Project # 4: "Clean Diesel Technologies for Air Pollutant Reduction Measurement of Air Pollutant Emissions from Biodiesel Blends" (Biodiesel Project)

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Goals and Objectives

Due to the global scarcity of fossil fuels and the associated rising costs, the necessity of developing economically feasible alternative fuels is increasing with daily importance. Perhaps this energy dilemma will be solved not by finding a "perfect" energy source, but by finding many alternatives that can be used concurrently. One obvious benefit of using waste frying oil as an alternative fuel source is that the feedstock is already there. No food sources will be lost for human consumption thereby driving up the price by decreasing supply. In fact, prior to reclaiming this waste oil for biodiesel production purposes, most restaurants had to pay a company to come and pick up their used oil. Today, many restaurants are being paid for their used frying oil, or at least given a credit to be used toward their next purchase of new frying oil.

This research project focused on optimizing the amount of catalyst used in the transesterification process of waste frying oil into biodiesel fuel. The main byproduct of this process is glycerol. If too much catalyst is used, it increases the cost of the process, but if not enough is used then all of the potential biodiesel fuel won't be converted.

Equipment, Methods and Experimental Procedures

To begin the research, the participants first had to procure a sample of waste frying oil. After several attempts, a source was located that was willing to give some oil. The Graduate Student Mentor explained the transesterification process to the teachers and also gave them detailed instructions on two different methods of performing a titration on the sample (as shown in **Figure 1**). Since every batch of waste frying oil is different, a titration must be performed prior to its conversion. One method of titration helped them to identify how much free fatty acid was present in the waste frying oil by using phenolphthalein as an indicator and adding drops of sodium hydroxide until the solution turned pink. The teachers then used math to figure the percentage of free fatty acid that was present in the solution. The amount of catalyst is determined by this process. The other method of titration was to make test samples with varying amounts of the catalyst. For this, the teachers used an alkaline catalyst, sodium hydroxide (NaOH)), which was dissolved in methyl alcohol (MeOH), as shown in **Figure 2**.

At the beginning of the research, the participants did not have an idea how important the percentage of free fatty acid was to the transesterification process, but they soon learnt it. They made several attempts to convert this oil to methyl esters and glycerol, but were unsuccessful. The mentors discussed with them why their transesterification process didn't work and asked the teachers to go back and look at the percentage of free fatty acids present in their sample. It was a large percentage, over 4.5%. Free fatty acids are formed from heating the oil over and over. So it was decided to locate a different source of oil.

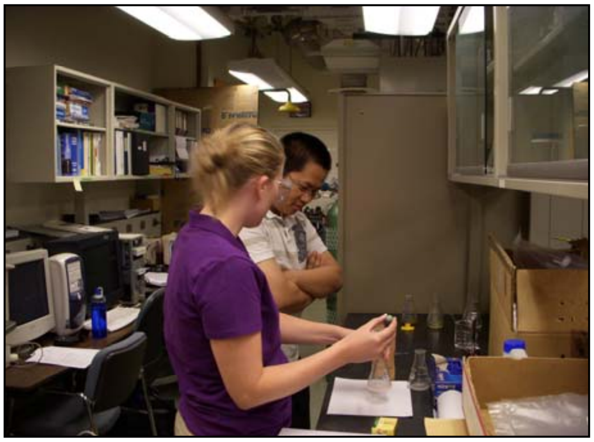

Figure 1. Teacher Performing a Titration

Figure 2. Teacher Dissolving NaOH in Methyl Alcohol

After obtaining different waste frying oil, the participants performed the titrations on this new sample. This sample had a much lower amount of free fatty acid as indicated by the titrations, around 0.6%. The participants made three small test batches, varying the amount of sodium hydroxide (NaOH) for each. All three samples reacted successfully, producing biodiesel. They measured the amount of glycerol produced with each amount of catalyst and chose the quantity that yielded the most glycerol, indicating the most complete reaction. The smallest amount of catalyst didn't cause a complete reaction as indicated by the amount of glycerol, and the largest amount of catalyst produced a solid mass of byproduct instead of glycerol. They scaled the successful transesterification amounts to produce ten gallons of biodiesel fuel. After washing their biodiesel fuel several times to more fully remove any un-reacted methanol, glycerol, and other impurities, they performed a fuel analysis.

To ensure a quality product, each batch of biodiesel fuel produced needs to be analyzed. After training on how to use the gas chromatography-mass spectrometry machine, the participants analyzed their samples. They were very happy to discover that their biodiesel closely modeled the standard.

The high percent free fatty acid oil still presented a problem. Research ensued to locate information regarding transesterification methods of oil with high free fatty acid percentages. Two methods were found. The first involved an acid pre-treatment (sulfuric acid) followed by transesterification using NaOH dissolved in MeOH. Another method utilized only sulfuric acid as a catalyst. The research team decided to investigate each method and to compare the results.

The main drawback with using an acid to perform the transesterification is there is a longer reaction time. Sulfuric acid is the acid used as the catalyst in both the acid prewash and the pure acid methods. The acid pretreatment method worked nicely but it did take a bit longer and the pretreatment added an additional step in the process. When the participants analyzed the biodiesel fuel using the gas chromatography-mass spectrometry machine, results showed that their process yielded a quality consistent with the standard sample that was given to them.

In order to perform the sulfuric acid catalyst transesterification, a minimum of fifty hours of reaction time was necessary. During these fifty hours, the mixture needed to be heated and stirred continuously. The participants were given instructions on how to use a hot plate with an electric stirring mechanism, as shown in **Figure 3**. They also had to use a cooling tube with water continuously flowing through it so that we could stop the methyl alcohol from evaporating. Samples were taken at regular intervals. After the fifty hours they analyzed, (see **Figure 4**) their biodiesel fuel and again compared it to the standard sample.

Figure 3. Preparing the Five Neck Flask for 50 Hour Reaction

Figure 4. Taking a Sample of the Acid Catalyzed Reaction

Highlights of the Research Findings

The participants successfully identified a process to produce biodiesel with desired properties from waste frying oil. The research is continuing. When the results from all three methods of transesterification are completed, an attempt to come up with a recommendation for which type of method to use will be made based on the amount of free fatty acid present in the sample waste frying oil.

Plans for Classroom Implementation

A unit of instruction was developed for implementation during the upcoming school year. One is for middle grades science and one is for high school math, namely, Advanced Placement Statistics.

At the middle school science level, the focus will be on oil dependence and the use and development of alternative fuel sources. The students will be actively engaged throughout the unit. Students will be divided into teams and each team will choose an alternative fuel source. The teams will represent companies that produce and market that fuel. Each team will research their energy source and develop a poster that highlights the pros and cons of their fuel, including cost and availability. A team of teachers will act as "congress" and will grade the presentations. The poster session will include a debate, with each team given ten minutes to present their case.

At the high school level, collaboration will occur with a science class so that students can perform the transesterification in small 100 mL batches. AP Statistics focuses on analyzing data and communicating the findings. Students will be engaged in dialogue concerning the importance of finding alternative energy sources and their personal use of fuel. Many of them drive and should have valuable input including costs incurred by them and the need to find renewable energy sources without increasing the cost. Data will be gathered in the science classroom and analyzed in the statistics class. Due to the amount of time it takes to perform a titration the students will investigate if titrations need to be performed for each batch of oil received from a chain of restaurants. Students will collect samples from restaurants over time and analyze the samples for the amount of free fatty acid. The students will also conduct an experiment to determine if amounts of free fatty acid within samples of oil from chains of restaurants are statistically different. Then they will determine if a difference exists between the chains of restaurants. It is up to the students to determine what kind of hypothesis test to perform and to check for assumptions to verify that they are performing the correct test in each case. Findings will be presented to the class in the form of a PowerPoint presentation and a written report will be turned in including a recommendation backed up with mathematical support.