long and tedious. After going through multiple phases of ideas and ruling out each one due to either unpracticality (determine the water of crystallization by titration), time constraints (electrolysis of copper sulphate) or lack of an independent variable (copper cycle). I realized that all my ideas were mostly based on an interest in green chemistry which led me to do further research in that specific area and come across the synthesis of biodiesel in *Green Chemistry Experiments in Undergraduate Laboratories* by Jodie T. Fahey and Lynn E. Maelia. This topic of this IA is the transesterification of vegetable oil and alcohol to produce ethyl esters

Background

Biodiesel is made through the process called transesterification, where 1 mole of triglycerides (organic oil/fats) reacts with three moles of alcohol to produce three moles of alkyl esters (biodiesel) and one mole of glycerol (co-product). Triglyceride is a molecule that is made up of three fatty acids attached to a glycerol backbone (biodieseleducation).

Usually, the minimum of a 3:1 molar ratio of alcohol to oil/fat is required to produce biofuel but a 6:1 ratio is better as it forces the reaction further to the product side (biodieseleducation.org) of the equilibrium. Multiple experiments can be done to increase the purity of the biodiesel by pushing the equilibrium, but one should be enough for this IA. The extra alcohol can then be recovered from the final solution through distillation. The catalyst can also be recovered from the glycerol layer. This IA will include none of these additional procedures to purify the crude diesel due to time constraints.

For the creation of my procedure, I took parts from two lab manuals I came across online. One from an MIT chemical engineering professor (Prather) and another from the National Biodiesel Education Program (biodieseleducation). I combined parts from both manuals to create my procedure with modifications from my beta tests. During beta testing, I initially put a stopper over the flask as suggested by the lab guide from the Massachusetts Institute of Technology, but the pressure built up by the oil kept pushing the stopper off, so I decided to remove that from the lab procedure. I also found there to be no effect on preheating the canola oil before adding the ethanol solution compared to adding it before heating. Thus, I chose to keep things consistent

with no prior heating on any of the trials. Lastly, I neglected to completely dissolve the sodium hydroxide and it ended up creating a semi-solid blob of oil that had the same properties as soap. I was careful to not repeat these mistakes during my actual experiments.

Research Question

What ratio of triglycerides to alcohol creates a reaction that is the most efficient for creating biodiesel with minimal co-products or unreacted reactants?

Hypothesis

I hypothesise that the efficiency of the reaction will be quite low at first until the 1:6 ratio of canola oil to ethanol, at which there may be a larger increase. The increments would then gradually decrease till 1:12 (the upper limit of this experiment). This is because the University of Idaho recommends a 1:6 ratio to be efficient with time and push the equilibrium to the reactants side whereas you only need a 1:3 ratio according to stoichiometric. Below are the general reaction equations (msjchem).

Triglyceride + Ethanol ⇌ Ethyl Ester + Glycerol

RCOOR1 + 3 R2OH ⇌ R1OH + RCOOR2

Variables

Table 1: List of variables

Type	*Variable*	*Method and reason for control if needed*
Independent	Volume of anhydrous ethanol solution with dissolved NaOH	The 5 variations of this experiment would be the volume of the reagent solution. 2.6 mL, 8 mL, 12 mL, 16 mL, 24 mL
Dependant	Volume of crude biofuel	After each trial, the recorded data point is the amount of synthesised crude biofuel
Control	Temperature	All the trials used the same model of hotplate with

		the setting set to 2. Through beta testing, I found this to be approximately 80℃. An increase in temperature will also cause an increase in the rate of reaction.
	Method of heating	As stated in the B*ackground* regarding my beta tests, all trials were carried out without any prior heating done to the canola oil. Variances in starting temperature will influence the rate of reaction.
	Reagent solution	The solution was prepared at the start of the lab with more than enough to run through all 15 trials. The concentration of catalyst would vary if the solution was not preprepared resulting in varying rates of reaction.
	Type of reagent oil	All the vegetable oil used in this experiment came from the same 950mL bottle of canola oil. Different vegetable oils have different fatty acid structures which may influence the reaction.
	Volume of canola oil	Each trial would use exactly 50.0 ± 0.5 mL of oil. There may only be on independent variable.
	Catalyst	NaOH is the only catalyst used (instead of the more expensive and caustic KOH). Different catalysts may have an effect on reaction rate.
	Alcohol	Ethanol is the only alcohol used (instead of the more expensive and toxic ethanol). Different reagents may have an effect on reaction rate.
	Length and mixing (agitation) of each reaction	Each trial lasted 20 minutes on the hotplate with a 10 second swirl given every minute. More mixing would increase the reaction rate.
	Method of complete(ish)	The separation process would be halted after 5

	separation of the products	minutes of no visible changes to ensure that all trials would be separated to the same extent
	Size of reaction flasks	All trials took place in a 125.0 mL Erlenmeyer flask to prevent discrepancies during the reaction. A larger or smaller flask may cause different reaction rates.
	Size of graduated cylinders	All measurements of the ethanol solution were done in a 25.0 ± 0.1 mL cylinder to keep uncertainties consistent.
	Concentration and volume of neutralizing HCl	All trials had 10.0 ± 0.1 mL of 6.0 M HCl added after the reaction to neutralize and separate any potential unreacted ethanol from the biodiesel.

Materials

Equipment

Table 2: List of equipment

Name	*Usage*	*Quantity*
250.0 mL Erlenmeyer flask	Holds the ethanol solution	1
125.0 mL Erlenmeyer flasks	Contains the main reaction	5
Size 5 rubber stoppers	Seal the 125 mL flasks to leave overnight	5
Size 6 rubber stopper	Seal the 250 mL flask when not in use	1
100.0 mL graduated cylinders	Measures volume of canola oil and synthesised crude biofuel	2
25.0 mL graduated cylinders	Measures ethanol solution to be used in a trial and the neutralizing HCl	2
100.0 mL beakers	Gathers the products after separation	2
Separatory funnel	Separates crude biofuel from other products	2
Funnel	Assists in the transferring of liquids	3
Stopwatch	Controls the length of each trial	1

Pair of heat resistant gloves	Assists in the swirling of the reaction	1

Reagents/Catalysts

Name	*Quantity*
Canola oil	950 mL
Anhydrous ethanol	200.0 mL
Sodium hydroxide	7.0 g
6.0 M HCl	150.0 mL

Procedure

1. Mix 200mL anhydrous ethanol with 7.0g of sodium hydroxide
    a. Optionally put over hotplate to speed up the dissolving as this step may take a few hours and it is very important that all the NaOH dissolves
2. Measure 50mL of canola oil in a graduated cylinder and pour into a dry and clean 125mL Erlenmeyer flask.
3. Measure out 2.6 mL of the ethanol solution and pour into the flask with oil
4. Place flask on a hotplate with the temperature set to 2 (out of 10)

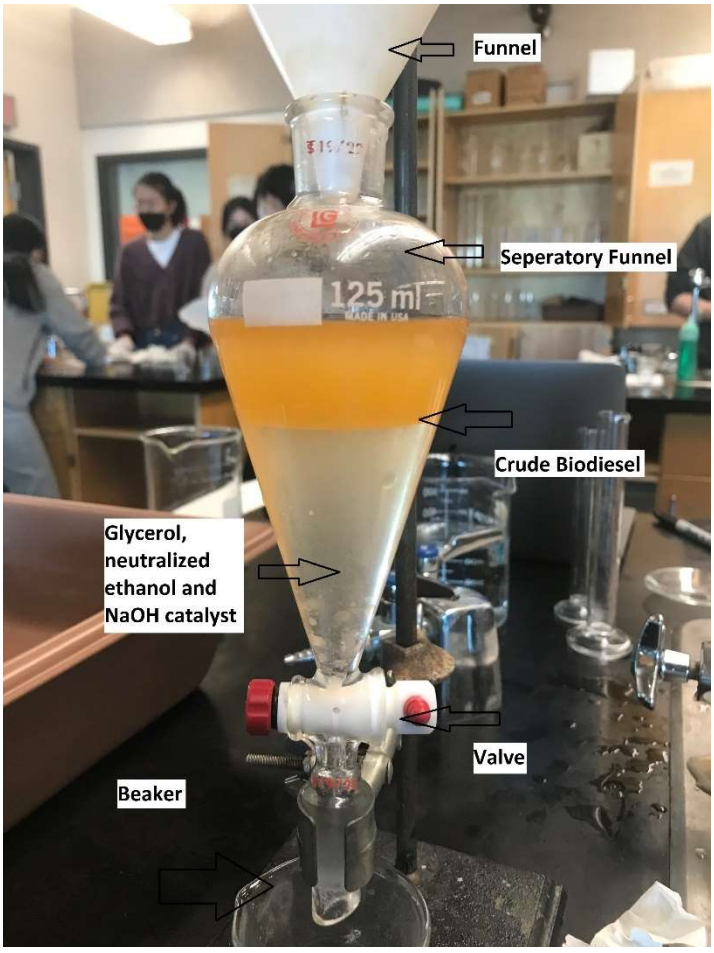

Figure 1: Products after a few minutes in a separatory funnel

5. Every minute, swirl the mixture by grabbing the neck of the flask
    a. Optionally, one could be wearing heat resistant gloves

6. After 20 minutes, take the flask off the hotplate and let cool
7. Repeat steps 2-6 with different amounts of ethanol solution
    a. This could be done in conjunction with up to 3 or 4 experiments (depending on how many can fit on the hotplate)
8. After the flask has cooled to room temperature, add 10 mL of HCl to neutralize the unreacted ethanol and glycerol and there should be two distinct layers
9. Put the solution in a separatory funnel and give the solution to separate
    a. Stop the separation after 5 minutes of any visual changes
    b. See figure 1
10. Repeat steps 8 and 9 until the data table is filled

Safety

Table 3: List of potential safety hazards

Type	*Hazard*	*Measures*
Physical	Hot oil	Safety goggles worn at all times over eyes, flasks put on a hotplate to cool, volume used in each trial kept small to prevent dangers
	Glassware	In the event of an accident, make sure to clean up all shards of glass with a dustpan and dispose of them in the designated waste bin for broken glass
Chemical	NaOH	Do not handle with bare hands and instead use weigh boats to measure the desired volume
	Ethanol	Use funnels to prevent spillage when measuring
	Product disposal	The by-products of glycerol and neutralized ethanol are to be diluted and disposed of down the sink
	Unused reagent disposal	The small quantity of unused ethanol solution is to be disposed of in the inorganic waste bin

Data & Observations

Table 4: Raw data from the experiment

Ethanol solution volume (± 0.1mL)	*Trial 1 (± 1mL)**	*Trial 2 (± 1mL)*	*Trial 3 (± 1mL)*	*Qualitative Observations*
2.6 ethanol	Soap	Soap	Soap	The most foaming when swirled that dissipated fast at the start of the reaction but grew slower as the trial went on
8.0 ethanol	45.0 mL	45.5 mL	46.5 mL	A bit of foam that always dissipated in the light-yellow mixture
12.0 ethanol	53.0 mL	50.0 mL	51.5 mL	Mixture had even less foam
16.0 ethanol	53.5 mL	56.0 mL	54.0 mL	Mixture grew darker in colour
24.0 ethanol	55.0 mL	54.5 mL	55.0 mL	Darkest shade of yellow with minimal foaming

*See *Calculating Uncertainties for a sample calculation on uncertainty*

Data Analysis

Calculating Uncertainties

Table 5: Sources of uncertainty

Source of uncertainty	*Degree*
Measuring canola oil in a 100.0 mL graduated cylinder	50.0 ± 0.5 mL
Measuring ethanol solution in 25.0 mL graduated cylinder	± 0.1 mL
Measuring final crude biodiesel product in 100.0 mL graduated cylinder	± 0.5 mL

Sample uncertainty calculation for second 12.0 mL trial

$$(50.0 + 0.5mL) + (16.0 \pm 0.1mL) + (50.0 \pm 0.5mL) = 50.0 \pm (0.5 + 0.1 + 0.5)$$
$$\approx 50.0 \pm 1mL$$

Calculating Standard deviation

Sample standard deviation calculation for 12.0 mL trials

$$51.5mL - 53.0mL = 1.5mL;\ 51.5mL - 50.0mL = 1.5mL$$

$$51.5ml - 51.5ml = 0mL$$

$$(1.5mL)^2 + (1.5mL)^2 + (0mL)^2 = 4.5mL$$

$$\sqrt{\frac{4.5mL}{3}} = 1.22474mL = 1.22mL$$

Calculating Atom Economy

$$\%\ Atom\ Economy = \frac{Molar\ Mass\ of\ Product}{Molor\ Mass\ of\ All\ Reactants} \times 100\%$$

Substance	*Molar mass*	*Source*
Canola Oil	876.60 g/mol	chem.wilkes.edu
Ethanol	46.07 g/mol	Periodic table
Biodiesel	292.12 g/mol	Biodieseleducation.org

Sample atom economy calculation for 12.0 mL trials

$$\frac{\frac{292.12g}{mol} \times 51.5g}{\left(\frac{876.60g}{mol} \times 50.0ml\right) + \left(\frac{46.07g}{mol} \times 12.0g\right)} \times 100\% = 33.9\%$$

Experiment Analysis

Table 6: Analyzed data

*Volume of ethanol**	*Moles of oil to moles of ethanol*	*Average volume of crude biodiesel*	*Atom economy of average volumes*	*Standard deviation*
2.6 mL	1:1	n/a	n/a	n/a
8.0 mL	1:3	45.7 ± 1 mL**	30.2%	0.76 mL
12.0 mL	1:4.5	51.5 ± 1 mL	33.9%	1.22 mL
16.0 mL	1:6	54.5 ± 1 mL	35.7%	1.32 mL
24.0 mL	1:9	54.8 ± 1 mL	35.6%	0.29 mL

*Dissolving NaOH does not have any significant effect on the volume of ethanol in this experiment

Graph of Results

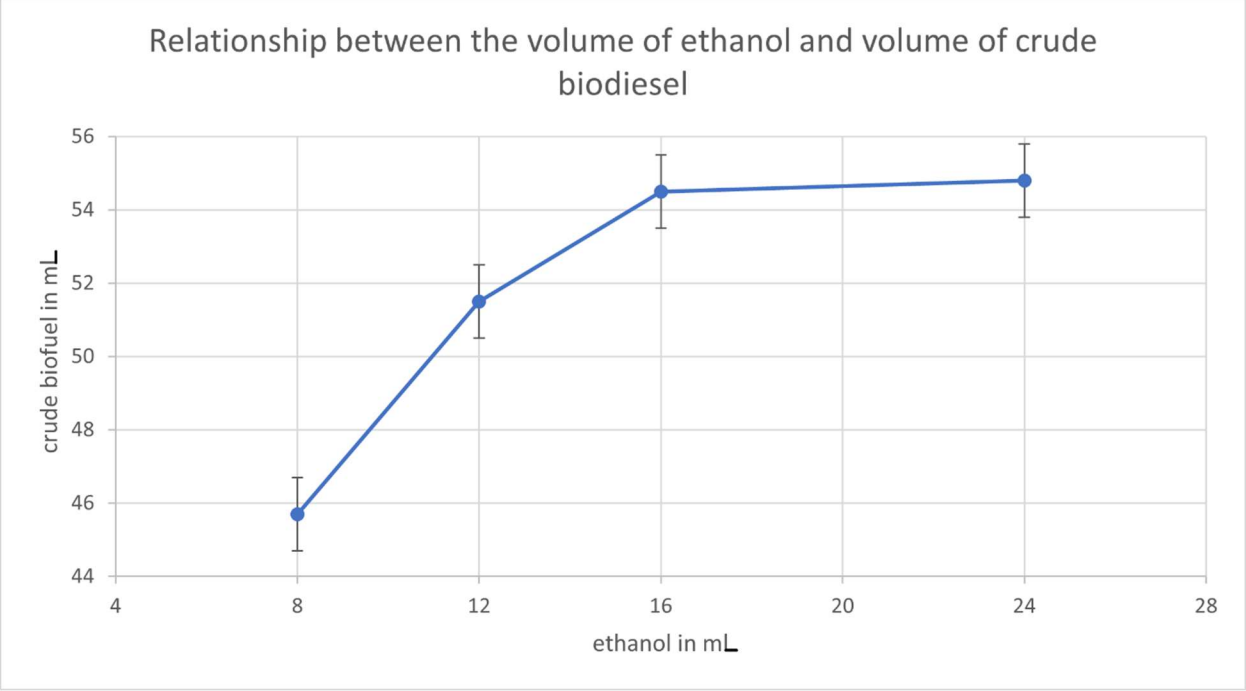

Figure 2: Graph of the main data

The graph above shows a clear logarithmic relationship between the volume of ethanol and synthesized crude biodiesel.

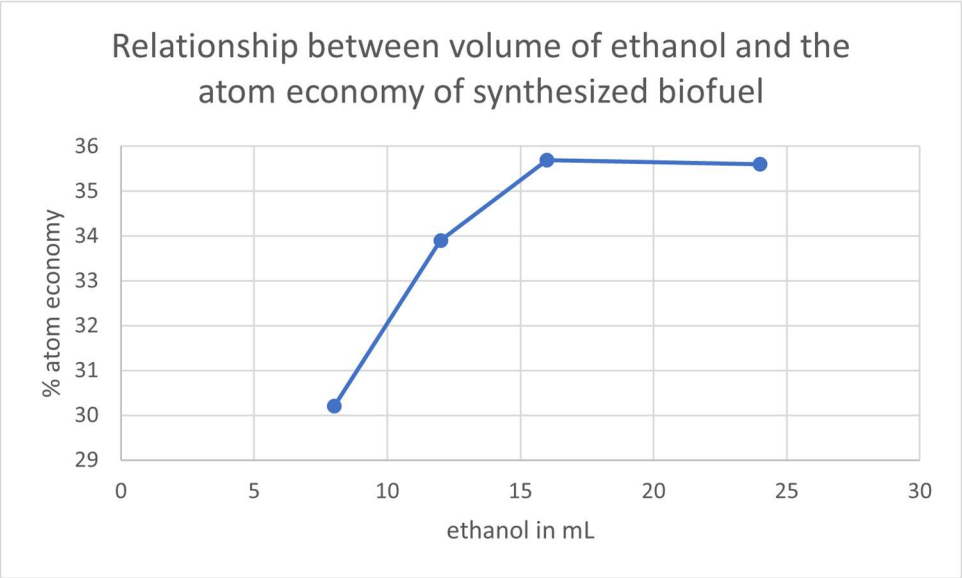

Figure 3: Graph of atom economy

The graph above shows a similar trend to the previous one except for the crucial difference that the last data point is not the highest.

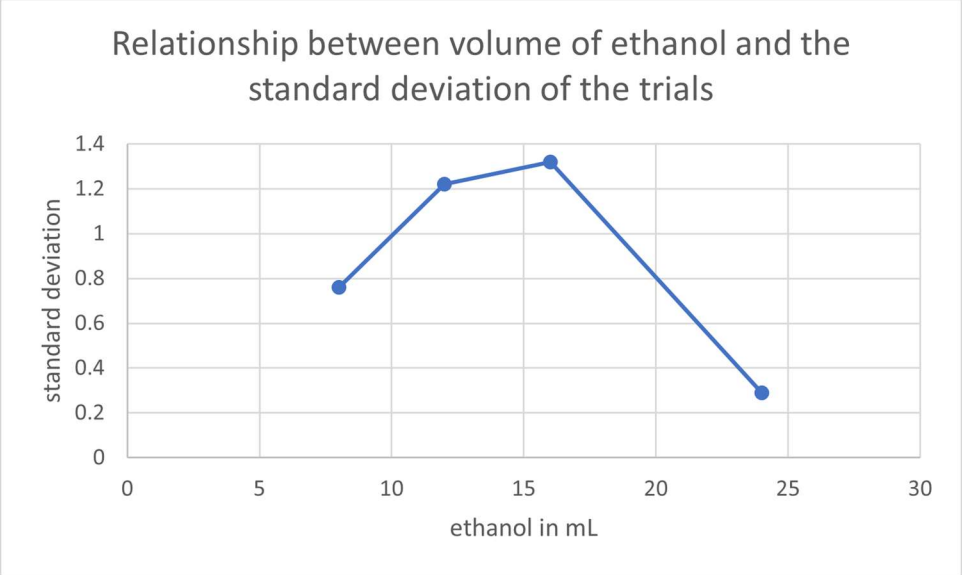

Figure 4: Graph of standard deviation

This graph shows the relationship between the average volume of crude biofuel produced in three trials and the standard deviation between the values.

Conclusion

To answer the research question of "what ratio of triglycerides to alcohol creates a reaction" that is the most efficient for creating biodiesel with minimal co-products or unreacted reactants?". The graphed data in figure 3 has shown that a ratio of 1 to 6 of canola oil and ethanol to be the most efficient in synthesizing biodiesel. This answer would likely hold up true for even larger ratios of ethanol to canola oil as there was a very minimal change in the quantity of synthetized biodiesel in regards to 16 mL (1:6) and 24 mL (1:9) volumes of ethanol as can be seen in figure 2. 16 mL of ethanol produced an average of 54.5 mL of biofuel over the course of three trials whereas 24 mL of ethanol produced an average of 54.8 mL. A shoddy 0.6% increase in yield with over 50% in ethanol. The 50% increase in ethanol for this reaction is not fully reflected in atom economy due to the large difference in molar mass between canola oil (876.60 g/mol) and ethanol (46.07 g/mol). This difference makes it hard to see the wastage of ethanol in atom economy as can be seen in figure 3, a 50% increase in ethanol usage with pretty much the same yield in biofuel only has the effect of 0.1% (35.7%-35.6%) on total atom economy.

The other ratios of 1:3 and 1:4.5 are not even considered as their atom economy is proportionally much lower than 1:6 and 1:9 as can be seen in figure 3. When it comes to something such as synthesizing biofuel, every single percent of atom economy matters as these operations are usually taken to a very large scale commercially and the small differences are greatly magnified. It's also important to be efficient from both an environmental and economic perspective.

The data in graph 4 is not the primary focus of my IA. Yet, I still found the data to hold value. Even though there is no literary value to use as a means of comparison, one can notice that there is a steady upward trend that suddenly drops when it comes to data coming from biodiesel synthesized from 24 mL of ethanol. This signifies that the increase from 16 to 24 mL of ethanol made the reaction far more consistent with fewer degrees of variation.

Evaluation

One of the largest strengths of this IA would be the extensive list of control variables as an attempt to limit the rate of reaction from being affected by external factors. This in turn

allowed the experiment to produce reliable and consistent data that was straightforward to analyze.

However, there is also a fair share of limitations present. Step 5 requires a human to manually swirl the reaction to speed it up. However, in all the lab guides I have read, a stirring hotplate (which my facilities lacked) is highly recommended for both a faster and more thorough reaction. Additionally, in step 9 of the procedure, one must use a separatory funnel. It would be the most optimal to let the product sit for hours or even days for complete separation of biodiesel. However, due to time constraints, there are small amounts of glycerol and ethanol in each of the final samples. Hence, why I referred to the final product as crude biodiesel throughout the IA. The last major limitation would be in my analysis where I used the molar mass of purified biodiesel. As there is no way to find the actual molar mass of the synthesized crude biodiesel, that number was the best option I had. In a perfect world, I would get all the biodiesel samples to as high of a purity I could before measuring and doing calculations. However, this again is a time intensive activity that I could not afford during this IA.

An extension to my investigation could lay in either purifying the crude biofuel or testing the synthesized biofuel in an engine to measure how much power is produced. Both are focused on real world application of the conclusions reached from the data gathered from this experiment. I was initially planning on testing the synthesized crude biofuel in an engine to conclude my IA. However, due to time and equipment constraints, I was unable to accomplish this.

Bibliography

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