Continuous Biodiesel Production II: Purification Via Silica Gel Columns

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Abstract

Many different processes have been investigated to produce biodiesel from various triglyceride feedstocks. Both batch reactions and continuous flow processes have been developed in an effort to lower the production costs of biodiesel to make it competitive with diesel fuel derived from crude oil. All production methods currently produce a mixture of biodiesel, catalyst and unreacted oil feed stock that must be purified before the fuel can be distributed for use. We report the results of a project designed to develop a continuous production cycle for biodiesel. This work focuses specifically on the purification of the mixture as it exits the trans-esterification process. Current methods rely on a density-driven process in large settling tanks. While effective, this stop in the biodiesel production process adds significant time as the mixture separates, tanks are emptied and cleaned before a new batch can be processed. Purification in this work is accomplished by forcing the mixture through a silica-gel column via a pressure differential. Column height and diameter, flow rates and pressure differentials are varied in order to achieve continuous purification as the mixture exits the trans-esterification process.

Introduction

Biodiesel is a fuel composed of mono-alkyl esters of long chain fatty acids derived from vegetable oils or animals fats, designated B100, and meeting the requirements of the American Society for testing and Materials (1). Store-bought vegetable oil and waste vegetable oil obtained from a local restaurant were used to synthesize biodiesel at a medium level in UNC-Pembroke's biochemistry laboratory. The fundamental goal is to learn to make biodiesel and the ideal conditions for an appreciable yield. To determine if biodiesel was in fact synthesized, the Gas Chromatography-Mass Spectrometry (GC-MS) instrument and bomb calorimetry were utilized.

Materials and Methods

Materials

To convert vegetable oil into biodegradable diesel, we used the following components: vegetable oil, methanol, and potassium hydroxide. The amounts of these components were subject to change with each trial as we worked to improve the maximum yield of biodiesel. To convert the previously stated components into biodiesel, we used the following equipment: round bottom flasks, funnel, sonicator, separatory funnel, NMR, GC-Mass Spec, Buchner Funnel, silica gel, Luer Lock syringe, test tubes, Peristaltic pump, and IR analyzation. As we worked towards increasing our yield, the equipment used, and its efficiency increased as we researched further into our method.

Methods

Purification

- Wash silica gel with ethyl acetate
- Dry the gel in the oven at 40 degrees Celsius for a few minutes
- Pack the gel into the column (length: 6.8 cm, diameter: 1 cm)
- Pass the reacted solution through the column with the help of the pump for the separation of glycerol, sodium salt of fatty acid, methanol, from biodiesel and unconverted oil.
- The biodiesel was collected and analyzed for the conversion using benchtop NMR (nananalysis).

Apparatus

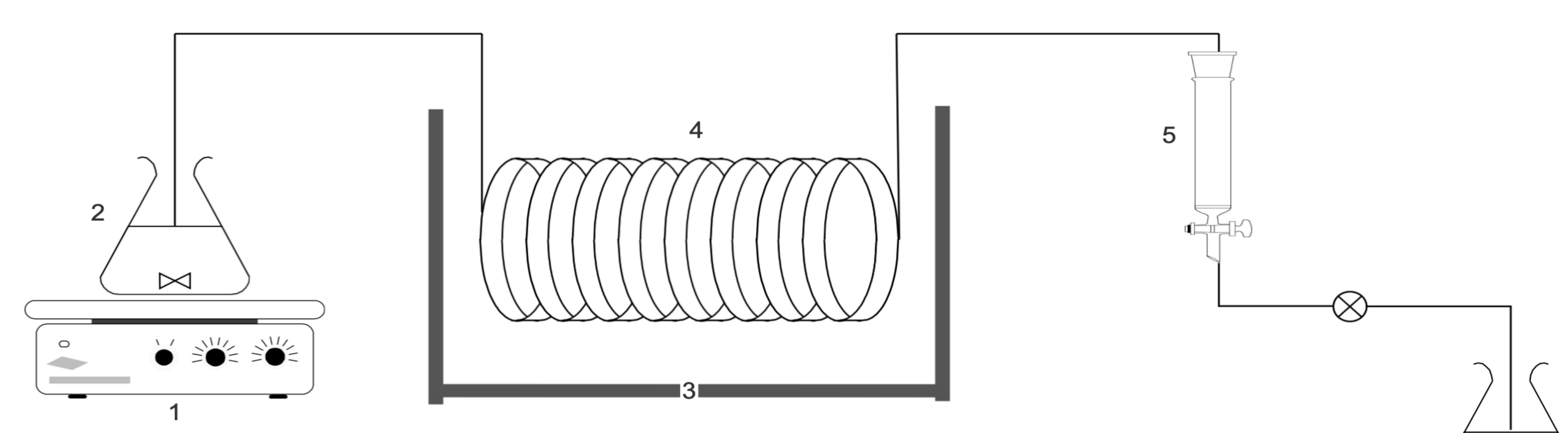
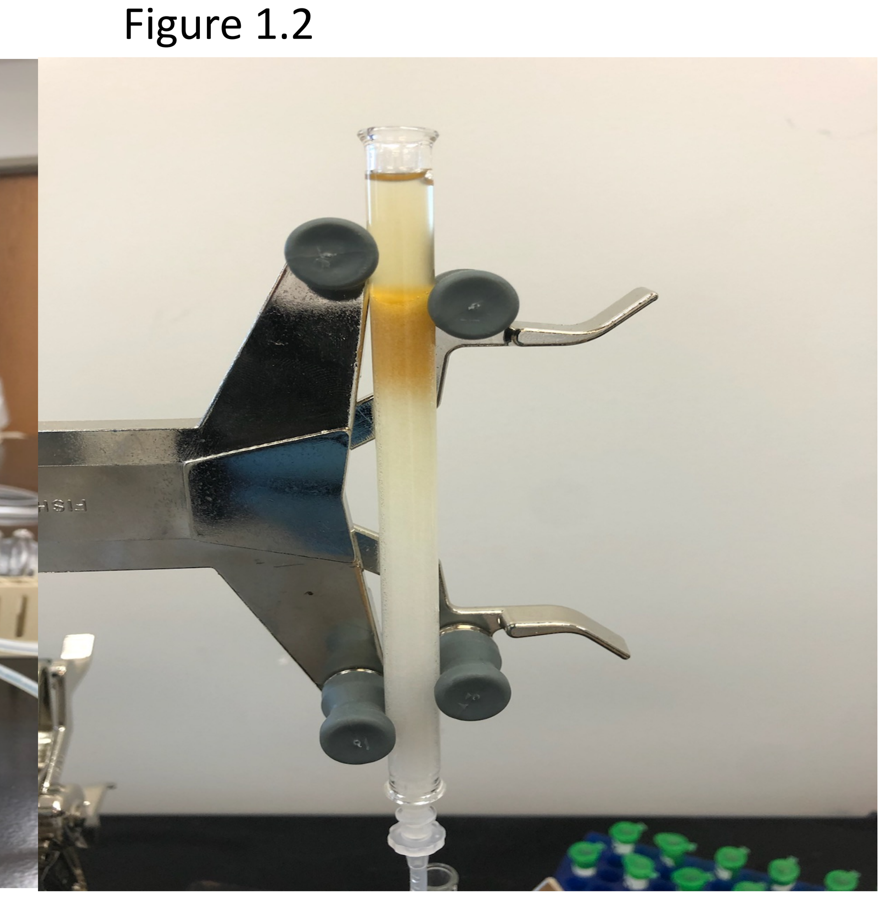


Fig.1- schematic diagram of continuous process of vegetable oil to biodiesel

1- agitator, 2- reaction vessel, 3- sonicator, 4-tubular rector, 5- separation column, 6- collection vessel

Figure 1.1

Figure 1.2



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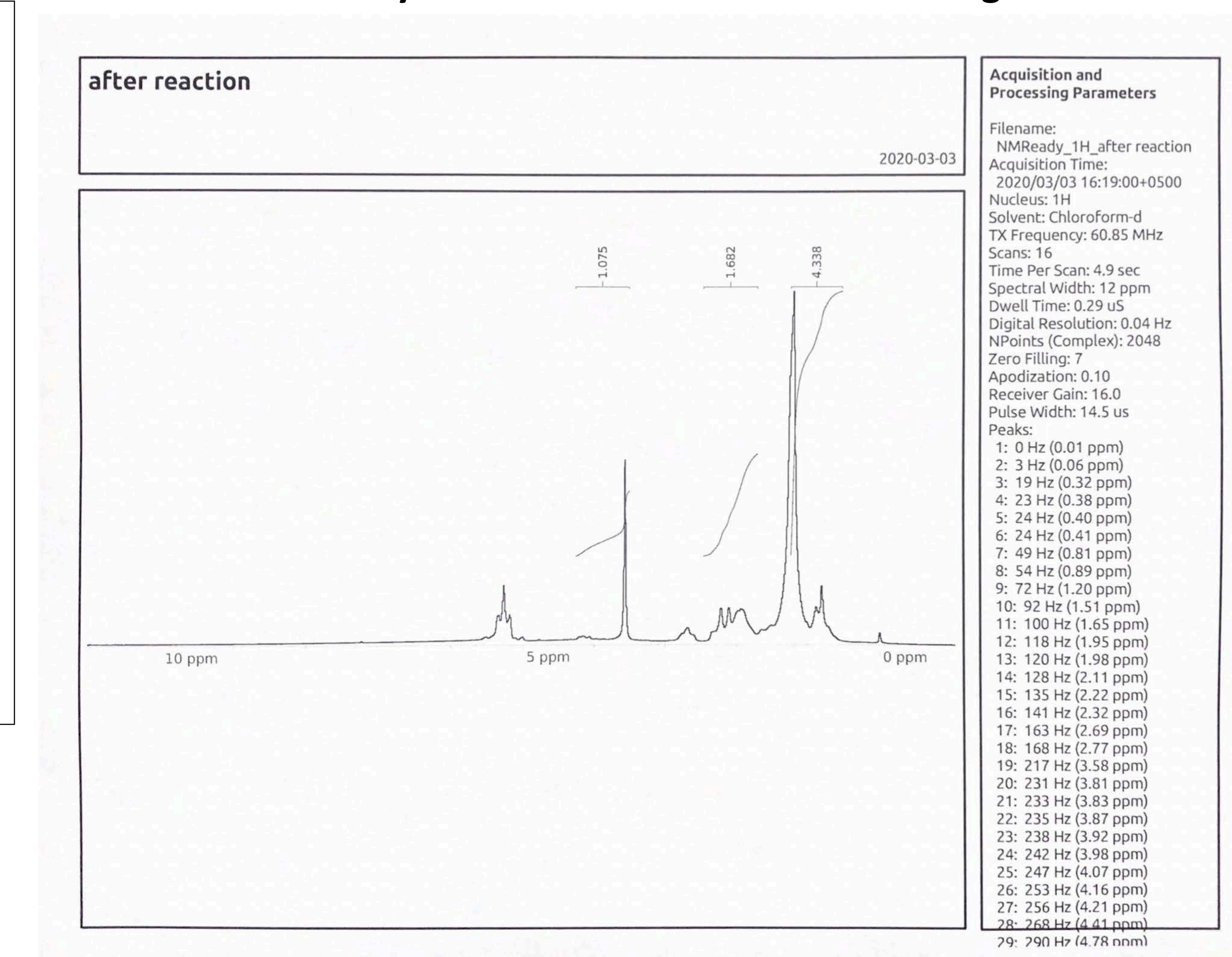
Results: NMR Analysis of Biodiesel After Reaction- Figure 2



Discussion

Through this experiment it was determined that Silica gel, in the stationary phase, needed to be washed with ethyl acetate to remove all polar molecules for the regeneration of the column. In this case, polar molecules are methanol, potassium salt of free fatty acid, glycerol, and hydroxide ions. The nonpolar molecules, biodiesel and unconverted oil, were passed through the column without any interaction. This separation was achieved continuously using a pump at a flow rate of 12 mL/min. The column was loaded with approximately 3.25 grams of silica gel. Observing Figure 1.2, it shows that there is a clear separation of glycerol and other polar molecules in the column. This column can handle a maximum of 10 mL at a time. The resulting solution was analyzed using benchtop NMR (nananalysis), as shown in Figure 2. The data looks very encouraging to proceed in the future by using a larger column with an efficient pump.

Acknowledgements

We would like to thank PURC for granting the travel funds allowing us to attend the ACS Conference. We would like to thank Drs. Siva Mandjiny and Steven Singletary for their guidance throughout this project. We would also like to thank the Dean, Dr. Jeff Frederick, for supporting this work, as well as the entire Department of Chemistry and Physics especially Ms. Carolyn Parsons, at the University of North Carolina at Pembroke.