

Final Technical Report

Project Title: Messiah College Biodiesel Fuel Generation Project

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Recipient: Messiah College

Project Location: One College Ave., Grantham, PA 17027

Project Period: June 1, 2008 – December 31, 2011

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Final Scientific and Technical Report

Project Title: Messiah College Biodiesel Fuel Generation

Award Number: DE-FG36-08GO88068

Recipient: Messiah College

Project Location: One College Avenue, Grantham, PA 17027

Project Period: June 1, 2018 to December 31, 2011

Date of Report: March 15, 2012

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I. Executive Summary

The world's dependence on petroleum-based fuels has led to a state in which global economies are significantly affected by the rise and fall of oil prices. As oil reserves continue to decrease and demand continues to increase, the impact of this issue will grow exponentially. Significant advances have been made in the research, development, testing and distribution of alternative fuels such as biodiesel and ethanol. These renewable fuels provide an alternative to petroleum and also the promise of a more sustainable future. However the success and growth of these renewable fuels has created considerable debate regarding the use of food crops to make fuel rather than to feed the world. The advances in the biodiesel industry over the last two-decade, has mostly focused on large-scale producers and little attention has been paid to the small-scale opportunities. The Messiah College Biodiesel Fuel Generation project was developed to investigate small-scale biodiesel fuel production as a recycling process and to verify the feasibility of producing high quality biodiesel at a small scale.

Biodiesel is an environmentally friendly alternative to petroleum-based diesel fuel for use in unmodified diesel engines or as a fuel oil substitute. Biodiesel is produced by a reaction that occurs when vegetable oils are combined with an alcohol and a catalyst. Its primary advantage is that it is an agriculturally based, non-toxic, biodegradable and renewable fuel. Biodiesel combustion produces less carbon monoxide, unburned hydrocarbons and particulate emissions than petroleum diesel fuel.

Although several large-scale biofuel production plants are in various stages of development in central Pennsylvania, processor and process research and development has not yet been accomplished to support ASTM D 6751 certifiable biodiesel production by entrepreneurs and institutions seeking to leverage the opportunities afforded by biodiesel for recycling and other small-scale applications. Small-scale production has become a viable commercial option, given the low cost of production equipment. However, equipment for process research and quality testing is expensive, and prevents many small-scale producers from improving or certifying their processes. Moreover, there are no local laboratories readily available to small-scale producers in central Pennsylvania that provides access to the test equipment and expertise needed for process optimization and quality assurance.

Small-scale biodiesel production is an approach in which the end user is also the producer and creates biodiesel fuel from locally available feedstock to be used as a supplement or replacement for diesel fuel or heating oil. The end user can be an individual, a small business, a municipality, a school district, a community, a university, or any other group or organization that uses petroleum based diesel fuels or heating oil and has access to a biodiesel feedstock. Generally the feedstock available to small-scale biodiesel producers is waste vegetable oil that is either created by the end user directly or the end user collects it from the community. In some cases small-scale producers use clean vegetable oil that is being produced from crops that have been grown and pressed. This approach will be discussed in more depths later in this report.

Many obvious and significant concerns arise when considering the concept of small-scale biodiesel production. Does the fuel produced meet the stringent requirements set by the commercial biodiesel industry? Is the process safe? How are small-scale producers collecting

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and transporting waste vegetable oil? How is waste from the biodiesel production process handled by small-scale producers?

These concerns and many others were the focus of the research performed in the Messiah College Biodiesel Fuel Generation project over the last three years. This project was a unique research program in which undergraduate engineering students at Messiah College set out to research the feasibility of small-biodiesel production for application on a campus of approximately 3000 students. This Department of Energy (DOE) funded research program developed out of almost a decade of small-scale biodiesel research and development work performed by students at Messiah College. Over the course of the last three years the research team focused on four key areas related to small-scale biodiesel production: Quality Testing and Assurance, Process and Processor Research, Process and Processor Development, and Community Education.

The objectives for the Messiah College Biodiesel Fuel Generation Project included the following:

1. Preparing a laboratory facility for the development and optimization of processors and processes, ASTM quality assurance, and performance testing of biodiesel fuels.
2. Developing scalable processor and process designs suitable for ASTM certifiable small-scale biodiesel production, with the goals of cost reduction and increased quality.
3. Conduct research into biodiesel process improvement and cost optimization using various biodiesel feedstocks and production ingredients.

II. Terms and Definitions

Acid Number- The acid number establishes the acidity of the petroleum product being tested. This term refers to test methods and equipment associated with ASTM Standard D 664 for the determination of acid number of Petroleum Products by Potentiometric Titration.

ASTM D 6751- The standard specification for biodiesel fuel blend feedstock (B100) for middle distillate fuels. This is the industry standard by which biodiesel quality is certified.

Biodiesel- A diesel fuel alternative created through the transesterification of fatty acids with an alcohol to produce a fatty acid methyl ester.

Catalyst – Refers to the agent used to facilitate the reaction between alcohol and fatty acids to produce fatty acid methyl esters or biodiesel. Unless otherwise noted, this refers to potassium hydroxide (KOH) as related to this project. Sodium hydroxide (NaOH) was also used at times and will be specifically referenced as necessary.

Clean Vegetable Oil- Filtered and refined vegetable oil prior to cooking use or other applications.

Cloud Point- The cloud point is the temperature at which a petroleum product begins to form crystals or gel in cold temperatures which hinder operation of the fuel in the system it is being used. This term refers to test methods and equipment associated with ASTM Standard D 2500 for the determination of the cloud point of Petroleum Products.

The Collaboratory - A center at Messiah College which focusses applied research and project-based learning, in partnership with client non-profit organizations, businesses, governments and communities in this region and around the world. Areas of engagement include science, engineering, health, information technology, business and education. The twofold mission is:

To foster justice, empower the poor, promote peace and care for the earth through applications of our academic and professional disciplines.

To increase the academic and professional abilities of participants, their vocational vision for lifelong servant-leadership and their courage to act on convictions.

DOE- The United States Department of Energy, the sponsoring organization of the Messiah College Biodiesel Fuel Generation Project.

Flash Point- The flash point is the point at which the vapor above a liquid combusts when exposed to a flame. This term refers to test methods and equipment associated with ASTM Standard D 93 for the determination of flashpoint by Pensky-Martens Closed

Cup Tester.

Glycerin- The by-product of the biodiesel production process, which is refined to remove excess methanol and the processed in other applications.

Messiah College – the recipient of Department of Energy funds to small-scale biodiesel production. A Christian college of the liberal and applied arts and sciences located in Grantham, Pennsylvania.

Methanol – This is the alcohol used for biodiesel production throughout this project.

Research Team – The group of students, staff, and faculty at Messiah College that performed the research, development, and testing associated with this project.

Total and Free Glycerin- The total and free glycerin are the byproducts that remain after the biodiesel reaction occurs. Bond glycerin is the glycerin that has not be fully reacted through the transesterification process and requires a more thorough reaction to remove. Free glycerin is the glycerin still suspended in the biodiesel solution and can be removed through further washing and filtration. The combination of bonded and free glycerin is the total glycerin. This term refers to test methods and equipment associated with ASTM Standard D 6584 for the determination of total and free glycerin in B-100 Biodiesel Methyl Esters by Gas Chromatography.

Waste Vegetable Oil- Vegetable oil that has been used for cooking or other applications that is consider waste and will be discarded.

Water and Sediment- The water and sediment are the remnants that have not be removed through the washing and drying process. This term refers to test methods and equipment associated with ASTM Standard D 2709 for the determination of water and sediment content in middle distillate fuels by centrifuge.

III. Actual Accomplishments versus Goals and Objectives

Project Objectives

Much past and current biodiesel research and development, and attendant funding, has focused on issues relevant to large scale production process and processor design. Large-scale biofuel production plants are now in various stages of development in our region. What is lacking, however, is processor and process research and development to facilitate ASTM D 6751 certifiable biodiesel production by entrepreneurs and institutions seeking to leverage the opportunities afforded by biodiesel for recycling and other small-scale applications. Small scale production could become a viable commercial option, given the low cost of production equipment. Equipment for process research and quality testing is expensive, and prevents many small-scale producers from improving or certifying their processes. Moreover, no laboratory is readily available to small-scale producers in central Pennsylvania that provides access to test equipment and expertise needed for process optimization and quality assurance.

Objectives for this project are to:

1. Equip a laboratory for developing and optimizing processes and processors for small scale biodiesel production.
2. Develop a scalable process and processor design suitable for ASTM certifiable small-scale biodiesel production.
3. Identify quality enhancement and cost reduction protocols for process and processor design using feedstock and production ingredients readily available to small scale producers.

Outcomes for this project will be:

1. A laboratory to enable ongoing research and development in support of entrepreneurs, municipalities, and others pursuing ASTM certifiable small scale biodiesel production in our region.
2. Documentation of best practices for ASTM certifiable small scale biodiesel production.
3. Scalable process and processor design based on new research into optimizing design variables for quality assurance and cost.

Project Scope

This is a research and development project focusing on biodiesel conversion technology in the Biomass program of the DOE EERE, and assists with DOE goals of increasing biofuel production and building the biofuel industry. The project is in the Waste Processing pathway C milestone (M.7) with an emphasis on Thermochemical Conversion program barriers (Tt). The focus is small scale biodiesel production, an important niche market opportunity with potential to serve the recycling efforts of municipalities, institutions, and entrepreneurs. The project advances small-scale process and processor equipment design capable of ASTM D 6751 certifiable biodiesel production. Activities include literature research, a best practices survey, laboratory research, engineering design, prototype performance testing, and quality analysis. Outcomes are publicly available to entrepreneurs, municipalities, and other entities seeking to develop commercial or public small scale conversion facilities.

Examples of municipalities recycling waste oil to biodiesel include Loveland, CO and San Diego, CA. Examples of private small scale producers providing recycling services to local communities include Piedmont Biofuels near Greensboro, NC, Greycle Fry-O-Diesel in

Philadelphia, PA, Yokayo Biofuels in northern CA, Greycycle in Tuscon, AZ, and Rocky Mountain Sustainable Enterprises in Boulder, CO. The volume potential for biodiesel production by recycling is modest, but it is a contribution independent of agricultural economics and one that is highly participatory, offering significant potential to increase awareness and engender public support for alternative fuels.

Phase 1- Planning and Infrastructure

Space Development

1. **Planned Activities:** The purpose of this task was to identify and equip a space to house the necessary equipment for development and optimization of small scale biodiesel processes and processors, ASTM quality assurance studies, and performance testing of biodiesel fuel. This task served as a means to determine that utilities and other infrastructure are suitable to house and power the equipment required to achieve these goals. The activities associated with this task included:
 - Space Selection
 - Laboratory Layout Design
 - Utility Infrastructure Survey and Provision
 - Safety Infrastructure Survey and Provision
 - Bench and Storage Provision.

The key milestone for the purpose of internal tracking for this task was the acquisition of space to be used for laboratory development.

2. **Actual Accomplishments:** This task and all associated subtasks have been completed in accordance with Table C of the Quarterly Summary report. The task of selecting space was primarily performed by the research team as well as the Dean of the School of Science, Engineering, and Health and college administration. Several different options were considered during this process which included on-campus laboratory space, rental of a portable space to be placed on-campus, and off campus rental space. After analyzing the cost, feasibility, and accessibility issues associated with each option, a space was selected. The space selected for laboratory development is part of an existing on-campus engineering laboratory. This existing space has been temporarily divided and approximately one third of it is used for biodiesel research and development. The space is divided using temporary partitions and a second door has been installed through the masonry wall into the hallway. This provides access to the room without disrupting classes that may be occurring in the adjacent lab. The door and temporary dividers have been installed and the room has been populated with necessary equipment in accordance with the Laboratory Layout Designs.

Several Laboratory Layout Designs were created to fit the selected space. These layout designs take into account research and considerations for prototype processor design equipment, quality testing equipment, bench and storage requirements, safety provisions, and accessibility for ease of use. The main consideration for the laboratory layout design were safety, access to electricity, access to storage areas, and maximization of workable area. A final layout design has been selected for the room

based on the requirements of the testing equipment. While this layout was developed, a utility infrastructure survey was completed to determine modifications that would be required to the existing space. Based on the laboratory layout design, only minor modifications were required to provide adequate infrastructure for the laboratory. The main modifications were the addition of a second door to allow access to and from the hallway and the additions of electrical outlets to accommodate the power requirements for the gas chromatograph. The provisions to perform these modifications to the infrastructure of the selected space have been completed and any further modifications will be performed as necessary.

A safety infrastructure survey was also performed with the college and township's safety officials to ensure that the selected space would meet all safety requirements. Equipment such as fire extinguishers, eye wash apparatus, and spill containment materials have been purchased and installed in the selected space. All students, faculty, and staff that work in the lab space have been trained to use this equipment and know the proper procedures to follow in the event of a fire, accident, or spill. Bench and storage provisions have also been assessed. Benches have been received and installed in the lab according to the laboratory layout design. Storage provisions have been met through the use of standing cabinets as the room's temporary division from the existing lab space. These cabinets and the storage areas in the lab furniture provide ample storage space for the lab. Proper storage containers for the necessary chemicals and reagents have been purchased as well as chemically resistant benches. A flammables cabinet has been installed in the lab and chemical resistant furniture has been purchased and installed.

Upon the completion of the development of the laboratory space, it was determined that further space would be required to install and operate a small scale reactor for the purpose of research and development. This reactor was previously housed in a trailer that was parked outside. This trailer has been used as the primary reactor for several years, yet each winter production stops due to the cold temperatures. In order to continue research, development, and testing throughout the winter months, the test reactor required a dedicated, heated indoor space for it to be installed. Because the current lab space is part of an academic classroom and contains the quality testing equipment, it was not feasible to install and operate a biodiesel reactor in this space. A survey was performed of other spaces available on campus to be used for this purpose, however nothing feasible was determined. The survey was then expanded to off campus spaces within a close proximity to campus. A large heated garage space was found within a short distance of the laboratory space. A preliminary survey was performed to determine if this space would be appropriate to house the test bed reactor. It was determined that this space is ideal for the installation and operation of a small scale test bed reactor and would provide ample space, power, and storage for the required equipment and materials. The owner of this space agreed to lease it to the project team for as long as the team would need the space. The space became available in early August 2009 and the team moved the current reactor and other necessary equipment into the garage to begin operation prior to the start of the academic year.

3. **Explanation of Variance:** There is no variance between the actually activities of this task and the planned activities. However, there is a variance in the completion date. As a result of delays in arrival of equipment, the installation of chemically resistant benches and furniture was delayed until the first week of May 2009. This subtask was to be completed by December 15, 2008 but was initially delayed by the installation of the hallway door to the lab. Installation of this door was completed in early February 2009 and completion of the task was furthered delayed by the late arrival of the chemically resistant benches. This work was completed by May 5, 2009, at which point the Space Development Task was complete.

Laboratory Development

1. **Planned Activities:** The purpose of this task is to establish a laboratory capable of performing the most pertinent quality assurance tests associated with ASTM D 6751 for small scale biodiesel production. The activities associated with this task included:
 - Test Selection
 - Equipment Acquisition
 - Equipment Installation
 - Equipment Calibration.

The key milestone for this task come as the first deliverable for the project in the form of a laboratory capable of performing a select number of ASTM D 6751 quality assurance tests. A key milestone for the purpose of internal tracking in completing this deliverable was the completion of equipment set up and calibration.

2. **Actual Accomplishments:** The task of test selection was completed primarily by the PI and the research team with the assistance of several experts in both biodiesel production and testing. The goal of this task was to determine which test will be performed in-house and which tests will have to be outsourced to another testing laboratory facility. Extensive research has been done into the selection of tests that will be performed in accordance with ASTM D 6751. The factors that govern this research and ultimately the test selection include: the expected frequency of test performance, the costs associated with the test, the cost associated with outsourcing the test, and the significance of the test to small scale production. Part of this research included a visit by the PI to a biodiesel test facility at Intertek Labs in Carteret, New Jersey. This lab was chosen based on the recommendations of personnel at Keystone Biofuels, a local commercial biodiesel producer in Shiremanstown, Pennsylvania. The purpose of this visit was to visualize and survey the requirements of a laboratory capable of performing all tests in accordance with ASTM D 6751. Laboratory personnel gave the PI a tour of the facility, showing each test and its required equipment associated with ASTM D 6751 quality testing for biodiesel. This allowed the PI to determine the space requirements for specific tests and to receive recommendations in regard to manufacturers and suppliers of testing equipment. This visit also provided a connection to a facility capable of performing biodiesel quality testing that is beyond the capabilities of the laboratory being developed for this project.

Another aspect of this research involved gathering the specifications for the tests required by the *ASTM D 6751 Standard Specification for Biodiesel Fuel Blend Stock (B100) for Middle Distillate Fuels* standard. Each test standard specified by ASTM D 6751 has been acquired, reviewed, and evaluated based on equipment required, overall cost, relevance to small scale production, and practicality. Based on these criteria certain tests have been ruled out of what is considered possible for this project, specifically *ASTM D 613 Standard Test Method for Cetane Number of Diesel Fuel Oil*. This test requires a complex engine apparatus of which there are few in the country. The PI was able to see one of these engines at the Intertek Lab and determined that is not feasible for this project due to space requirements, complexity of use and overall cost. The remaining tests were evaluated based on cost estimates from manufacturers and suppliers. It was the goal of the project to perform as many of the tests as possible associated with the ASTM D 6751 quality standard for biodiesel. The PI and research team selected five key tests to perform in the on campus laboratory. These include:

- ASTM D 93 Standard Test Methods for Flash Point by Pensky-Martens Closed Cup Tester*
- ASTM D 2709 Standard Test Method for Water and Sediment in Middle Distillate Fuels by Centrifuge*
- ASTM D 2500 Standard Test Method for Cloud Point of Petroleum Products*
- ASTM D 664 Standard Test Method for Acid Number of Petroleum Products by Potentiometric Titration*
- ASTM D 6584 Test Method for Determination of Free and Total Glycerin in B-100 Biodiesel Methyl Esters by Gas Chromatography.*

Quotations were received from multiple vendors and manufacturers of the equipment required to perform these tests and the PI selected the most practical and cost effective equipment for the lab.

The lab has received equipment necessary to perform the following tests:

ASTM D 93 Standard Test Methods for Flash Point by Pensky-Martens Closed Cup Tester. The equipment selected to perform this method was the Koehler Instruments K16200 Pensky-Martens Closed Cup Flash Point Tester (See Photo No. 1 in Appendix A.). The PI and research team have performed this test on samples of biodiesel from the current processor system. This equipment and testing method is straightforward to operate and after a brief time of familiarization and practice, the research team became proficient at determining the flash point of samples in accordance with the ASTM test method. This piece of equipment is also useful for determining the methanol content of glycerin that has been processed in the methanol recovery system. The flash point tester is used to determine the flash point of glycerin after methanol recovery has been performed to determine if recovery was sufficient and the methanol can be safely stored and disposed.

ASTM D 2709 Standard Test Method for Water and Sediment in Middle Distillate Fuels by Centrifuge. Initially the PI selected and ordered the Accuspin Model 400 Bench Top

Centrifuge and all necessary accessories based on the recommendation of a technical representative from an equipment manufacturer. However this centrifuge could not accommodate the required 100 ml capillary tipped sample tubes in accordance with the test method. This centrifuge was returned and a credit was issued. Further research was performed to establish the necessary centrifuge able to perform the testing with the appropriate sample tubes. It was determined that the Koehler Instruments K61002 Heated Oil Test Centrifuge and its accessories could accommodate the sample tubes and this apparatus has been purchased(See Photo No. 2 in Appendix A.). Arrival of this centrifuge was delayed because the item was back order by the manufacturer. All other necessary equipment and apparatus to perform this test was received and upon arrival the new centrifuge was set up and the research team began performing this test on samples. This piece of equipment required a short period of familiarization and practice for the research team to become proficient at performing tests. This centrifuge is also a valuable tool in working with alternative feedstocks and determining their value in biodiesel production. Specifically the research team has used this piece of equipment to test methods for degumming crude soybean oil.

ASTM D 2500 Standard Test Method for Cloud Point of Petroleum Products. The equipment selected to perform this test was the Koehler Instruments K46100 Bench Model Cloud and Pour Point Bath and all necessary accessories (See Photo Nos. 3 and 4 in Appendix A.). This apparatus has been installed and after a brief period of familiarization and practice the research team began using it to perform cloud point testing in accordance with the ASTM method.

ASTM D 664 Standard Test Method for Acid Number of Petroleum Products by Potentiometric Titration. The equipment selected to perform this test was Denver Instruments Model 350 Potentiometric Titrator (See Photo No. 5 in Appendix A.). This apparatus was installed and calibrated and testing began immediately to determine the total acid number of biodiesel samples. The research team worked with a titration expert from Denver Instruments to install and calibrate the titrator as well as to begin performing the required test method. This instrument has required extensive training and practice by the research team to become proficient at performing this test. The titrator requires frequent calibration and it has been difficult for the team to establish consistent results. Further work was done to perfect the team proficiency at using this piece of equipment. The team calibrated the titrator and used it to perform numerous tests on sample of biodiesel. These tests provide consistently accurate results and the team is comfortable both calibrating and performing testing with this piece of equipment. The autotitrator is also useful in titrating samples of feedstock to determine the appropriate amount of catalyst required to perform the transesterification reaction. In the past this has been done by manual methods, however with the aid of this instrument, very accurate titrations can be performed and better reactions can be facilitated to produce biodiesel.

ASTM D 6584 Test Method for Determination of Free and Total Glycerin in B-100 Biodiesel Methyl Esters. The equipment selected to perform this test method was the Thermo Scientific Trace GC Ultra with the TriPlus Auto Sampler (See Photo No. 6 in

Appendix A.). This system is designed specifically to perform the ASTM D 6584 test method. This equipment did not arrive until the end of June 2009 and it was not installed until late September 2009. This delay in installation came from minor delays in modification required to the laboratory space to accommodate the system as well as scheduling issues to coordinate installation with the manufacturer. However the system was installed and functional. The research team received on site training from an applications engineering from the manufacturer to begin operating the biodiesel package in November 2009. This instrument required extensive training and familiarization by the research team. The calibration required for the system is complex and must be done regularly. The gas chromatograph has been successfully calibrated and used to test numerous samples of biodiesel. These results were very consistent but seemed to consistently miss detection of free glycerin. Further work was done to determine if the calibration of the gas chromatograph is incorrect or if in fact there was not free glycerin in the samples to be detected.

The PI and research team have worked to develop the training that is required for the equipment selected and set up this training for all necessary personnel. Over the course of the project, the PI was able to train the research team to operate a majority of the tests the lab is capable of doing. Further work needs to be done with future team members to train them in operation of the gas chromatograph.

As the team worked to perform all of the tests selected for the lab, work was also being done to determine what other tests can and should be performed. The five tests that have been selected are considered part of Critical Specification Testing by the BQ-9000 quality program for biodiesel production. The remaining Critical Specification Tests required by the BQ-9000 program are: *ASTM D5453- Standard Test Method for Determination of Total Sulfur in Light Hydrocarbons, Spark Ignition Engine Fuel, Diesel Engine Fuel, and Engine Oil by Ultraviolet Fluorescence (Sulfur Content)*, *EN 14112- Fat and oil derivatives - Fatty Acid Methyl Esters (FAME) - Determination of oxidation stability by accelerated oxidation test (Oxidation Stability)*, and *ASTM D6751 Annex- Cold Soak Filtration Testing (Cold Soak Filtration)*. Preliminary investigation showed that the Cold Soak Filtration testing could likely be performed using equipment already housed in the lab while the Sulfur Content and Oxidation Stability testing would require the acquisition of specialized equipment. As further research was performed, the feasibility and significance of these tests was determined and the decision was made to not pursue equipment to perform these tests.

Throughout the project equipment training of all research team members was completed and a decision was made to not pursue the remaining tests to complete the BQ-9000 Critical Specification Testing. All necessary personnel were trained to use the purchased equipment and test existing biodiesel samples. Further equipment and supplies were purchased for the lab as necessary over the project period.

3. **Explanation of Variance:** There was no variance between the planned activities and the actual accomplishments for this task. Variance in the completion dates occurred

based on delays in the completion of the Space Development task, delays in the arrival of ordered equipment, and issues related to installation and calibration. The proposed completion date for this task was May 5, 2009, however based on the previously mentioned delays and expected delays, actual completion was July 1, 2009. Further delays in the arrival of equipment, modification to the lab space, and the necessity to return and order a new centrifuge have pushed the completion date of this task back to November 15, 2009.

Phase 2- Best Practice Benchmarking

Best Practice Benchmarking

1. **Planned Activities:** The purpose of this task was to study literature and network with exemplary commercial and municipal small scale biodiesel producers to identify and benchmark the primary technological and business parameters associated with the industry. Parameters such as process and processor technology, feedstock availability, quality assurance testing, capacity, cost, safety, energy requirements, waste management, environmental impact, and technology development processes will be consider. The activities associated with this task included:
 - A literature review
 - Identifying a slate of enterprises for benchmarking
 - Identifying technology and business challenges
 - Developing a site survey procedure
 - Site visits and interviews
 - Benchmarking of state-of-the-art small scale production processes and processors
 - Preparation of a final literature review and benchmarking documentation.

The key milestone for this task was the second project deliverable of the completion of the literature review and benchmarking documentation.

2. **Actual Accomplishments:** This task was primarily completed by the PI and the research team. Research was completed to create a list of topics to be investigated through the literature review and benchmarking of state-of-the-art small scale production. Specific areas of research include the following:
 1. State of the art process and processor technology
 - The Mcgyan Process
 - Microwave Processing
 - Ultrasonic Processing
 - Continuous Process/Processor Technology
 2. State of the art business practices for small scale producers
 3. Known feedstocks
 - Current feedstocks
 - Future feedstocks
 4. Current production methods

- Transesterification- Base Catalyzed
 - Esterification- Acid Catalyzed
 - Combined Acid/Base Reaction
 - Washing
 - Drying
5. Quality assurance methods
 - Professional- BQ-9000, ASTM D6751
 - Small scale- Flip Test, 27/3 Test
 6. Chemicals Required
 - Alcohol- Methanol, Ethanol
 - Catalyst- KOH, NaOH, Sulfuric Acid, Etc.
 - Additives- Cold Flow, Anti-bacterial
 7. Top research priorities and needs of small scale producers.
 8. Methanol Recovery
 9. Wash Water Processing

The top research priorities and needs of small scale producers include numerous areas of research as listed below:

1. Reduction of Energy Required
 - Oil Collection and Pre-treatment
 - Oil Heating
 - Washing
 - Drying
2. Reduction of Water Required
 - Improved Washing Methods
 - Reduction of total number of wash cycles
 - Dry Washing
3. Improved Drying Methods
4. Continuous vs. Batch Processing
5. Quality Assurance for the small scale producer
 - Local Testing Facilities
 - Cost
 - On-site testing
 - Process/processor capable of producing consistent high quality fuel
6. Waste Disposal/Use
 - Methanol Recovery
 - Uses for Glycerin
 - Soap
 - Composting
 - Dust Suppressant
 - Other
 - Wash Water Processing
7. Safety

Literature has been gathered and reviewed in these and many other areas. The PI and research team created summary reports for this literature and compiled this information into the final literature review and best practices reports. Student researchers and the PI completed best practice and literature review research focused in the areas of Feedstock, Alcohols, Catalysts, Production Methods, Washing Methods, Glycerin Processing, Use, and Disposal, Safety Issues, Chemical and Material Handling Issues, and Quality Testing and Assurance Methods. The PI attended the Sustainable Biodiesel Summit, January 31 through February 1, 2010, and the National Biodiesel Board Conference, February 1 through February 4, 2010, to research and network with exemplary commercial and municipal small-scale biodiesel producers. These conferences provided the opportunity to interact and network with producers from around the nation and world. They also provided access to cutting edge technologies, theories, and processes in the biodiesel production industry.

Contacts were also made with several commercial and municipal producers that were used as benchmarking enterprises. A list of enterprises for benchmarking can be seen below:

- Piedmont Biofuels- Pittsboro, North Carolina
- Keystone Biofuels- Shiremanstown, Pennsylvania
- Lake Erie Biofuels- Erie, Pennsylvania
- Middletown Biofuels- Middletown, Pennsylvania
- Dickinson College Biodiesel- Carlisle, Pennsylvania

The research team and the PI were able to perform site visits at the Keystone Biofuels production facility in Shiremanstown, Pennsylvania, as well as the Dickinson College production facility in Carlisle, Pennsylvania. The team also attended the Fueling the School Intercollegiate Biodiesel Conference held at Dickinson College.

During the project period, research was gathered for the Best Practices reporting including a visit to Dickinson College to discuss biodiesel on a college campus and the purchase of the 2nd Edition of the Biodiesel Handbook (Knothe, Krahl and Van Gerpen). This information was combined with the previously collected data and best practices research to complete the Literature Review and Best Practices Report.

The research team also acquired the “Journal of ASTM International: Selected Technical Papers: STP 1477 Biofuels” as further research to establish the best practices in the biodiesel industry. This resource was used to compare findings in site visits, interviews, and other technical documents to establish the most common best practices in this industry and how they apply to small scale production of biodiesel.

The PI and research team completed the final site visits to businesses that have been identified as exemplary in the area of both commercial and small scale biodiesel production. Further research was done through the site visits, interviews, and literature review to establish the top research priorities and needs of small scale biodiesel producers.

3. **Explanation of Variance:** To date there has been no variance between the planned activities and the actual accomplishments for this task. However there is a variance anticipated in the completion date of this task. The task was scheduled to be completed by June 1, 2009. Due to the class schedules of the research team as well as research and development being performed in other areas, extended site visits were not possible during the course of the academic year. The research team and the PI purposed that the completion date of the Best Practice Benchmarking Task be extended to March 30, 2012 to allow for further site visits and additional time to thoroughly research the topics listed above. The team proposed that this deliverable to be completed in the final stages of the grant work to allow all research and findings developed over the course of the grant period to be included in the literature review and best practices study. Please see Table C. in the Quarterly Summary Report for proposed adjusted completion dates.

Phase 3 - Process/Processor Test Bed Design and Implementation

Process/Processor Test Bed Development

1. **Planned Activities:** The purpose of this task was to identify the core process and processor design variables and establish hardware options for implementing alternative designs to these variables through a research test bed. The activities associated with this task included:
 - Identifying the core process design variables
 - Hardware implementation of design building blocks
 - Identifying measurement and instrumentation objectives
 - Test bed design.

The key milestone for this task was the third project deliverable of the completed test bed design.

2. **Actual Accomplishments:** Research, planning, and design of the process/processor test bed were completed. Based on the teams research the following core process design variables were identified:
 - Oil Pressing- During the project period the team performed research into the area of small scale seed pressing as a means to produce vegetable oil. This research came about through the purchase of a number of small presses that allowed the team to work with multiple locally available feedstocks to establish best practices for small scale seed oil pressing (See Photo No. 8 in Appendix A.). The goal of this research was to incorporate pressing into the final system design as a means of producing edible cooking oil that can be used for cooking and the resulting waste vegetable oil would then be used for biodiesel production. This model allows the crops used as feed stock to meet the needs of the food market and then be converted to fuel afterward. Research associated with pressing oil from seeds includes:
 - Seed Preparation/Moisture Content- The most important element to extracting oil from oil bearing seeds is the preparation and drying of

the seeds. Drying the seeds to reduce the overall moisture content remove water from the seeds and therefore produces a higher quality oil when extracted. A member of the research team established best practices for seed preparation and determined the moisture content of seeds prior to pressing.

- Pressing Rate- The screw presses the team is using to extract oil from seeds are complex piece of equipment that allow for various coarse and fine adjustments to fine tune the equipment and create the most ideal settings for oil extraction. These settings have been researched and documented so that the team can work with the presses to fine tune these parameters to achieve the most ideal press rate and therefore the greatest rate of oil extraction.
 - Feedstock- The key advantage to having the ability to press seeds into oil is access to new local available feedstock. Using the presses the team worked with locally grown sun flowers and soybeans. Using these crops, the team was able to fine tune the press to achieve the highest press rate and greatest oil extraction.
- Oil Pre-Treatment
- De-watering/Drying
 - Filtration
 - Degumming of crude oils- With the addition of the pressing research, as stated above, the important of degumming crude vegetable oils becomes a more pressing issue. Preliminary research and collaboration with other local small scale pressing operations has shown that the degumming of crude oils is a fairly simple process that can be achieved through several methods. The first method is a water washing process with the addition of citric acid to hydrate the phospholipid gums in the oil. When hydrated these gums will then settle out of the oil and can be removed either using a centrifuge or basic filtration process. The second method is the use of a press filter which is a series of increasingly fine mesh filters that remove gums as the oil is pumped through them under high pressure. This method is used on an industrial level therefore the team is certain that it will also be successful on the small scale.
 - Esterification (Acid Treatment) of High Free Fatty Acid content oils
 - Centrifugation- This area was specifically researched as a method to accomplish de-watering/drying of oil as well as filtration. Centrifugation also provides a method for removing gums from crude oils directly after pressing has been completed. A centrifuge

was used to filter and purify vegetable oil prior to biodiesel production. The research team tested different feed rates into the centrifuge as well as different rotational speeds for the centrifuge to establish the ideal parameters for filtration (See Photo No. 12 in Appendix A.). The results of this research will be reported in the upcoming reports.

- Oil Heating Method
 - Electric Resistance Heating Elements- This is the method the research team has used for a number of years with very good success. The resistance heating elements are inexpensive, simple, and readily available for application in small scale biodiesel production systems.
 - Microwave- Minimal research was done in using microwave heating as a means to heat vegetable oil for the biodiesel production process. The research team worked with two microwave heating systems and attempted to use these to consistently and uniformly heat oil with limited success. The research team was not specifically familiar with microwave technology and only achieved limited success in using these systems for oil heating. Based on this preliminary work the team felt that microwave heating sources were not technically or financially feasible for small scale production systems and continued research into more traditional oil heating sources.
 - Uniformity- The research team has determined that uniform heating of the oil is the most significant aspect of the heating method. This was accomplished by circulating oil over electric heating elements.
- Alcohol- The research team uses methanol for all biodiesel production and has established that it produces the highest quality fuel. Limited work has been done with ethanol and there was marginal success in producing biodiesel. The main focus of research has been using methanol recovered through the teams methanol recovery research in the biodiesel production process as well as to determine the ideal concentration of methanol to achieve the highest quality biodiesel.
- - Type- Methanol, Ethanol, Other
 - Source- Commercially available or self made (ethanol)
 - Purity- Through the team's research into methanol recovery the issue of what purity of methanol is required for biodiesel production and can this level of purity be achieved through small scale methanol recovery systems. The team established that methanol must be at least 85% pure to be feasible to reuse in the production process.

Using the methanol recovery system the team is recovering methanol consistently with purities of greater than 98%. This recovered methanol is then blended with new methanol and used for biodiesel production. In considering the use of ethanol for biodiesel production, as a means of making the process more sustainable, the question of ethanol purity also arises. This area has not been researched by the team, however through conversation with other small scale producers the issue has become apparent. Can a small scale ethanol production process produce ethanol that is of purity high enough to be used for biodiesel production?

- Volume- Common practice in biodiesel production, both commercially and small scale, is to use excess methanol in the process to ensure that the reaction which creates biodiesel is not reversed during settling. Typically this requires using 20% methanol by volume of oil, while only 10% of the methanol is consumed in the reaction. While developing the test bed processor the team considered this issue and used the test bed to explore the minimum amount of methanol that can be used in the reaction while still producing a high quality biodiesel product.
- Catalyst- The research team uses KOH as the primary catalyst for biodiesel production and this is based on the viscosity of the resulting glycerin. With NaOH the glycerin byproduct is very viscous and difficult to pump when cold. The KOH glycerin is much less viscous and easier to work with as it cools.
 - Type- KOH, NaOH, other.
 - Source
 - Purity
 - Quantity
- Alcohol/Catalyst Mixing Method
 - Natural Mixing- The research team has primarily been using KOH and Methanol based on the fact that the two mix easily and do not require pumping or mechanical mixing. The KOH dissolves readily in the Methanol and the team has had good success in achieving high quality biodiesel with this method.
 - Pump Mixing
 - Ideal Flow Rates
 - Mechanical Stirring

- Oil/Methoxide Mixing Method
 - Natural Mixing
 - Pump Mixing- The research team has primarily used a circulating pump to mix the Methoxide and Oil and the resulting biodiesel has been high quality. Further work will be done to establish the ideal flow rates and mixing times for this method.
 - Ideal Flow Rates
 - Mechanical Stirring
- Glycerin Separation Methods
 - Centrifuge- To date this research has not been done due to extended research using the centrifuge to filter and purify vegetable oil prior to processing into biodiesel.
 - Gravity- The team has primarily allowed glycerin and biodiesel to separate by gravity and the resulting fuel has been of an acceptable quality. However using centrifugation it is anticipated that separation time will be reduced from just using gravity separation and it is also believed that washing of the resulting fuel will be easier due to the reduce glycerin remnants.
- Biodiesel Washing Methods
 - Water Washing- The team has only researched water washing methods for biodiesel and the resulting biodiesel has been very high quality. However to achieve high quality biodiesel, large quantities of water are required therefore future research will focus on reducing overall water required either through dry washing, additives or other washing methods not yet determined.
 - Dry Washing
 - Additives
- Biodiesel Drying Methods
 - Hydrocyclone- A literature review has been done to learn about the hydrocyclone but no tangible testing has been performed.
 - Centrifuge- In the near future, a centrifuge will be used to separate biodiesel and wash water and the results of this research will be reported. To date this research has not been performed due to extended work in using the centrifuge to process waste vegetable oil prior to processing it into biodiesel.
 - Bubbling- The team has been using the bubble method to dry biodiesel with very good success. The simplicity of this method

makes it very desirable, economical, and realistic for small scale operations.

- Heat Lamp- Limited research has been done using a heat lamp combined with the bubble drying method to reduce the overall drying time. However no tangible results have been collected to show that the addition of the heat lamp improves the drying process.
- Filtration of Final Product- Currently the team use a diesel fuel filter as its final stage of filtration for biodiesel. The team has found that after thorough washing and drying, this simple filter works well to remove any remaining sediment from the final product. This method is cost effective, accessible, and very feasible for application in small scale production processes.
- Methanol Recovery
 - During production process- Research has been done to determine the feasibility of recovering methanol directly after the production process. There appears to be research showing that this is both possible and effective, however the research team feels that the possibility of reversing the reaction is a risk that must be considered and researched further.
 - From waste glycerin- All of the teams work has been done performing methanol recovery on waste glycerin as it has the highest concentration of methanol compared to other aspects of the process.
 - From wash water- Lab testing has been done to determine the validity of performing methanol recovery on wash water. To date the results of this testing show that minimal amounts of methanol can be recovered however further research must be done to establish the feasibility of this process on a larger scale.
 - Purity of Recovered Methanol- The research team has spent considerable time developing methods to verify the quality of recovered methanol and has had good success in this area. The team has also achieved good success in using high quality recovered methanol to produce biodiesel.
 - Distillation Column Design- Work was done to establish the ideal column design and distillation system for a methanol recovery system of this scale. Prior work was done using reflux columns and water cooled distillation columns. This work produced high quality methanol yet required large amounts of time to do so. Current work is being done using simple distillation columns that incorporate vapor expansion chambers and air cooled columns.

Based on the initial list of variables, hardware options for the test bed design have been researched and purchased. Research was performed to evaluate the necessary measurement and instrumentation objects for the test bed design and these elements have been determined and purchased also. Based on the core process design variables identified, the research team established what properties and characteristic should be monitored in the test bed and then determined the necessary instrumentation to perform this monitoring. Initial investigation established that pH, temperature, and flow rates are primary properties that will require measurement in the test bed. Instrumentation to monitor these properties has been researched and evaluated. Another aspect of the test bed that was developed was the possibility of using controllable flow rate pumps to precisely control the mixing and transfer of substances in the process. A Programmable Logic Controller (PLC) system was considered for the incorporation of instrumentation and controllable pumps. A PLC system allowed the team to precisely control and monitor the different elements of the process. The completion of this task led to the final test bed design. Upon completion of this design, the test bed including the necessary hardware, instrumentation and monitoring systems were installed. As stated earlier, a space was acquired to install and operate the test bed reactor. This space held the pervious reactor that was being used. The test bed was also installed at this location so that feedstock, catalyst, alcohol and all other necessary chemicals and supplies will be readily available.

The design of the test bed was completed as well as construction and implementation. This system was designed based on the use of a PLC, electronically controlled valves and pumps, temperature monitoring, pH monitoring, flow rate, level and pressure sensors. A 35 gallon insulated stainless steel tank was purchased and used as the main reaction vessel for this final test bed design. This final test bed design allowed for precise control and monitoring of all aspects of the production, washing, and drying process.

A new addition to the list of design variables above was the element of methanol recovery. As the team moved forward in the development of a test bed processor and worked to improve the overall quality of biodiesel produced through small scale processors, the importance of the methanol recovery element in overall design continued to be proven. The research team proposed that an integrated methanol recovery process and system be included in the overall design of the test bed as well as the completed small scale processor. Without an integrated methanol recovery system, the processor design is incomplete and leaves the end user with methanol laden glycerin byproduct and wash water that are technically hazardous materials which must be properly disposed of. The additions of a methanol recovery system to the test bed and final design provided a system that not only produced a safe glycerin byproduct but also produced recovered methanol that can be recycled back into the biodiesel production process. This created a system for a small scale producer that is environmentally friendly, more sustainable, and more cost effective. For several years small groups of students have been working in conjunction with the biodiesel team to develop methanol recovery prototype systems. The current prototype system is used to recover methanol from the glycerol produced by the biodiesel processor. Over the

course of this project the team incorporated this methanol recovery prototype into a small scale biodiesel processor prototype to produce a single system.

One aspect of the work done to develop a methanol recovery prototype that directly correlated to the biodiesel test bed design was the development of a temperature monitoring system. This system consists of an 8-channel thermocouple data logger system attached to a dedicated laptop with recording and analysis software installed. The purpose of the monitoring system is to identify, measure, and track critical temperatures in either the methanol recovery system or the biodiesel test bed and then determine correlations between these critical temperatures and the overall system performance and output.

Throughout the project period, the methanol recovery test bed design was completed and prototypes were developed. This test bed allowed the user to monitor the critical temperatures. Using this system the recovered methanol was tested for purity by checking the specific gravity using hydrometers. Based on the results of testing, new distillation column designs were developed to both increase quality as well as reduce the overall time to recover methanol from the glycerin. A newly developed column showed positive results for both increasing recovery time as well as improved methanol quality. Once recovery of the methanol appears to stop from the system, the remaining glycerin is tested to determine its flash point. The results of the flash point test establish the remaining methanol in the glycerin and determine how safe the glycerin is for disposal and transport.

A new prototype methanol recovery system was designed and implemented with very good success (See Photo No. 11 in Appendix A.). The new design employs an insulated 35 gallon stainless steel tank, the same style of tank being used for the biodiesel test bed design. This new methanol recovery system is completely electronically controlled and incorporates an in-tank heater which produces more uniform heating for the glycerin. This system also employs a new distillation column design that does not require chilled water cooling. Through the use of a longer copper column and a heater exchanger, methanol vapor is condensed through an air cooled system that is more efficient and appears to produce higher quality methanol. The electronic controls in this new prototype improve the overall safety of the system as well as performance through more uniform heating and overall temperature control.

Work was done to determine the appropriate size for the biodiesel test bed processor. Previously the team was operating a 40 gallon batch processor that was used to make biodiesel (See Photo No. 9 in Appendix A.). This system can easily be manipulated and sensors can be installed for it to function as a test bed processor however the overall output may be too large for precise control. The research team also developed a smaller 5 gallon prototype processor unit to determine its feasibility as a test bed processor. This system is very simple and only uses two tanks and one pump (See Photo No. 7 in Appendix A.). All of the supplies to build this system are readily available at local hardware stores and it is easily assembled. The 5 gallon prototype is also small and would allow for very precise control of the design variables. It is likely that this

processor design will be more similar to the final test bed design. However the final design for the test bed processor will likely be an even smaller unit, possible a 1 or 2 liter system that can be bench mounted and controlled by a computer.

A second small test bed reactor was created using a single 3 gallon tank with a pump and in-line heater (See Photo No. 10 in Appendix A.). This system was also very simple to construct and performed well for the purpose of testing small batches of oil to determine how suitable these are for biodiesel production. This system is also designed to run off of 12 Volt power allowing to be operated by a car battery or directly from an alternative energy source such as solar panels. One of the key benefits of this system is that it can be completely disassembled and transported in a regular sized duffle bag or suitcase. This makes this processor easy to transport for demonstration or for onsite testing in remote locations.

It was determined that the test bed system will be constructed using a 35 gallon stainless steel cone bottomed tank. A local vendor was discovered that has numerous tanks and is selling them at a very reasonable cost. The tanks are ideal for a biodiesel test bed application and could be used to produce batches of biodiesel ranging from 1 to 25 gallons. These tanks also have several ports and outlets that would easily accommodate the implementation of sensors and monitoring equipment. These tanks were purchased and the test bed system was constructed and completed by early September 2010.

The test bed was constructed in a manner that allowed for the team to easily change hardware elements. These hardware elements included the heating system, the pump, plumbing, and valves. The final design also allowed for precise control of the process elements of the system. These elements include the pump speed, heating rate, settling time, washing process, and drying process. After the production of the biodiesel is complete in the test bed processor, the resulting glycerin and biodiesel were tested to determine quality and purity. Glycerin was tested to determine methanol content using the flash point test. Biodiesel was tested using the equipment in the laboratory to determine total and free glycerin, acid number, cloud point, flash point and water and sediment. The results of these tests helped the team to determine the validity of the hardware and process configuration used to produce each batch of fuel made in the test bed processor.

The final test bed processor design was completed and the prototype began to be assembled. This design included a 35 gallon stainless steel tank, galvanized steel plumbing, electronic solenoid valves, and a Programmable Logic Controller (PLC). The design for the methanol recovery prototype was also completed and the prototype has been constructed and programmed. The methanol recovery system prototype has been found to function as designed and is currently being tested to determine ideal settings for electronic controllers.

The PLC biodiesel processor system design was completed and construction was completed shortly thereafter. Verification that the system and its programming operate

as designed was completed and testing of the system and the resulting biodiesel followed. The PLC processor design, construction and programming were completed. The system was tested to verify that it operated as designed and these tests have been positive. The system is operating very well in accordance with the initial design and programming. Several batches of biodiesel have been produced in the system and the quality of these batches have been verified. Further research and testing occurred over the course of the project period to fine tune the system and increase overall quality and performance.

Testing of the PLC processor continued with similar results to those found previously. It was determined that the quality of the initial feedstock used in this processor needs to be improved to establish consistently high quality biodiesel production from the PLC processor. Variation in vegetable oil feed stock quality has made it difficult to produce consistently high quality batches of biodiesel from this processor. The performance of the PLC processor is exactly as planned and all components are functioning well. By creating a consistently high quality vegetable oil source, the fine tuning of this system will be completed.

Testing of the newest methanol recovery prototype also continued with excellent results. The system continues to perform as designed and also consistently achieves purity levels of recovered methanol that exceed 90%. This system operates on a daily basis and has processed approximately 600 gallons of waste glycerin to date. A final operation and maintenance manual for this system has been completed and can be seen in Appendix D.9.

The final construction of the test bed design was completed and testing of the system was performed to verifying operation in accordance with the design. The prototype was used to produce biodiesel and this product was then tested to establish quality in accordance with ASTM D 6751. The research team continued to operate the methanol recovery system to determine any long-term design flaws that it may face. Manuals for each system developed through this research project have been created. These draft manuals have been reviewed and can be seen in Appendix D.

3. **Explanation of Variance:** There is currently no variance between the planned activities and actual accomplishments for this task other than the addition of the methanol recovery element of the test bed and final processor research and design. The completion date for this task does vary from the original completion date. Due to delays in lab development and best practice benchmarking, finalization of the test bed design was postponed. The development of the basic test bed system using readily available parts has allowed for further design consideration. Though this system allows for precise control of the process parameters, it does not incorporate the measurement and monitoring instrumentation currently. The final test bed design was completed by May 15, 2010. While completing the design, hardware and instrumentation were ordered so that when the design was complete the system could be assembled quickly.

Process/Processor Test Bed Implementation

1. **Planned Activities:** The activities associated with this task included:
 - a. Purchasing hardware and measurement and control instrumentation for the test bed.
 - b. Constructing the test bed.
 - c. Installing measurement and control instrumentation.
 - d. Verifying that the test bed performs as designed.

The key milestone for this task was the fourth project deliverable of the completed implementation of a test bed that performs as designed.

2. **Actual Accomplishments:** The final stages of the implementation of the newest test bed were completed. As stated in the previous task, the new test bed design was complete and the appropriate hardware and measurement and control instrumentation for the test bed were purchased. The test bed was constructed and the measurement and controlling instrumentation were installed and calibrated for the system. A new space was established to construct and install the test bed that provides a safe, dedicated area to house the test bed as well as allow easy access to the necessary chemicals and supplies for its use. After the construction and installation were complete the performance of the test bed was verified in accordance with the original design.

All of the components for the test bed were acquired. These included a 35 gallon stainless steel tanks used as the main reaction vessels, temperature monitoring equipment, a Programmable Logic Controller (PLC) and the required software, the heating system for the test bed, and a centrifuge to be used for separation purposes. The stainless steel tank is fully insulated and provides ample ports for the installation of measurement and control instrumentation. The tank is also pressure rated which will allow for testing at both positive and negative pressures. The temperature monitoring equipment is a combination of the wireless thermocouple monitoring system already in use as well as a large probe thermometer that was inserted into the tank to verify internal temperatures. The PLC and necessary software were acquired and it was verified that the PLC and software can communicate and function properly. This software was purchased and installed for the research team to learn how it works and develop the control program for the processor. The heating system for the test bed is a thermostat controlled heating element that mounts directly into the stainless steel tank. The thermostat on the heating element was already programmed to the appropriate temperature for biodiesel production and will function well for this use. This final stage of the test bed implementation was installing the plumbing system on the tank and verifying that all systems function as designed.

All mechanical components were purchased and installed. Leak testing has been performed on all of the plumbing and all solenoid valves were tested to ensure operation. The pump and circulation system were fully installed and operated in accordance with the design. The final step required to complete the test bed processor was the installation and programming on the electrical control system through the

Programmable Logic Controller. This equipment was purchased and the designs for the electrical system and programming code for the PLC were completed.

A centrifuge was purchased to be used with the test bed processor for a number of functions. The initial use for the centrifuge will be to process waste vegetable oil prior to biodiesel production. This removes water and sediment from the oil prior to production and should increase the overall quality of the fuel produced. Research was done to determine the ideal feed rate of oil into the centrifuge as well as the appropriate RPM setting for the centrifuge to produce the highest quality oil. Research was also done with the centrifuge to determine if it is capable of separating glycerin that is suspended in the biodiesel after settling has occurred. This reduces the overall settling time and also improves washing methods. By removing more glycerin from the biodiesel prior to washing, the washing will be easier to perform and will occur more rapidly. The final use for the centrifuge would be to separate the wash water from the clean biodiesel producing a drier fuel. By using the centrifuge to remove water from the fuel, overall settling time for washing will be reduced and the final product will be of higher quality.

Testing was performed using the centrifuge to establish the optimal feed rate and operating speed to achieve the highest quality oil from the system. A testing program was set up to study the oil feed rate into the centrifuge as well as the operation speed of the centrifuge independently. Oil samples were collected prior to centrifugation and after centrifugation for each batch tested at each setting. These samples were then tested in the lab to establish the quality improvement realized at each setting. In the lab tests were performed to determine the Free Fatty Acid content, by titration, for each sample. Each sample was also processed through a lab centrifuge to determine how much material still remained in the oil. Studying these variables allowed the research team to identify the ideal operating parameters for the system. Analysis of the data showed that a feed rate of approximately 20 gallons per hour with the centrifuge operating at approximately 3300 revolutions per minutes achieved the highest quality oil. A copy of this report can be found in Appendix D.

Implementation of the Methanol Recovery Test Bed System was also completed and significant testing was performed. Using the initial design the system performed as planned and was able to remove methanol from glycerin to a level that rendered the glycerin safe for disposal however the overall time required for this was still significant, approximately 20 hours. Through analysis of the system design, the decision was made to add a supplemental heating tape to the main circulation pipe to reduce the overall time required to heat the system to recovery levels. This heating tape was purchased and installed. The electronic control system was modified to include the supplemental heater and the new system was tested. The addition of the heat tape caused the system to perform as expected. Overall heating to the point of recovery time was reduced dramatically and overall time required to recover enough methanol to achieve safe glycerin properties was also significantly reduced. The required time to run the system fully was reduced from approximately 20 hours to about 12 hours and the overall energy required to perform this reduction was fairly minimal.

The final construction and assembly of the biodiesel test bed system was completed through the installation and programming of the PLC system. The centrifugation system was tested at the optimal system settings that were identified to verify the results. Further testing was performed with the methanol recovery test bed to verify operating parameters. Design improvements were considered for the methanol recovery system to establish how to further reduce overall recovery time. These considerations included operating the system under vacuum settings, increasing the surface area of the system to allow for high evaporation rates and the use of engineered nozzles to facilitate flash evaporation as the glycerin circulates through the system.

3. **Explanation of Variance:** There is no variance between the planned activities and the actual accomplishments for this task during this quarter. It should be noted that the proposed completion date for this task was changed from January 5, 2009 to April 1, 2011. This allowed for adequate time to complete the test bed design and install all necessary hardware and instrumentation to the test bed. This delay was based on delay from previous tasks that have not allowed the test bed design to be completed.

Phase 4 – Processor/Process Optimization

H. Process Optimization

1. **Planned Activities:** The purpose of this task was to demonstrate the capacity for process optimization based on the literature review and best practice benchmarking. Using chemicals, feedstock, and process improvements identified through this research, analysis was done to determine which combinations improve biodiesel production, fuel quality, and efficiency of the process. Variables such as cost, time, safety, energy yield, waste management, and environmental impact were the criteria considered to analyze process optimization. The activities associated with this task included:
 - a. Development of an experimental test plan for process design improvement
 - b. Development of prototype designs to improve the production process
 - c. Testing and verification of prototype design processes
 - d. Identification and evaluation of quality assurance test protocols.

The key milestone for this task was the fifth project deliverable of a production process able to produce biodiesel that meets ASTM D 6751 quality requirements.

2. **Actual Accomplishments:** Research into the optimization of the production process has taken place using all test bed prototypes and lab testing. Previously produced samples have been tested in accordance with ASTM D6751 using the new lab instruments. To date samples tested have met the ASTM D6751 requirements for Water and Sediment, Flash Point and Cloud Point. Limited results have been achieved in Total and Free Glycerin and Acid Number testing due to calibration issues. As these issues were worked out, testing continued to determine the quality of previously produced samples. The research team also completed a study to compare the quality of biodiesel

to the catalyst used in production. Research was done to compare biodiesel created using Potassium Hydroxide and Sodium Hydroxide catalysts. For each catalyst three different samples were produced: one with a minimal catalyst, one with a precise catalyst and one with excessive catalyst. As these samples were completed testing was done to determine their quality in accordance with ASTM D6751. A copy of the final presentation of this testing can be seen in Appendix D.

Testing was completed on the previously mentioned catalyst study. Biodiesel samples were created from both Potassium Hydroxide and Sodium Hydroxide. Methanol was the alcohol used to produce all samples. These samples were created in a lab setting using a hot plate, stirring plate, separatory funnels, and standard glassware. The oil used to create these samples was a standard soy bean based cooking oil. The oil was fresh and had not been used for cooking previously. By using fresh oil the team was able to maintain a consistent low free fatty acid level in the oil. Each sample batch was produced in exactly the same manner. Oil was heated to 140 °C on a hot plate. The catalyst and methanol were thoroughly mixed in a separate container until the catalyst was completely dissolved in the methanol. When the oil reached temperature the methanol/catalyst solution was add to the oil and allowed to mixed for a defined period. At the completion of the mixing, the solution was transferred to separatory funnel where the biodiesel and glycerin were allowed to settle for 24 hours. After 24 hours the glycerin layer was drained and measured and a sample of the unwashed biodiesel was removed. These unwashed samples represent the first samples to be tested. The remaining biodiesel was then washed once using 50% water by volume. The room temperature water was added to the biodiesel and mixed for a predefined amount of time on a stirring plate using a magnetic mixing bar. The biodiesel and water were then transferred back to the separatory funnel and allowed to settle. After 24 hours the water layer was removed and another sample of biodiesel was taken to represent a single wash sample. The final washing was performed by once again adding 50% water by volume, mixing and then settling until the wash water that settled was clear. This typically required three additional washes, for a total of 4 wash cycles. After the final settling period the remaining biodiesel was used at the fully washed sample. Please see the list below for a more specific explanation of each sample produced:

Potassium Hydroxide Samples

- K1- Minimal Catalyst (4 grams/Liter), Unwashed Sample
- K2- Minimal Catalyst (4 grams/Liter), Single Washed Sample
- K3- Minimal Catalyst (4 grams/Liter), Fully Washed Sample

- K4- Precise Catalyst (8 grams/Liter), Unwashed Sample
- K5- Precise Catalyst (8 grams/Liter), Single Washed Sample
- K6- Precise Catalyst (8 grams/Liter), Fully Washed Sample

- K7- Excess Catalyst (12 grams/Liter), Unwashed Sample
- K8- Excess Catalyst (12 grams/Liter), Single Washed Sample
- K9- Excess Catalyst (12 grams/Liter), Fully Washed Sample

Sodium Hydroxide Samples

N1- Minimal Catalyst (2.5 grams/Liter), Unwashed Sample
N2- Minimal Catalyst (2.5 grams/Liter), Single Washed Sample
N3- Minimal Catalyst (2.5 grams/Liter), Fully Washed Sample

N4- Precise Catalyst (5 grams/Liter), Unwashed Sample
N5- Precise Catalyst (5 grams/Liter), Single Washed Sample
N6- Precise Catalyst (5 grams/Liter), Fully Washed Sample

N7- Excess Catalyst (7.5 grams/Liter), Unwashed Sample
N8- Excess Catalyst (7.5 grams/Liter), Single Washed Sample
N9- Excess Catalyst (7.5 grams/Liter), Fully Washed Sample

The amount of methanol used to produce each sample was 20% of the volume of the oil used. This value is based on the standard procedure used by the majority of small scale producers as observed in the literature review and best practice research. The catalyst amounts are also based on these findings. For Potassium Hydroxide the standard catalyst concentration for fresh oil is 8 grams/liter. The minimal samples were produced using a 50% reduction and the excess samples were produced using a 50% increase. For Sodium Hydroxide the standard catalyst concentration is 5 grams/liter and the minimal and excess samples were produced using the same concentrations as listed above for the Potassium Hydroxide samples.

After all samples were produced and labeled, testing began to determine the quality of these samples based on the tests our laboratory is capable of performing. Each of the 18 samples was tested for Acid Number, Water and Sediment, Flash Point, Total and Free Glycerin and Cloud Point. Additional testing of these samples included Sulfated Ash and the 27/3 test. The 27/3 test is a common test performed by small scale producers to determine if the transesterification reaction occurred completely. The research team performed this test to determine its validity compared to the ASTM standard test. The results of the testing of these samples can be seen in Appendix D.

The goal of this study was twofold. First, the team wanted to perform a thorough investigation using all of the lab equipment to determine how proficiently it can be used and to bring to light any tests or equipment that required further explanation, calibration, or development. After completing this study it was determined that further work was required to determine if the gas chromatograph is calibrated correctly. After testing all 18 samples, the gas chromatograph detected no free glycerin in any of the samples but did appear to accurately detect the total glycerin in each sample. It is believed that the lack of free glycerin data is incorrect; however it is possible that all free glycerin settled out of the samples. The team worked to verify the gas chromatographs calibration and resolve these issues. The team also struggled to accurately perform the sulfated ash testing. After numerous attempts, the team was unable to produce a test sample with any noticeable sulfated ash. It appears that all of the ash was burning off in the furnace. Further research was also done in the procedure

to test for sulfated ash and the team practiced the method until accurate results were produced.

The second major goal of this study was to establish a base knowledge of the effects of catalyst concentration and water washing on the quality of the biodiesel produced. These are two key aspect of the production process and are believed to have a great effect on the overall fuel quality. The correlations between catalyst concentration and fuel quality as well as the effects of water washing on fuel quality were determined as the data from this testing is analyzed.

Another study was performed to research the critical temperatures and their connection to quality for the methanol recovery system. As previously mentioned, a temperature monitoring system was developed to be used for both biodiesel and methanol recovery process development. For this study the monitoring system was used to track critical temperatures in the methanol recovery system and then correlate these temperatures to the quality of methanol recovered. Thermocouples were placed at several key locations on the system. These locations included three points along the distillation column, one point in the bulk glycerin tank, one point in the head space of the reactor, one point along the cooling water inlet, one point along the cooling water outlet and one point in the cooling water reservoir. These critical temperatures were monitored and recorded over numerous runs of the system. Though the final data is still being compiled and analyzed, initial analysis shows that maintaining a consistently cool temperature of the cooling water greatly increase recovery speed and quality of methanol. This was expected in theory and has been proven by the data. Further correlations and connections will be determined as the data is analyzed and this will be reported next quarter.

Based on the finds of the previously mentioned methanol recovery study, a strong correlation was made between uniformly heating the bulk glycerin supply and consistently recovering high quality methanol. If the bulk glycerin supply is not heated uniformly and circulated well during this process, the resulting methanol recovered drops in quality and overall recovery time increases. A new system was designed and implemented. The newly designed system allowed the methanol laden glycerin to be heated more uniformly and reduced the overall time required to remove the methanol from the glycerin. This system employs an air cooled condenser system rather than the previously used water cooled system. The research team anticipated that this system will reduce the overall time recovered for the methanol recovery process and would also produce high quality methanol that could be reused in the biodiesel production process. The implementation of this system was completed and the analysis of its results can be seen in Appendix D.

Research and development was performed to set up a centrifuge filtration system to test the quality of oil before and after centrifugation and determine how this affects both oil quality and biodiesel quality. Designs were completed for an oil pre-treatment centrifugation system that would allow oil being used for biodiesel production to pass through the centrifuge in the hope of filtering out water and sediment. After the oil had

been processed in the centrifuge it was tested in the lab to determine how the quality was affected by this process. This oil was then be used to produce biodiesel samples which were also tested to determine how quality was affected. A flow meter was installed on the feed tank to allow the research team to experiment with different feed rates to the centrifuge. Work was done to establish the ideal feed rate and centrifuge RPM to establish the highest quality oil and this research was then connected to the overall quality of the biodiesel produced from this oil. This area was being specifically researched as a method to accomplish de-watering/drying of oil as well as filtration. Centrifugation also provides a method for removing gums from crude oils directly after pressing has been completed. A centrifuge was used to filter and purify vegetable oil prior to biodiesel production. The research team tested different feed rates into the centrifuge as well as different rotational speeds for the centrifuge to establish the ideal parameters for filtration. The results of this research are reported in Appendix D.

Centrifuge research continued to establish optimal settings and verify the practicality of the centrifuge system. Research continued to establish the optimal feed rate, heat application, and rotational speed of the centrifuge to achieve the highest quality oil possible. A thorough testing plan was established to test each of these variables and results can be seen in Appendix D.

Test plans was developed to perform studies on alternative feedstocks that are locally available to small scale producers. The addition of the pressing research and capacity to press seeds into oil allowed the team to explore a great range of feedstock crops. This research involved acquiring and testing samples of oil from different locally available feedstock sources and testing both the quality of the oil and the quality of the biodiesel produced. A key aspect of this research was testing oil at different stages of processing which included testing crude oil that has just been pressed, slightly degummed oils, fully degummed oils and waste vegetable oils of different feedstock sources. Some of the sources considered were sunflower oil, canola oil, soybean oil, and corn oil. A result of this research into feedstock use has lead to the realization of the importance of degumming crude oil prior to biodiesel production. The hydration and removal of the phospholipid gums is essential to producing an oil of the quality required for biodiesel production. Research has been performed into how this degumming can be performed and what a small scale degumming system design would look like for local producers that are capable of pressing oil from seeds.

Another aspect of this research that has become significant to the quality of small scale biodiesel quality is the process of pressing oil from seeds. The research team has been made aware of the importance of the pressing process and has partnered with a local individual who performs small scale pressing to establish a base of knowledge in this area. The partnership also allowed the team to receive samples of oil from different local feedstocks for testing purposes. The research team anticipated that small scale pressing would likely become an additional aspect of this research.

The research team worked to install a small scale mobile pressing system for feedstock research, testing and education. This pressing system was completed and a video explaining the system can be seen here:

<http://www.lancasterfarming.com/video/results/Messiah-College-Sunflowers>

The pressing system is mounted in a small box trailer and consists of a 5-ton oil press powered by a 20 HP diesel engine. This engine runs off of biodiesel produced by the research team. Initial testing of the system verified that it functions as designed and that it can successfully expel oil from the sunflower seeds. Further research will be performed to optimize the settings of the press to increase both quality and quantity of oil produced.

The research team was also able to partner with a local farmer to grow approximately 5 acres of sunflowers for feedstock research. This crop was planted in May of 2011 and harvested in early October 2011. The field produced approximately 3 tons of black oil sunflower seeds. These seeds were pressed for oil, the oil was purified to be used for cooking application on campus and the resulting waste vegetable oil was collected and converted into biodiesel. The research team hoped to demonstrate the capacity for a small plot of land to produce both a food and fuel group that can offset the needs of the campus. Currently 75 gallons of oil have been produced in initial testing with the pressing system.

The ability to demonstrate a complete and full circle process of biodiesel production was an important objective for the research team. To demonstrate that a crop can be grown, harvested, and processed into oil, that this oil can be purified for cooking, cooked with and the waste then used to produce high quality biodiesel allows the team to show the practicality of small scale biodiesel systems. The research team believes that a full circle process like that described above has been successfully modeled and can be scaled up to help colleges, business and municipalities offset both food and fuel needs for the community. The team is excited to continue this research and further develop this successful model that can then be applied at other locations.

The team continued making biodiesel using previous prototype processing systems with a very high level of success. All tested prototypes have been found to produce high quality biodiesel using cost effective, accessible, and reasonable simple system designs. These prototypes can be easily constructed and operated by small scale producers and consistently produce high quality biodiesel. A key element to process optimization that has come up has been the development of a streamlined process. As the final systems design was developed, the research team became more aware that a practical small scale biodiesel production system requires a number of process systems all tied together. These include oil pre-treatment and preparation, biodiesel production, washing, drying, and methanol recovery. Without a single streamlined process that includes at minimum the previously mentioned systems, the user or customer will be left unable to completely produce high quality biodiesel and to process the resulting by products in a manner that allows for recycling of methanol and safe disposal or use of

glycerin. As the team moved forward in optimizing the process of small scale production, the reality that a truly effective system requires a number of processes to be combined into a single streamlined system became more apparent.

As research continued with the centrifugation, biodiesel, and methanol recovery test beds, significant progress was made in the area of Process and Processor Optimization. Major strides were taken in the development of the previously mentioned test beds. This progress has continued to verify the feasibility of a streamlined, small scale biodiesel production system. This system begins with a bulk oil storage area. In this area, incoming waste vegetable oil will be stored and allowed to settle. Currently this is occurring in a 350 gallon IBC tote. Oil is off-loaded directly into the tote and the water, sediment and other materials settle out of the oil prior to entering the centrifuge system. To improve the settling process, especially through the colder months of the year, an in tank heat was added to the tote. The purpose for this heater is to minimally increase the temperature of the waste vegetable oil in order to reduce the viscosity and allow for more rapid separation by gravity of water and contaminants. Once the water and contaminants settle out of the oil to the bottom of the tank, this material is removed through a pipe that is used to draw from the very bottom of the tank. Using a vacuum collection system this material is removed from the tank and disposed.

The next station in the streamlined process is the centrifugation system. Oil is pumped from the bulk storage tote to an elevated holding tank. Oil then is feed to the centrifuge at a controlled rate. As the oil moves into the centrifuge it is heated to again reduce its viscosity and allow for water and sediment to more readily be removed. After passing through the centrifuge the oil is visible cleaner. At this point, the centrifuged oil is stored in a clean oil tote. In the future, after research has been completed, the oil will feed directly from the centrifuge to the heating and mixing tank of the biodiesel processor. One of the key benefits to this connection is that the oil leaving the centrifuge is pre-heated and will reduce the overall time required to heat the oil prior to biodiesel processing.

Once the oil is processed into biodiesel and allowed the settle, the glycerin is then pumped directly into the methanol recovery system. As stated above, this is currently not being done due to the testing each systems involved. Connecting the glycerin output directly to the methanol recovery systems will improve the safety and cleanliness of the system overall. It will also likely improve the quality of the methanol recovered from the system, as the glycerin will not be exposed to the humidity in the air for a long period of time. As recovery occurs, the methanol could in theory be feed directly back to the methoxide tank and be ready for the next batch of biodiesel to be produced.

The goal of creating a streamlined small scale system was to reduce the redundancy of pumps on systems, reduce the overall energy required to produce biodiesel and purified glycerin and create the safety and highest quality system possible.

The streamlined small scale system was thoroughly tested and used in daily operation with great success. Through settling in the bulk storage tank and then being filtered

through the centrifugation system, the overall quality of the waste oil, prior to biodiesel production, was dramatically improved. This improved quality of feedstock has also improved the quality of the resulting biodiesel and has reduced the time and volume of water required for washing this product. The team has also processed all of the waste glycerin to remove the excess methanol. Using the methanol recovery test bed, the team has been able to recover both high volumes and high purity methanol from the waste glycerin. This has two major benefits to the project, the first being that the glycerin can be disposed of safely and second that the recovered methanol can be reused in the biodiesel production process. All recovered methanol is being reused in biodiesel production. The streamlined small scale system has allowed the research team to extensively test and optimize each stage of the process to achieve the highest levels of quality, safety, and repeatability in the processes involved.

Work continued to complete the experimental test plan for design improvement. The catalyst study results were analyzed and conclusions can be seen in Appendix D. The resulting correlations and conclusions can be seen in the report in Appendix D. Results of the centrifuge study were also analyzed and can be seen in the report in Appendix D. Additional studies began to help enhance the production process and the results of these studies were used to develop prototype designs for quality enhancement in the production process. As these designs were developed and implemented the resulting products were tested to establish quality in accordance with ASTM D6751. These tests allowed the design to be verified or redeveloped for implementation. The implementation of the newly designed methanol recovery system was completed and testing and analysis were done to compare its performance to previous systems. The results of this analysis can be seen in the report in Appendix D. Further research was done with the centrifuge system to determine its effects on process improvements. The team also continued research into the significance of small scale seed oil pressing and its effects on the biodiesel production process.

3. **Explanation of Variance:** There is no variance between the planned activities and the actual accomplishments for this task.

Processor Development

1. **Planned Activities:** The purpose of this task was to demonstrate the capacity for processor optimization based on the literature review, best practice benchmarking, and process optimization work. Using processor hardware improvements identified through this research, analysis was done to determine which combinations improve biodiesel production, fuel quality, and efficiency of the process. Variables such as processor tanks, piping, and valve size, instrumentation and controls, construction techniques and fabrication materials were the criteria considered to analyze process optimization. The activities associated with this task included:
 - Development of an experimental test plan for processor design improvement
 - Development of prototype designs to improve the processor

- Testing of prototype design processors and verification of the repeatability of this processor to produce biodiesel fuel that meets ASTM D 6751 quality specifications.

The key milestone for this task will be the sixth project deliverable of a production processor able to produce biodiesel that meets ASTM D 6751 quality requirements.

2. **Actual Accomplishments:** Based on all of the research, testing, and development performed over the course of the project, a final processor design has been developed and constructed. This processor, as discussed above, is a streamlined system which includes a centrifugation system for oil purification, a biodiesel processing system for fuel production and a methanol recovery system for glycerin processing. The heart of the design for each system is embodied in the test bed system designs for each respective system. The current centrifugation system includes a bulk storage tote with an in tank heater, an elevated holding tank for oil to be feed to the centrifuge, and a variable speed Raw Power Centrifuge allows for a continuous feed of oil and heats the oil as it enters the system. In the final processor system design, the oil feeds directly from the centrifuge into the 35 gallon stainless steel biodiesel reactor. This reactor is controlled by a Programmable Logic Controller and measures the oil level and shuts off the feed once the tank is full. The research team has worked to determine how the system will establish the Free Fatty Acid Content of the oil automatically. This created a very complex system and it is easier to perform this test manually. The processor then continues to heat the oil until the appropriate temperature is reached and then mixes the methanol/catalyst solution with the heated oil. After a period of circulation the glycerin is allowed to settle for a period and then it is pumped out of the processor into the methanol recovery system. The remaining biodiesel then run through 3 washing cycles, with the wash water being removed from the system after each cycle. After washing is completed, the biodiesel is dried and ready to use. The operation and maintenance manual for this system can be seen in Appendix D.

The methanol laden glycerin that was pumped from the biodiesel processor system is then processed in the methanol recovery system to remove the excess methanol. This recovered methanol is tested to verify its quality and is then returned to the biodiesel processor system for use in future batches of biodiesel. The remaining glycerin, which is tested to ensure that the appropriate amounts of methanol have been removed, is then disposed of accordingly.

Throughout the course of this project the research team recognized the need to develop a biodiesel processor that met all of the needs of a small scale producer. This cannot be accomplished without a streamlined system that is able to first produce a high quality waste vegetable oil product, then a high quality biodiesel product and finally a means to process the hazardous methanol laden glycerin that results. Though complex, this system will truly meet the needs of a small scale producer and the estimated payback period on the system is relatively low, most likely about 2 to 3 years according to the capacity of the producer.

Each component of this streamlined system has been thoroughly researched, tested, and improved. Moving forward each system will continue to be tested to verify its performance and then the systems will be combined to create one overall streamline system. This completed system will then be tested to insure that it performs as anticipated.

The streamlined small scale system defined above was assembled and tested thoroughly with excellent results. At this point the system is still functioning as a test bed, allowing the research team to manipulate each independent system and verify its design.

As the implementation of the new test bed design and the new prototype biodiesel production processor have been completed and the test plan has been executed, design changes have been made and quality assurance and verification have been performed. The result of this work is a small scale design for a biodiesel production system able to produce high quality biodiesel in accordance with ASTM D6751. Coupled with this processor development was the development of the small-scale oil pretreatment system and the methanol recovery processor design, testing and quality assurance. As stated in the previous task, the research team feels that a final processor design that does not include at least oil pretreatment and methanol recovery is an incomplete design that will not serve the consumer or end user well.

The research team continued to test the final processor designs function and also specifically focused on testing the biodiesel produced from the systems to verify that it meets ASTM D 6751. All preliminary research and testing to data show that the overall system produces biodiesel that exceeds the specifications required by this standard.

3. **Explanation of Variance:** There is no variance between the planned activities and the actual accomplishments for this task because no major accomplishments have occurred during this quarter. The final processor system design has been completed and research, testing and r development have been completed.

Project Management and Reporting

1. **Planned Activities:** The purpose of this task was to promote the practices of sound project management and reporting and to disseminate reports and information to benefit current and future small scale biodiesel producers. The activities associated with this task included:
 - Collaboratory project management and reporting
 - Federal reporting
 - Summary dissemination.

The key milestone for this task will be the seventh, and final project deliverable of the multiple reports that will be required over the course of the project period.

2. **Actual Accomplishments:** Student researchers have worked to meet the requirements for the Collaboratory and Integrated Project Curriculum project management and reporting. Weekly project management task sheets have been submitted to advisors and the Wiki has been maintained. Federal reports have been completed and submitted to meet the required completion dates. The project budget has been thoroughly maintained and project spending has been tracked. All requirements of the Federal Assistance Reporting Checklist have been met to date. During the time spent presenting at the Pennsylvania Farm Show and through the “Messiah College Biodiesel Research Project” brochure that was created and disseminated for the research project, knowledge and information to date have been shared. A copy of this brochure can be seen in Appendix B.1. A list of individuals interested in receiving future reports, publications or other forms of dissemination is being compiled to use in the future.

3. **Explanation of Variance:** There has been no variance between the planned activities and the actual accomplishments for this task to date.

IV. Summary of Project Activity

The objectives for this project were to:

1. Equip a laboratory for developing and optimizing processes and processors for small scale biodiesel production;
2. Develop a scalable process and processor design suitable for ASTM certifiable small-scale biodiesel production; and
3. Identify quality enhancement and cost reduction protocols for process and processor design using feedstock and production ingredients readily available to small-scale producers.

The project outcomes for this project were:

1. A laboratory to enable ongoing research and development in support of entrepreneurs, municipalities, and others pursuing ASTM certifiable small scale biodiesel production in our region;
2. Documentation of best practices for ASTM certifiable small scale biodiesel production; and
3. Scalable process and processor design based on new research into optimizing design variables for quality assurance and cost.

This project has met these objectives and outcomes by establishing a laboratory facility and a research and development program that has advanced biodiesel processor and process design in support of small-scale producers. This work included researching the ability to produce ASTM certified fuel using various feedstocks, including recycled or unconventional materials, and production ingredients. The project has also developed scalable processor and process designs suitable for ASTM D 6751 certifiable small-scale biodiesel production.

Quality testing and assurance is one of the most critical issues related to small-scale biodiesel production. The process of making biodiesel is reasonable simple but the ability to prove that this fuel meets industry standards to ensure that it will not have negative impacts on systems in which it is used is much more difficult. Few, if any, small-scale producers have access to the laboratory equipment required to perform quality tests for biodiesel and these producers cannot afford to pay for samples to be tested by commercial laboratories. Biodiesel that is being produced by small-scale producers often has no formal testing performed on it and its quality is never verified. The research team at Messiah College was able to develop a small-scale biodiesel quality testing laboratory in which it performs key tests from the ASTM D 6751 (Include full name of document) Biodiesel Quality standard. The research team spent a significant amount of time researching the most critical tests for small-scale biodiesel produces and these five were chosen: Total and Free Glycerin by Gas Chromatography, Flash Point, Cloud Point, Acid Number, and Water and Sediment (Include ASTM numbers and names). All equipment and testing procedures used in this laboratory are in accordance with the ASTM D 6751 standard for biodiesel fuel.

This laboratory set the stage for the research team to then move on to process and processor research. With the ability to verify the quality of the product, the team then investigated the processes as well as the processor technology associated with small-scale biodiesel production. The specific processes and processor technology considered were: waste vegetable oil pretreatment, transesterification, washing, drying, and waste glycerin processing. The research team performed significant research to establish the best practices in small-scale biodiesel

production for these areas. This research then led to the design and construction of test bed processors in which these processes could be performed and the results tested for quality. Using the test bed processor, the parameters of each specific process could be controlled and modified to allow the research team to explore which produced the highest quality product.

Messiah College personnel have gained significant working knowledge about biodiesel production by designing and testing small scale systems that produce biodiesel from waste cooking oil collected from the College's campus dining facilities. The College uses this biodiesel in unmodified diesel engines and as a supplement to heating fuel oil. This project has enabled College personnel to extend their work into applied research and development that supports future commercial applications of small-scale biodiesel production. The academic work of this project has supported entrepreneurs and institutions in our region seeking to plan small business entrepreneurial ventures in biodiesel production.

The cycle of biodiesel production at Messiah College begins with the dining facilities purchasing oil to be used for cooking and frying applications. This oil is used in three different dining facilities until it is no longer of an acceptable quality for cooking. The oil is then disposed of in storage containers at each respective dining facility. At this point the biodiesel research team collects the oil and transports it to the biodiesel production facility for processing. The oil is deposited into a storage and settling tank to allow water and sediment to begin to settle out of the waste oil. The storage and settling tank is also equipped with a heater that is used during the colder months of the year to reduce the viscosity of the oil to allow for settling of water and sediment. Oil is then pumped from the upper portion of the storage and settling tank to be processed through the centrifuge filtration system. This oil is pumped up to a holding tank above the centrifuge. Oil is then fed by gravity into the centrifuge where it is heated and spun to remove any remaining water and sediment. The oil flowing from the centrifuge is then stored in a clean oil tank.

Oil from the clean oil storage tank is then transferred to the heating and mixing tank of the prototype processor. In this tank the oil is heated to 60 degrees Celsius by circulating the oil over a heating element for approximately 1 hour. As the oil is circulating a sample is taken for titration. This sample is titrated and the resulting titration number is used to determine the quantity of catalyst required. A thermostat turns off the heater when the oil reaches the set point temperature. During the heating cycle the methanol and catalyst are mixed in the Methoxide tank. Using a potassium hydroxide catalyst, the catalyst will naturally dissolve in the methanol. If a sodium hydroxide catalyst is used, the methanol catalyst mixture will require mechanical agitation or mixing. After the catalyst has completely dissolved in the methanol and the oil has reached temperature, the two are mixed and circulate for about an hour. After the methoxide/oil solution has mixed thoroughly, the circulating pump is turned off and the solution is allowed to settle. The solution has reacted into biodiesel and glycerin. The more dense glycerin will settle to the bottom of the tank over time. The more time the solution is allowed to settle, the more glycerin will settle out. The methanol laden glycerin is then drained and stored for methanol recovery later. The remaining biodiesel is pumped from the heating and mixing tank to the washing and drying tank. The biodiesel is now sprayed with water in a fine mist. The water settles through the biodiesel and washed out soaps and remaining sediment. The washing cycle occurs three times to ensure that all soap, catalyst, methanol and

sediment are washed out of the biodiesel. After washing is complete, a drying cycle begins in which air is bubbled through the clean biodiesel to dissolve all remaining water. At the completion of the drying cycle a sample of each batch of biodiesel is taken to be tested for quality prior to using the biodiesel. After the sample passes all necessary quality tests, the batch is then used in either heating or fuel applications.

After the glycerin settles out of the biodiesel it is drained off and taken to the methanol recover processor. In this process the methanol laden glycerin is pumped into a stainless steel processor in which it is heated by an in tank heater as well as a line heater to heated the glycerin above the boiling point of methanol, approximately 60 degree Celsius. As the glycerin cycles through the system the methanol begins to boil and the vapor rises into an air-cooled condensing column. As the methanol vapor condenses back to a liquid it travels through the column into a recovered methanol storage container. Each liter of recover methanol is tested for quality and if the quality is high enough the methanol is reused in the biodiesel production process. The remaining glycerin is also tested for flash point to ensure that all remaining methanol has been removed. Once the methanol meets an acceptable flash point it is then disposed of by composting.

Numerous testing iterations for each process have lead to there determination of the ideal parameters to produce high quality biodiesel for each process were established and the research team then began to develop processor technology able to replicate these processes. The result of this work was the development of the following: a centrifuged system for the pretreatment of waste vegetable oil, a manual and automated biodiesel processor, and an automated methanol recovery system. Though independent systems, each of these processors is required to create a complete small-scale biodiesel production system. Though each processor can function on its own, the complete system requires that all three of these processes be performed for a sustainable system to be established.

The final key area the research team focused on was the education of the community about biodiesel as a fuel in general and more specifically about the opportunities for small-scale biodiesel fuel production applications. The process of educating the community came in many forms. The initial and most obvious was working with the undergraduate students on the research team and their peers as they performed their research. These students performed the majority of the research, testing, design and development associated with this project and were required to present on their research multiple teams each semester. The educational program grew from here to include the faculty, staff and students of Messiah College as a whole. Members of the research team were involved in presentations, panel discussions, and sustainability festivals across the campus. Branching out further, the research team developed an exhibit for trade shows and engaged approximately 1500 individuals each year through the attendance of trade shows to present the work being done at Messiah College. The connections built at these trade shows lead to opportunities for presentations, workshops and trainings for community groups, professional organizations, and public and private middle schools and high schools. Throughout the three-year project period the research teams engaged with thousands of individuals about biodiesel in general as well as the specific work being done at Messiah College.

Messiah College Biodiesel Fuel Generation Project
DE-FG36-08GO88068

This education of the community was geared specifically to each audience being addressed, however a general informational brochure was produced and distributed at all events. This brochure is attached in Appendix B.1. A copy of a typical presentation is also attached in Appendix B.2. A copy of an “Introduction to Biofuels” presentation is also included in Appendix B.3. A history of biodiesel at Messiah College presentation is included in Appendix B.4. The goal of these education programs was to create a community that is more aware of what biodiesel is, how it is made, how it can be used, why it is important, and what can be done locally.

The culmination of the research performed over the course of this project led to two major accomplishments. The first was the design, development, construction, and testing of the following prototype systems:

Manual Biodiesel Processor- See Appendix D.4.

Methanol Recovery- See Appendix D.1, D.2, D.6, D.9.

Seed Oil Pressing- See Appendix D.10.

Automated Biodiesel Test Bed Processor- See Appendix D.11.

Centrifuge Filtration System- See Appendix D.5 and D.8.

The second major accomplishment was the development of a full circle process in which a crop of sunflowers was grown, harvested, processed, pressed into vegetable oil, purified for cooking, and finally the waste is collected and processed into biodiesel. This project is unique because it meets both the food and fuel potential for the crop and shows how a relatively small plot of land can be used to offset cooking oil and diesel fuel significantly.

Technology Transfer Activity

a. Publications

1. The Collaboratory Helps Fuel Pennsylvania's Future- This is an article, written by Messiah College, about the projects involvement with the Pennsylvania Farm Show. See Appendix C.1. for article or the link below.

http://www.messiah.edu/features/spring_2009/collaboratory_biodiesel.html

2. Biodiesel Fuel Production and Methanol Recovery- This article was written for the Fall 2009 Department of Engineering Newsletter at Messiah College. See Appendix C.2. or the link below.

http://www.messiah.edu/departments/engineering/news/EngrNewsFall09_000.pdf

3. Messiah Engineering Students Educating the Community About Biodiesel- This article was written for the Fall 2010 Department of Engineering Newsletter at Messiah College. See Appendix C.3. or the link below.

<http://www.messiah.edu/departments/engineering/news/EngrNewsFall10.pdf>

4. Automated Biodiesel Processor- This article was written for the Fall 2011 Department of Engineering Newsletter at Messiah College. See Appendix C.4. or the link below.

<http://www.messiah.edu/departments/engineering/news/EngrNewsFall11.pdf>

5. College Grows Sunflowers for Food and Fuel- This article was published on 10/15/11 in the Lancaster Farming Newspaper. See Appendix C.5. or the link below.

<http://www.lancasterfarming.com/-College-Grows-Sunflowers-for-Food-and-Fuel->

The following video was included with the online version of this story:

<http://www.lancasterfarming.com/video/results/Messiah-College-Sunflowers>

6. Flower Power: Messiah takes sunflowers from field to fuel- This article was written for the Fall 2011 version of The Bridge, the Messiah College Alumni Newsletter. See Appendix C.6. or the link below.

http://www.nxtbook.com/nxtbooks/messiah/bridge_2011fall/#/18

It also included the following video in the online version of the article:

<http://www.youtube.com/watch?v=Ok4OwF-f5Hc>

7. Sunflowers key part of Sustainability Project- This article was published in November of 2011 by the National Sunflower Association Magazine, highlighting the use of sunflowers in the biodiesel research project at Messiah College. See Appendix C.7. or the link below.

<http://www.sunflowernsa.com/magazine/details.asp?ID=771>

The same article was also published in the February 2012 edition of the American Agriculturalist magazine:

<http://magissues.farmprogress.com/AMA/AM02Feb12/ama013.pdf>

8. Messiah College used flower power to save money, environment- This is a summary of a news story by ABC27 out of Harrisburg, Pennsylvania, featuring the Messiah College biodiesel project and it's work with sunflowers. The story aired on July 21, 2011. See Appendix C.8. or the link below.

<http://www.abc27.com/story/15127221/messiah-college-tries-first-ever-full-circle-biodiesel-project>

9. Sunflowers Make an Impact- This article was written for the September 2011 version of the Swinging Bridge, the Messiah College student newspaper. See Appendix C.9. or the link below.

<http://www.messiahsb.com/news/sunflowers-make-an-impact-1.2616954#.Ty0giBWYV1w>

10. A Look into Messiah College's Green Revolution: College Raising Bar for Sustainable Campuses- This is a summary and video of a news story by FOX 43 out of Harrisburg, Pennsylvania, featuring the Messiah College biodiesel project and it's work with sunflowers. This story aired on July 26, 2011. See Appendix C.10. or the link below.

http://www.fox43.com/news/cumberland/wpmt-messiah-college-goes-green-20110726_0.678477.story

11. Fuel of the Future- This article is from the February version of the Swinging Bridge, the Messiah College student newspaper. It's discusses the use of sunflowers to fuel the future of the college. See Appendix C.11. or the link below.

<http://www.messiahsb.com/fuel-of-the-future-1.2432071#.Ty0hxxWYV1w>

12. Commercial Opportunities for Messiah College Biodiesel-“An alternative energy source for third world countries, the education market and small scale biodiesel development are three markets where this Messiah project has delivered value. This presentation by Mike Zummo was shared with a small, select group of local investors.” This is a video recording of a presentation I gave for Innovation Transfer Network Angel Investors about the possibilities for commercialization of small scale biodiesel processors.” See Appendix C.12. or the link below.

<http://www.innovationtransfernetnetwork.org/itnvideos/viewvideo/80/itn-videos/commercial-opportunities-for-messiahs-biodiesel>

13. Fried foods can be good for you- This is a brief story and podcast interview with Mike Zummo about how Messiah College is working with small scale biodiesel production.

<http://pennfuture.libsyn.com/webpage/fried-foods-can-be-good-for-you>

b. Web site or other Internet sites that reflect the results of this project

Please see the previous section for web sites with information about this project as well as the link below:

http://www.thecollaboratoryonline.org/wiki/Biodiesel_Project

c. Networks or collaborations fostered

The following organizations were part of the network and/or collaborators associated with this project:

Innovation Transfer Network
Collaboratory Energy and Sustainability Group
Mechanicsburg Middle School
Mechanicsburg Environmental Club
The Yellow Breeches School
Harrisburg SciTech High School
Harrisburg Math and Science Academy
The Milton Hershey School
Harrisburg University of Science and Technology
Harrisburg Area Community College

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The Green Center of Central Pennsylvania
Dickinson College
The American Society of Material
The American Society of Chemical Engineers
The Upper Allen Fire Department

Conferences, Meetings, Workshops, and Trade Shows Attended

2009 Pennsylvania Farm Show – Exhibit in the Pennsylvania Renewable Energies Area

2009 Sustainable Biodiesel Summit- San Francisco, CA

2009 National Biodiesel Board Conference- San Francisco, CA

2009 Biomass Platform Review- Presentation of project work to platform review committee for peer review and feedback.

2009 Carlisle Greenfest – Biodiesel exhibit for community members.

2009 Intercollegiate Biodiesel Conference at Dickinson College- This conference featured the mobile biodiesel processor and the research team presented work in a panel discussion about biodiesel on college campuses.

2009 Pennsylvania Bio-Mass Meeting by the Central PA Conservancy- The research team helped to organize this meeting and also present on the biodiesel work being done at Messiah College

2010 Pennsylvania Farm Show– Exhibit in the Pennsylvania Renewable Energies Area

2010 Pennsylvania Biodiesel Development Conference

2010 Biodiesel Symposium at Mechanicsburg Middle School- The research team assisted in planning and presenting at this event to educate local middle school students about biodiesel.

2010 Sustainability Symposium at Penn State School of Hospitality Management

2010 Carlisle Greenfest – The research team presented an exhibit to educate the community about biodiesel.

2011 Pennsylvania Farm Show– Exhibit in the Pennsylvania Renewable Energies Area

2011 Harrisburg University of Science and Technology Bioenergy Summit- The research team led a breakout session on food grain based biodiesel and reported results from the break out session to the entire summit.

d. Technologies/Techniques

Appendix D.1. – 2009 Methanol Recovery Report

Appendix D.2. – 2009 Methanol Recovery Seed Assistance Grant Report
(Includes 2010 Methanol Recovery Manual)

Appendix D.3. – 2010 Biodiesel Final Report

Appendix D.4. – 2010 Biodiesel Production Manual

Appendix D.5. – 2011 Centrifuge Testing Report

Appendix D.6. – 2011 Methanol Recovery Report

Appendix D.7. – 2012 Biodiesel Production Manual

Appendix D.8. – 2012 Centrifuge Manual

Appendix D.9. – 2012 Methanol Recovery Manual

Appendix D.10. – 2012 Pressing Manual

Appendix D.11. – 2012 Automated Processor Manual

Appendix D.12. – 2010 Biodiesel Quality Testing Final Presentation

Appendix D.13. – 2010 Methanol Recovery Final Presentation

Messiah College Biodiesel Fuel Generation Project
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Appendix A. - Photos

Appendix A. - Photos



Photo No. 1: Pensky-Martens Closed Cup Flash Point Tester



Photo No. 2: Heated Oil Test Centrifuge



Photo No. 3: Cloud and Pour Point Bath Apparatus

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Appendix A. - Photos



Photo No. 4: Cloud Point Sample Apparatus

Messiah College Biodiesel Fuel Generation Project
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Appendix A. - Photos

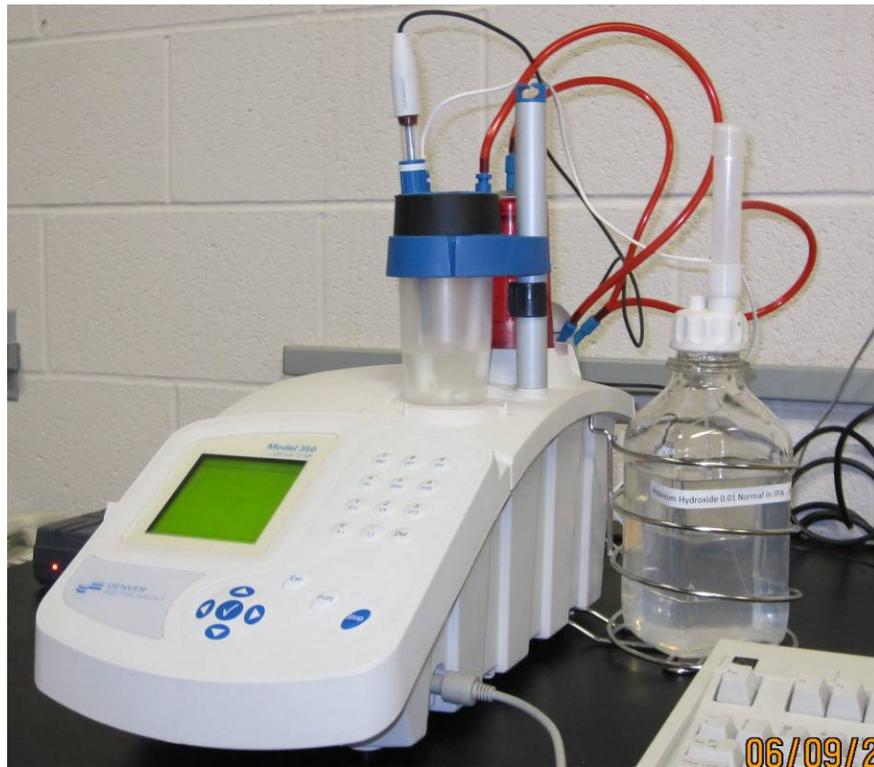


Photo No. 5: Denver Instrument Autotitrator

Messiah College Biodiesel Fuel Generation Project
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Appendix A. - Photos

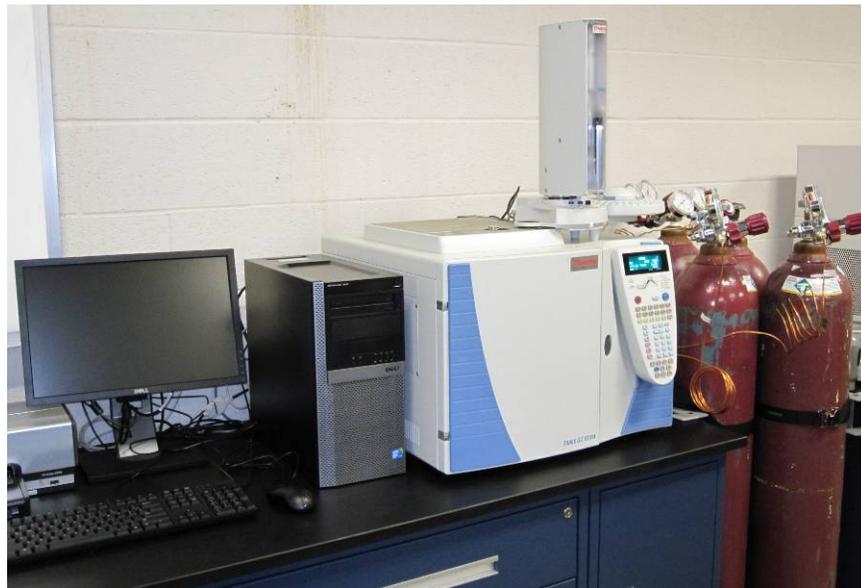


Photo No. 6: Trace GC Ultra with TriPlus Autosampler



Photo No.7: 5-Gallon Prototype Processor Unit

Messiah College Biodiesel Fuel Generation Project
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Appendix A. - Photos



Photo No. 8- 6 Ton Seed Oil Press

Messiah College Biodiesel Fuel Generation Project
DE-FG36-08GO88068
Appendix A. - Photos



Photo No. 9- 40 Gallon Test Bed Reactor Prototype



Photo No. 10- 3 Gallon Test Bed Reactor Prototype

Messiah College Biodiesel Fuel Generation Project
DE-FG36-08GO88068
Appendix A. - Photos



Photo No. 11- 35 Gallon Methanol Recovery Prototype

Messiah College Biodiesel Fuel Generation Project
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Appendix A. - Photos



Photo No. 12- Centrifuge Testing Setup

Messiah College Biodiesel Fuel Generation Project
DE-FG36-08GO88068
Appendix B. – Educational Materials

Appendix B. – Educational Materials

Messiah College Biodiesel Fuel Generation Project

DE-FG36-08GO88068

Appendix B. – Educational Materials

Appendix B.1.

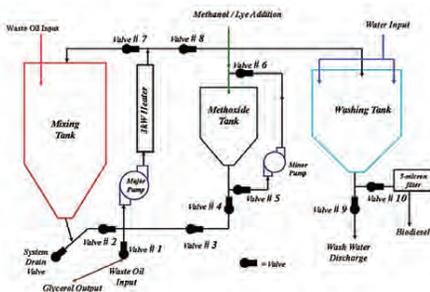
Messiah College Biodiesel Research Project Brochure

Biodiesel Team Research Outcomes

1. A laboratory to enable ongoing research and development in support of entrepreneurs, municipalities, and others pursuing ASTM D 6751 certifiable small scale biodiesel production in our region.
2. Documentation of best practices for ASTM D 6751 certifiable small scale biodiesel production.
3. Scalable process and processor design based on new research into optimizing design variable for quality assurance and cost

Biodiesel Process and Processor

The diagram shown is an exploded view of the "appleseed" biodiesel process that is used by the biodiesel team at Messiah College. After collecting oil at the dining facilities, the oil is filtered and put into the mixing tank on the left. The oil is then heated to 140° F using an inline 3kW heater. While the oil is being heated, methanol and sodium hydroxide (or potassium hydroxide) are mixed together in the methoxide tank according to the titration of the waste vegetable oil. When the oil reaches 140°, the methoxide mixture is pumped into the now hot oil and circulated to mix everything together. After the glycerin has time to settle out of the reaction, the biodiesel is pumped to the washing tank, where it is water-washed to remove any impurities left in the fuel. After a final gravity-fed filtration through a 5-micron filter the biodiesel is finished.



Messiah College Engineering

The mission of Messiah College is to educate men and women toward maturity of intellect, character, and Christian faith in preparation for lives of service, leadership, and reconciliation in church and society. Graduates of the engineering program will therefore be technically competent and broadly educated, prepared for interdisciplinary work in the global workplace. The character and conduct of Messiah engineering graduates will be consistent with Christian faith commitments. We accomplish this mission through engineering instruction and experiences, an education in the liberal arts tradition, and mentoring relationships with students.

www.messiah.edu/departments/engineering/



MESSIAH COLLEGE
DEPARTMENT OF ENGINEERING

Messiah Biodiesel Lab
Messiah College, Box 3034
One College Ave.
Grantham, PA 17027
717.796.1800, ext. 3170
Biodiesel@messiah.edu

the Collaboratory
for strategic partnerships
and applied research

MESSIAH COLLEGE



BIODIESEL RESEARCH PROJECT

Sustainability,
Stewardship, and
Christian Service

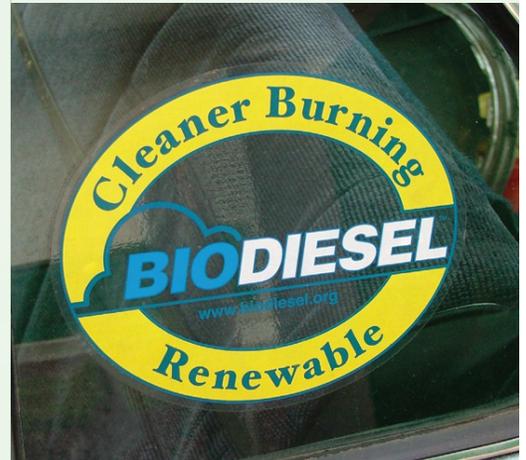
MESSIAH COLLEGE



The Collaboratory for Strategic Partnerships and Applied Research is an organization of Christian students, educators, and professionals affiliated with Messiah College. The mission of the Collaboratory is to partner with organizations, businesses, and communities in our region and around the world for projects in mathematical and information sciences, engineering, and business that serve disadvantaged people and care for the earth; and to develop our members' abilities and vocational vision for lifelong servant-leadership, and the courage to act on convictions.
www.thecollaboratoryonline.org/

What is Biodiesel

Biodiesel is an environmentally friendly alternative to petroleum base diesel fuel for diesel engines and a fuel oil substitute. It is produced by a reaction that occurs when vegetable fats are combined with an alcohol and a catalyst. Its primary advantages are that it is an agriculturally based renewable fuel which is non-toxic and biodegradable. Biodiesel combustion produces less carbon monoxide, unburned hydrocarbons, and particulate emissions than petro-based diesel. It is a closed carbon cycle fuel that emits no net greenhouse gasses. The carbon released during combustion of biodiesel fuel was previously removed from the environment by the oil producing plants.



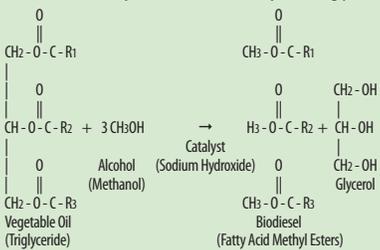
this work has focused specifically on the conversion of waste vegetable oil into high quality biodiesel fuel. With great success, research and development teams have used the waste vegetable oil from campus dining facilities to produce fuel for diesel-powered campus vehicles and as a heating oil substitute. In September 2008 the College was awarded a research and development grant from the United States Department of Energy to promote advances in small scale biodiesel production technology. This grant will provide the biodiesel research team with the ability to pursue ASTM certification testing for biodiesel as well as extensive process and processor research and development opportunities.

Biodiesel Team Research Objectives

1. Equip a laboratory for developing and optimizing processes and processors for small scale biodiesel production.
2. Develop a scalable process and processor design suitable for ASTM D 6751 certifiable small scale biodiesel production.
3. Identify quality enhancement and cost reduction protocols for process and processor design using feedstock and production ingredients available to small scale producers.

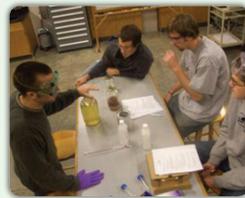
The Basic Reaction

100 pounds of oil + 10 pounds of methanol
 → 100 pounds of biodiesel + 10 pounds of glycerol



History of Biodiesel at Messiah College

For the past five years, student teams have been working in conjunction with the College's Department of Engineering and the Collaboratory for Strategic Partnerships and Applied Research to develop a process and processor systems for the production of biodiesel fuel. Students, faculty, and staff are diligently committed to promoting renewable energy technologies and establishing sustainable sources of fuel to reduce dependence on petroleum based sources. The majority of



Messiah College Biodiesel Fuel Generation Project

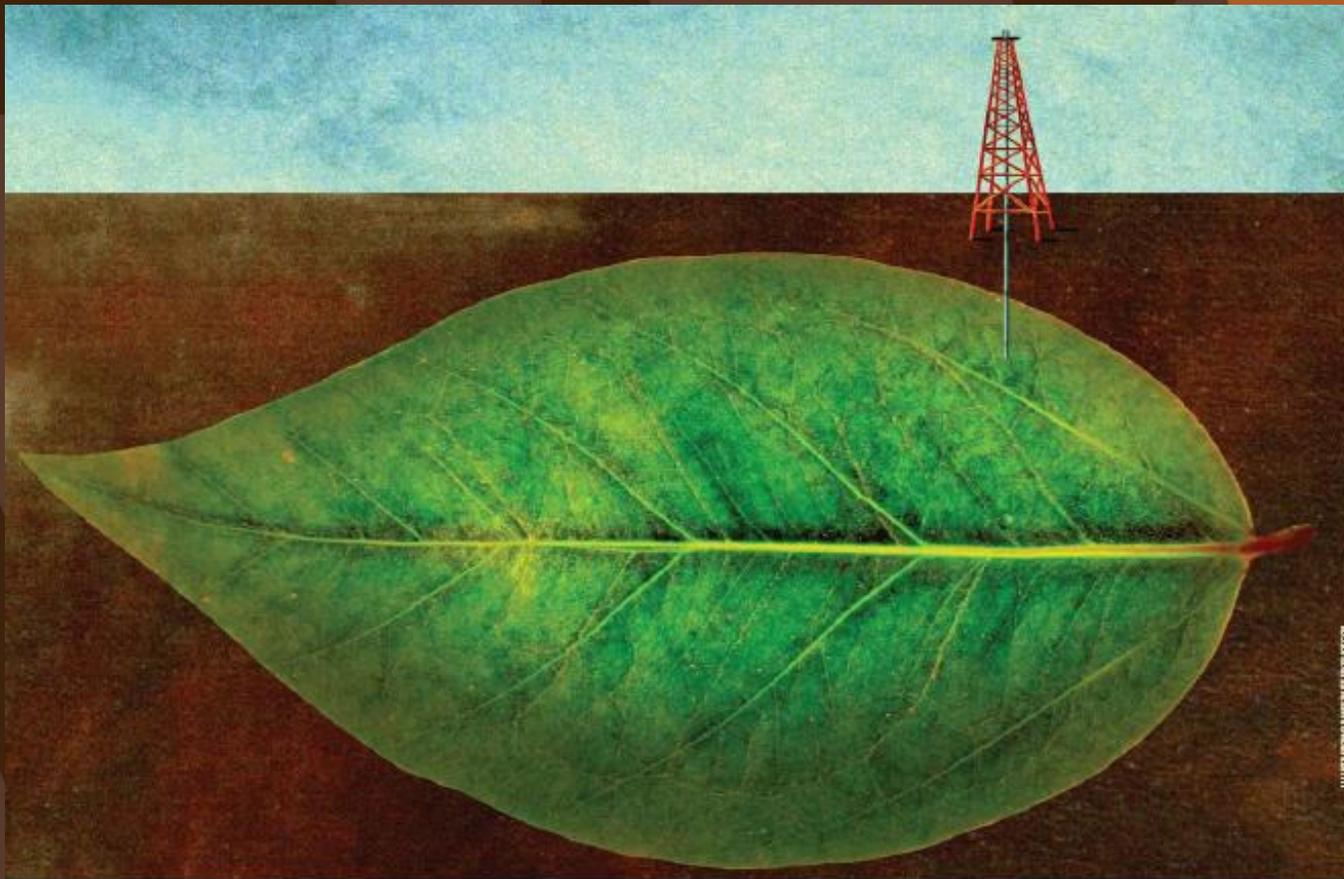
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Appendix B. – Educational Materials

Appendix B.2.

Sample Educational Presentation

Messiah College Biodiesel Research and Development Project



Mike Zummo

4/21/10

the Collaboratory
for strategic partnerships and applied research

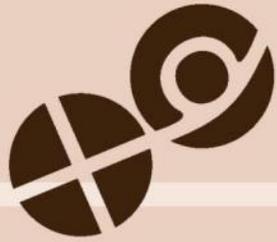


MESSIAH
COLLEGE



What is Biodiesel?

An ***environmentally friendly*** alternative to petroleum base diesel fuel. It is produced by a reaction that occurs when fats and oils are combined with an alcohol and a catalyst. Its primary advantages are that it is an ***agriculturally based renewable fuel*** that is ***non-toxic*** and ***biodegradable***. Biodiesel combustion produces ***less carbon monoxide, unburned hydrocarbons and particulate emissions*** than petro-based diesel. It is a closed carbon cycle fuel that emits ***no net greenhouse gasses***. The carbon released during combustion of biodiesel fuel was previously removed from the environment by the oil producing plants.



From the field to fuel.

Market Use
(Cafeteria Fryer)



Waste Product
(Collection)



Biodiesel
Production
(Small Scale
Processor)

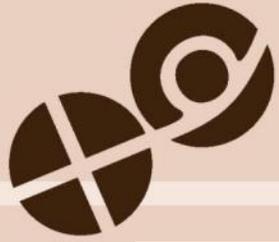


Quality
Testing



End Use
(Campus Vehicles)





It all begins at the farm.





Then moves to the market.





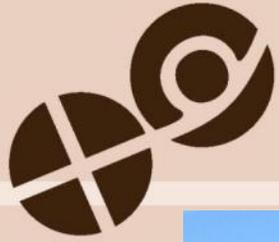
French Fries!





Waste Vegetable Oil





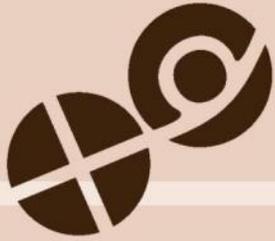
Their waste is our fuel!



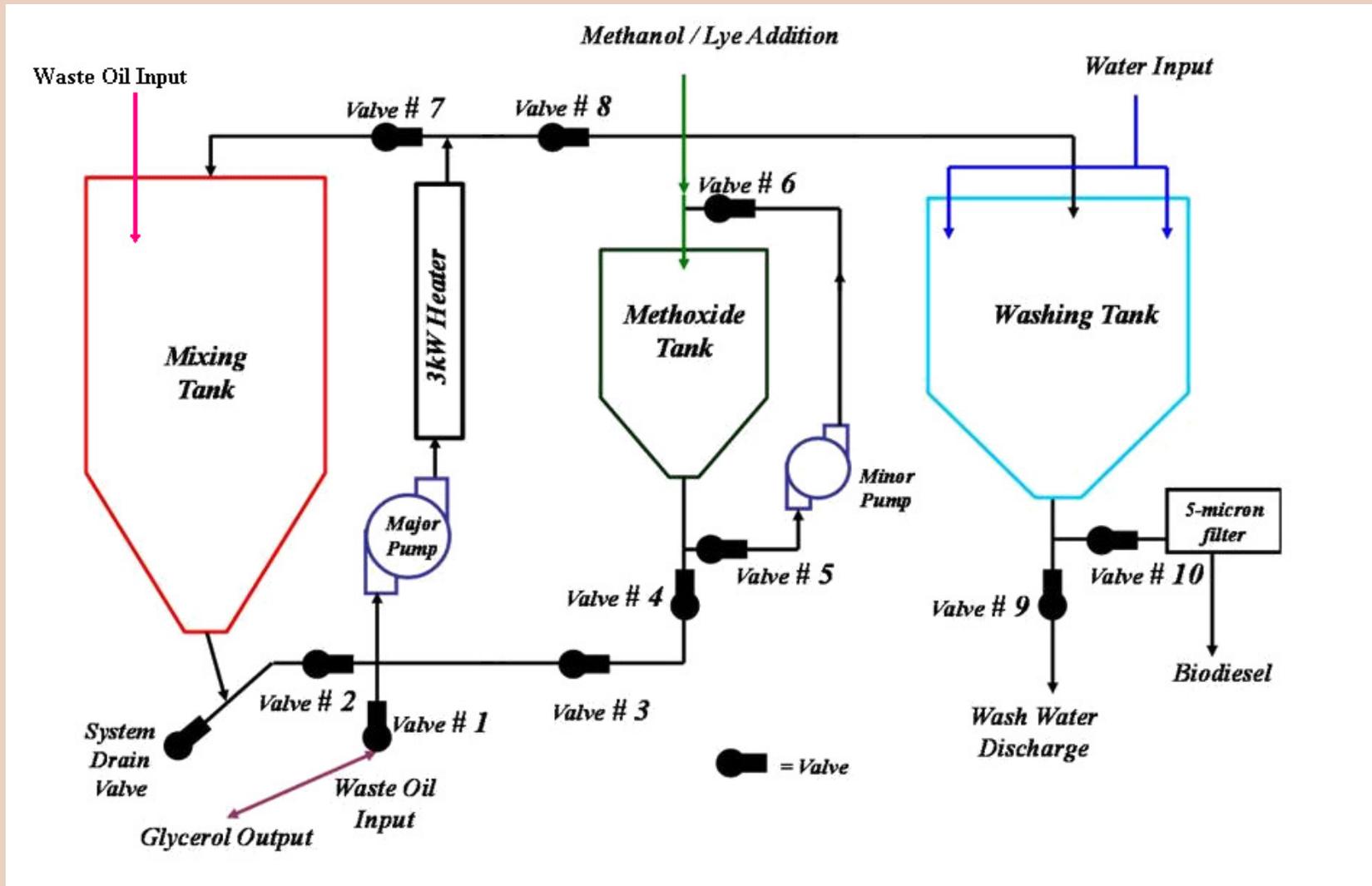


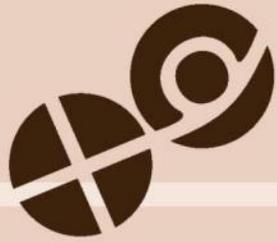
Biodiesel Production





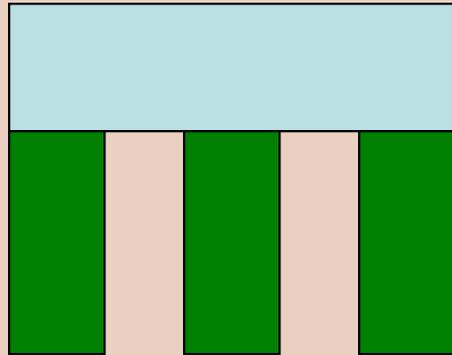
Our Process





Biodiesel Production...

TRIGLYCERIDE



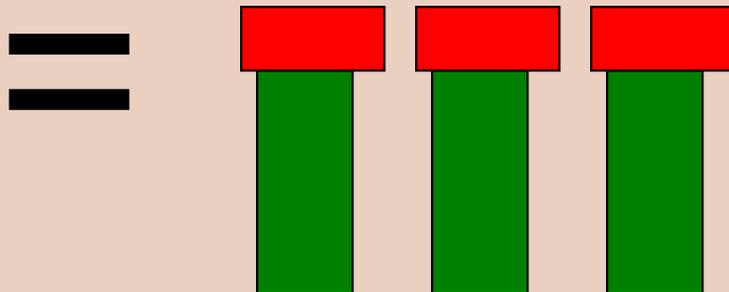
METHANOL



+

Catalyst

FREE ESTERS



(BIODIESEL)

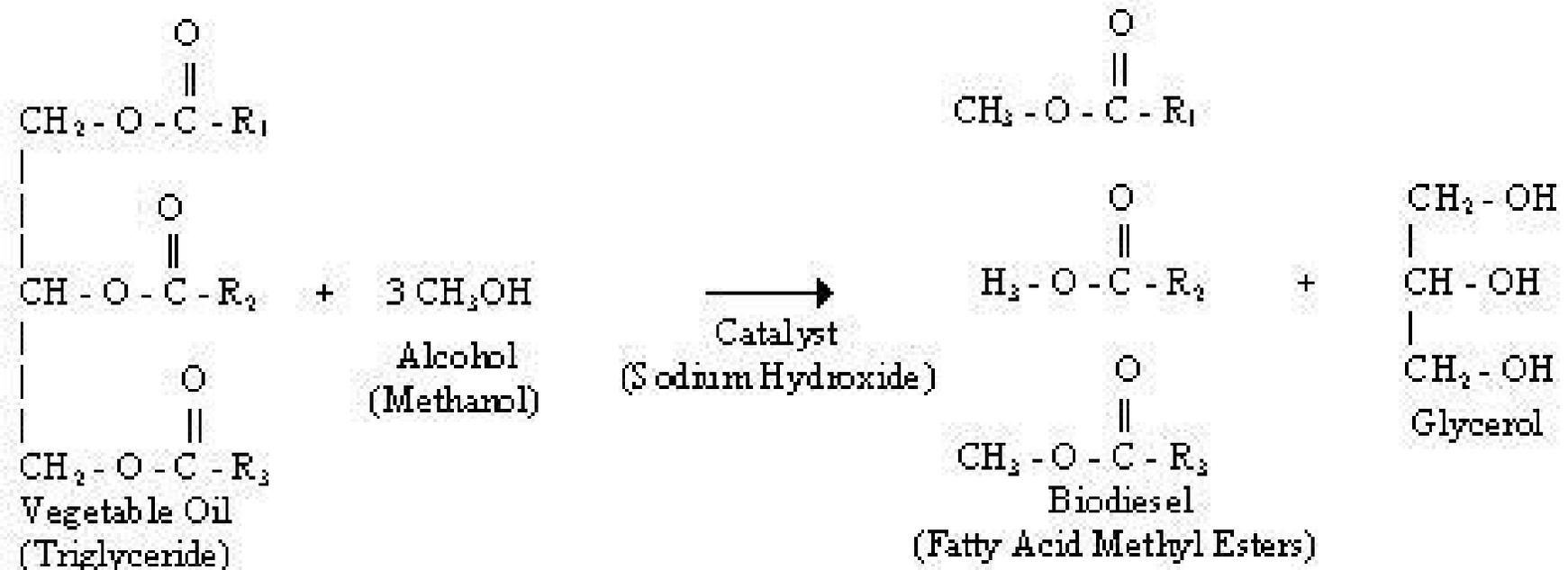
GLYCERIN

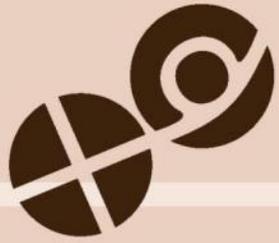
+





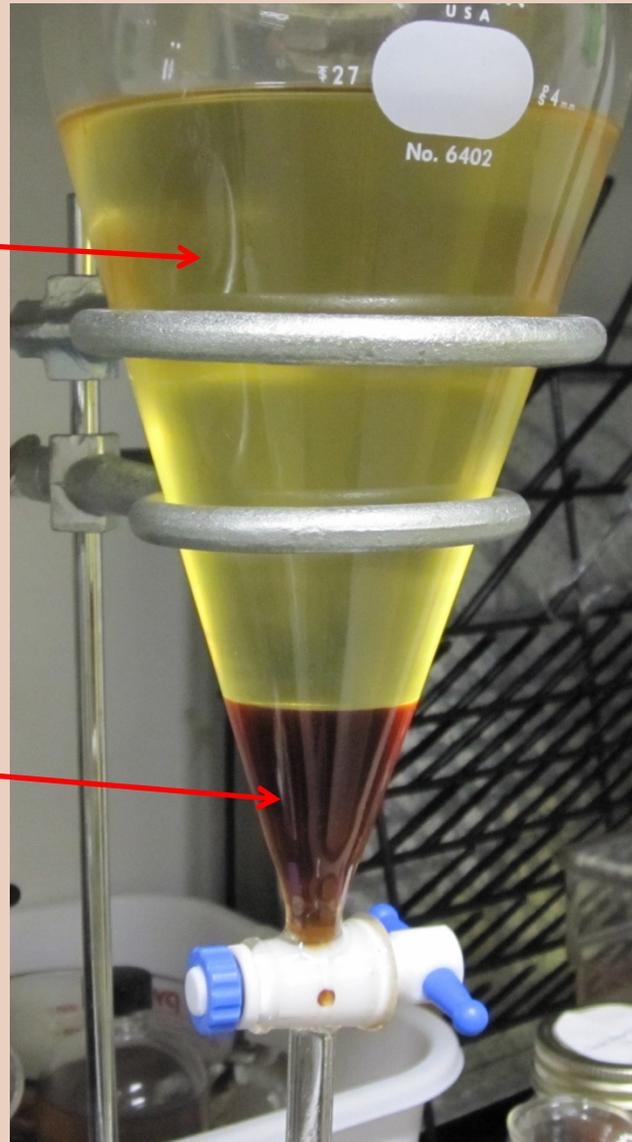
The Chemical Reaction...





Biodiesel and Glycerin

Biodiesel Layer



Glycerin Layer

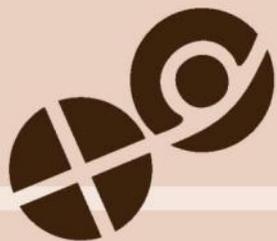




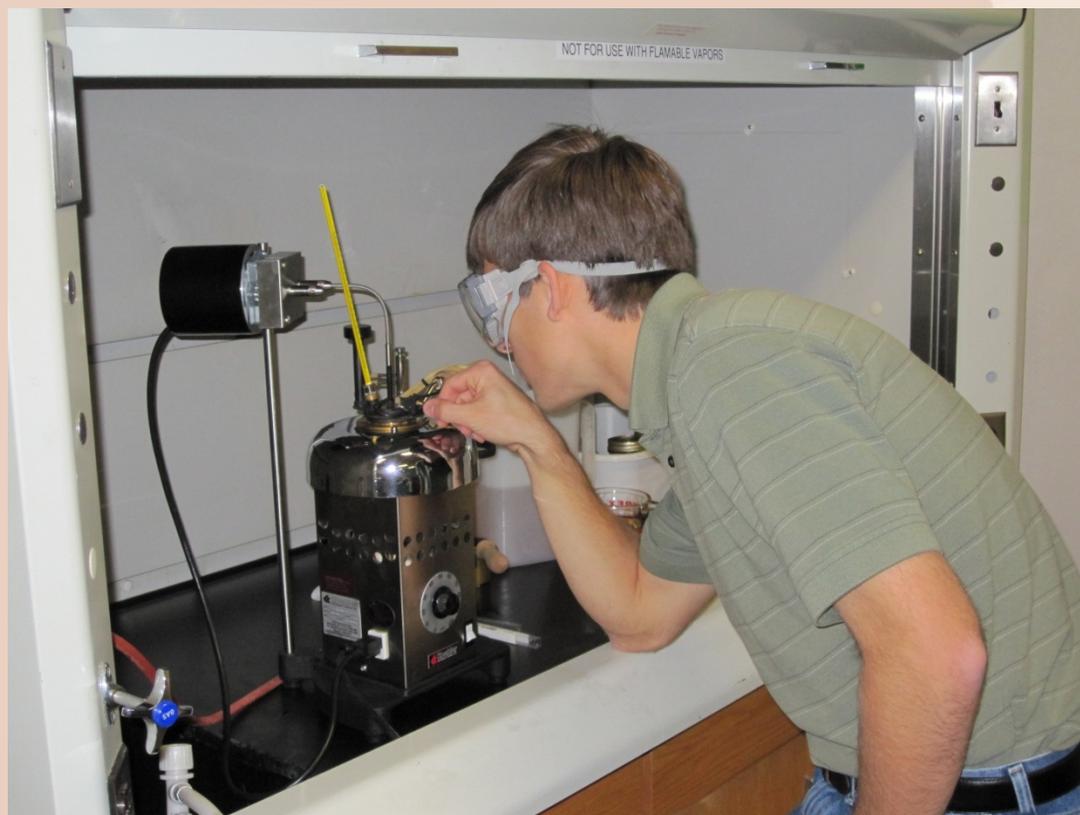
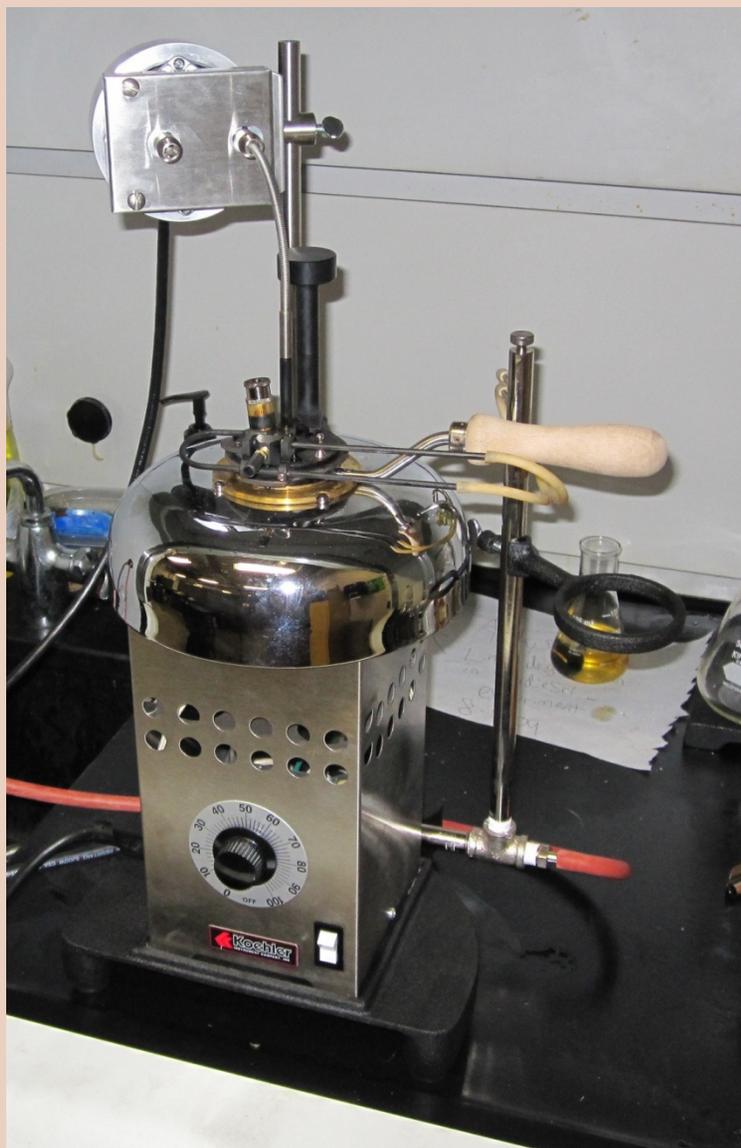
Quality Testing

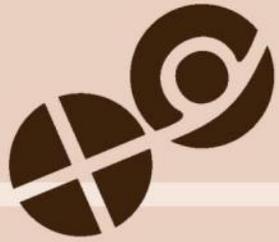
ASTM D6751

| Property | Method | Limits | Units |
|-------------------------------|---------------------|---------------------------------|---------------------|
| Flash point, closed cup | D 93 | 130 min | °C |
| Water and sediment | D 2709 | 0.050 max | % volume |
| Kinematic viscosity, 40 ° C | D 445 | 1.9 – 6.0 | mm ² /s |
| Sulfated ash | D 874 | 0.020 max | wt. % |
| Total Sulfur | D 5453 | 0.05 max | wt. % |
| Copper strip corrosion | D 130 | No. 3 max | |
| Cetane number | D 613 | 47 min | |
| Cloud point | D 2500 | Report to customer | °C |
| Carbon residue | D 4530 | 0.050 max | wt. % |
| Acid number | D 664 | 0.80 max | mg KOH/g |
| Free glycerin | D 6584 | 0.020 | wt. % |
| Total glycerin | D 6584 | 0.240 | wt. % |
| Phosphorus | D 4951 | 0.0010 | wt. % |
| Vacuum distillation end point | D 1160 | 360 °C max, at 90% distilled | °C |
| Storage stability | To be determined | To be determined | To be determined |

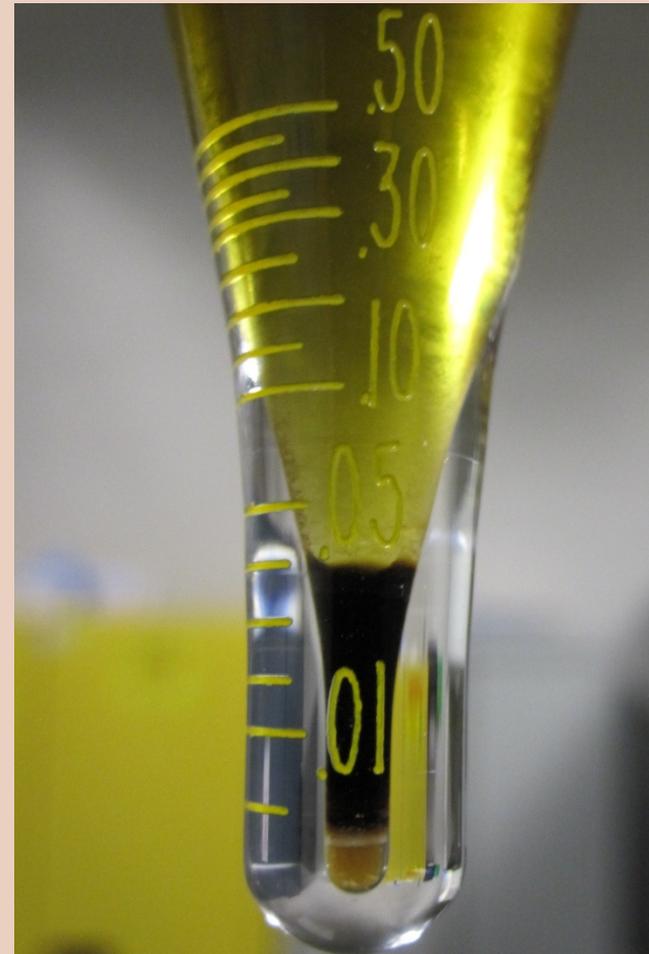


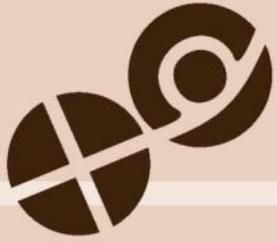
Flash Point Test



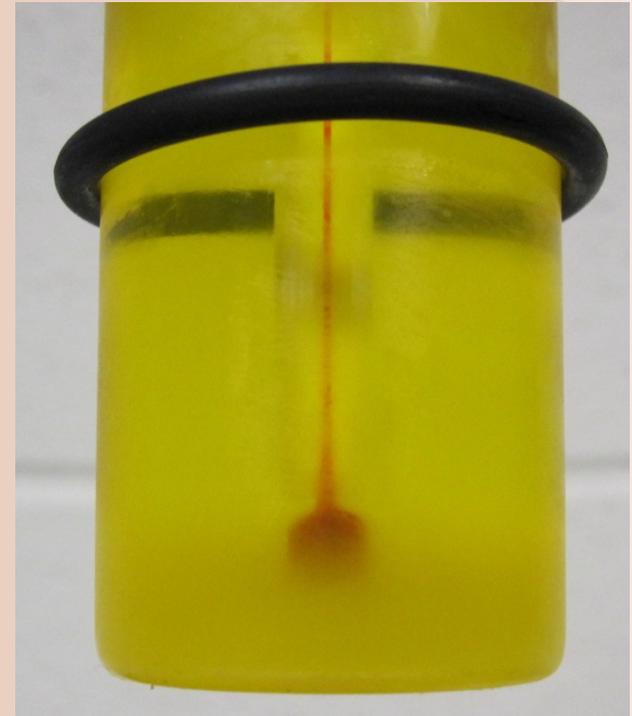


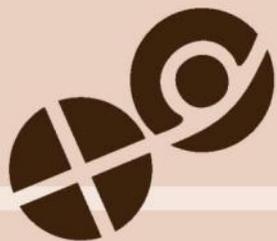
Water and Sediment Test



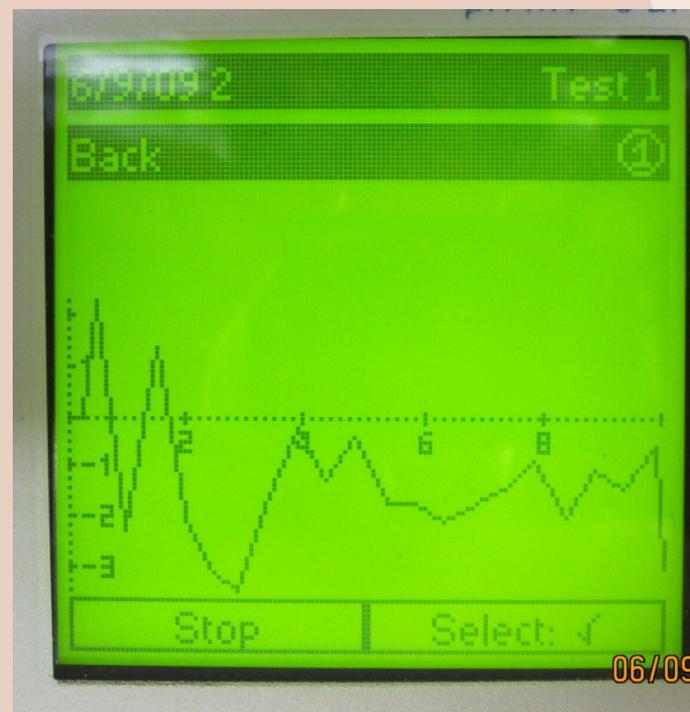
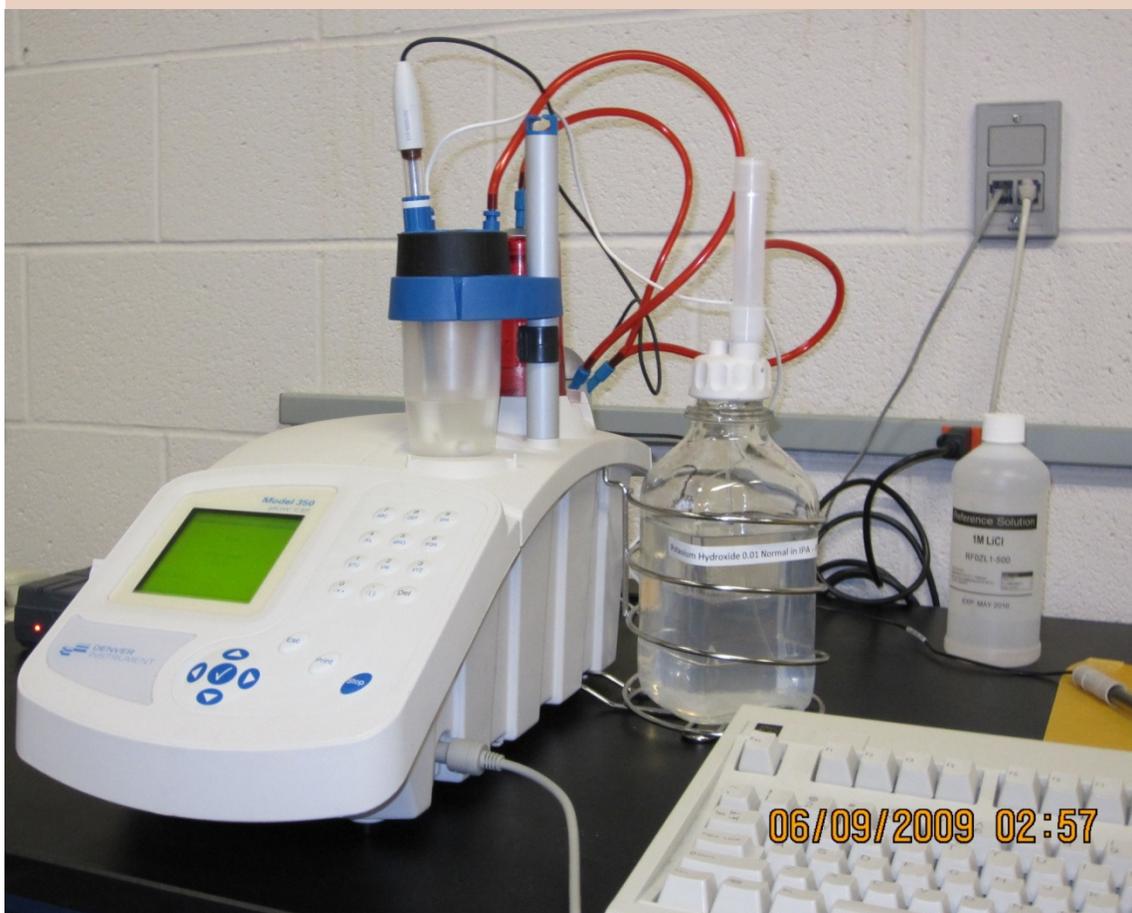


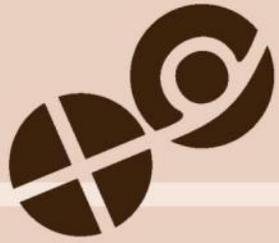
Cloud Point Test



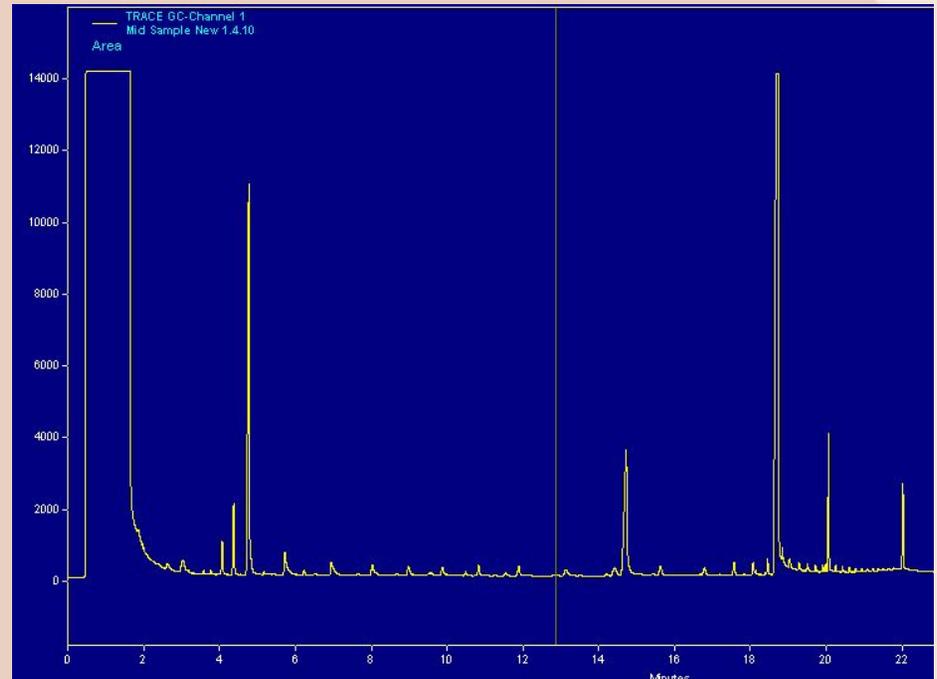


Acid Number Test





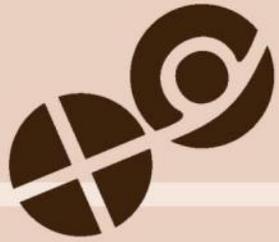
Total and Free Glycerin Test





How We Use Biodiesel





Power Production





Heating Oil Supplement



Questions?

Thank You For Your Time Tonight!

the Collaboratory
for strategic partnerships and applied research



Messiah College Biodiesel Fuel Generation Project

DE-FG36-08GO88068

Appendix B. – Educational Materials

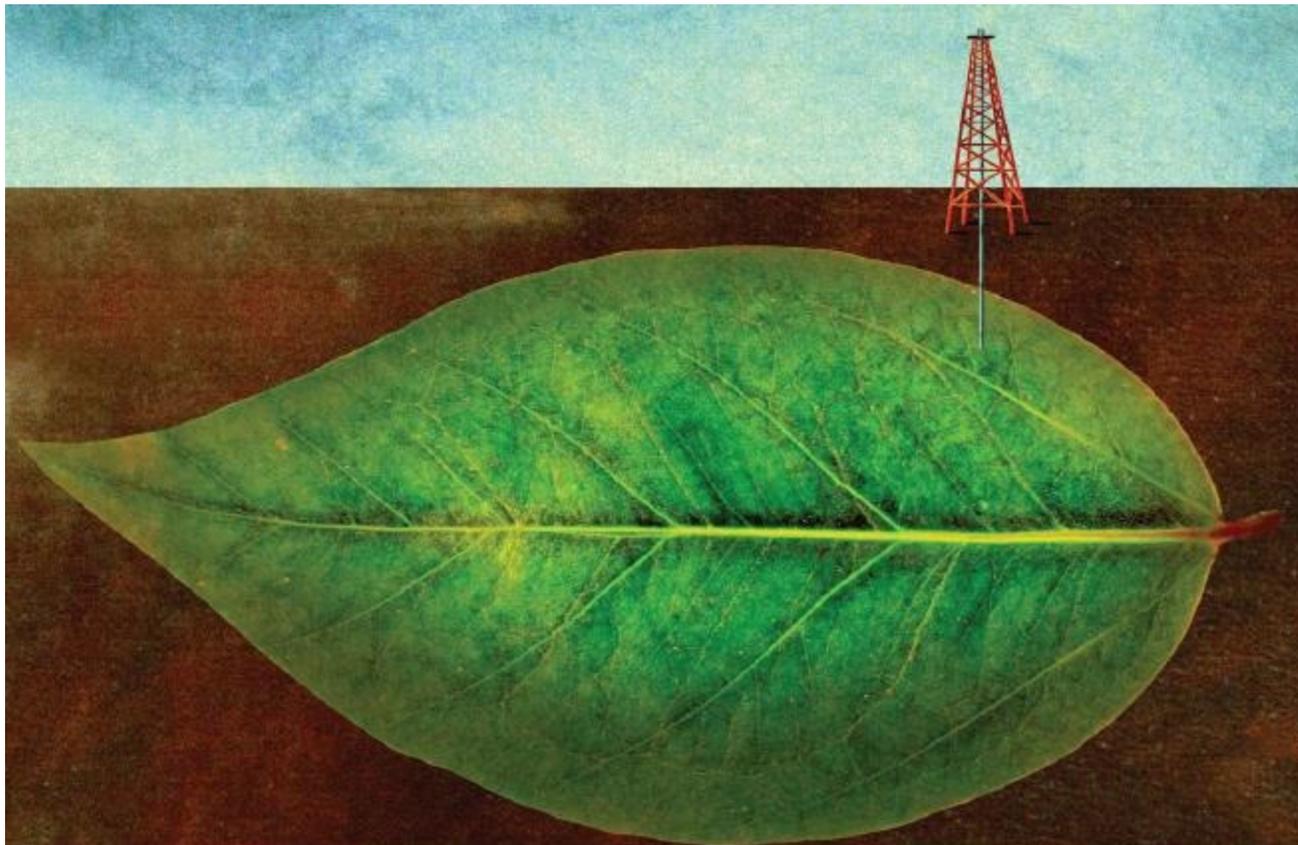
Appendix B.3.

Introduction to Biofuels Presentation

Introduction to Biofuels:

Vegetable Oil, Biodiesel and Ethanol

2/21/11

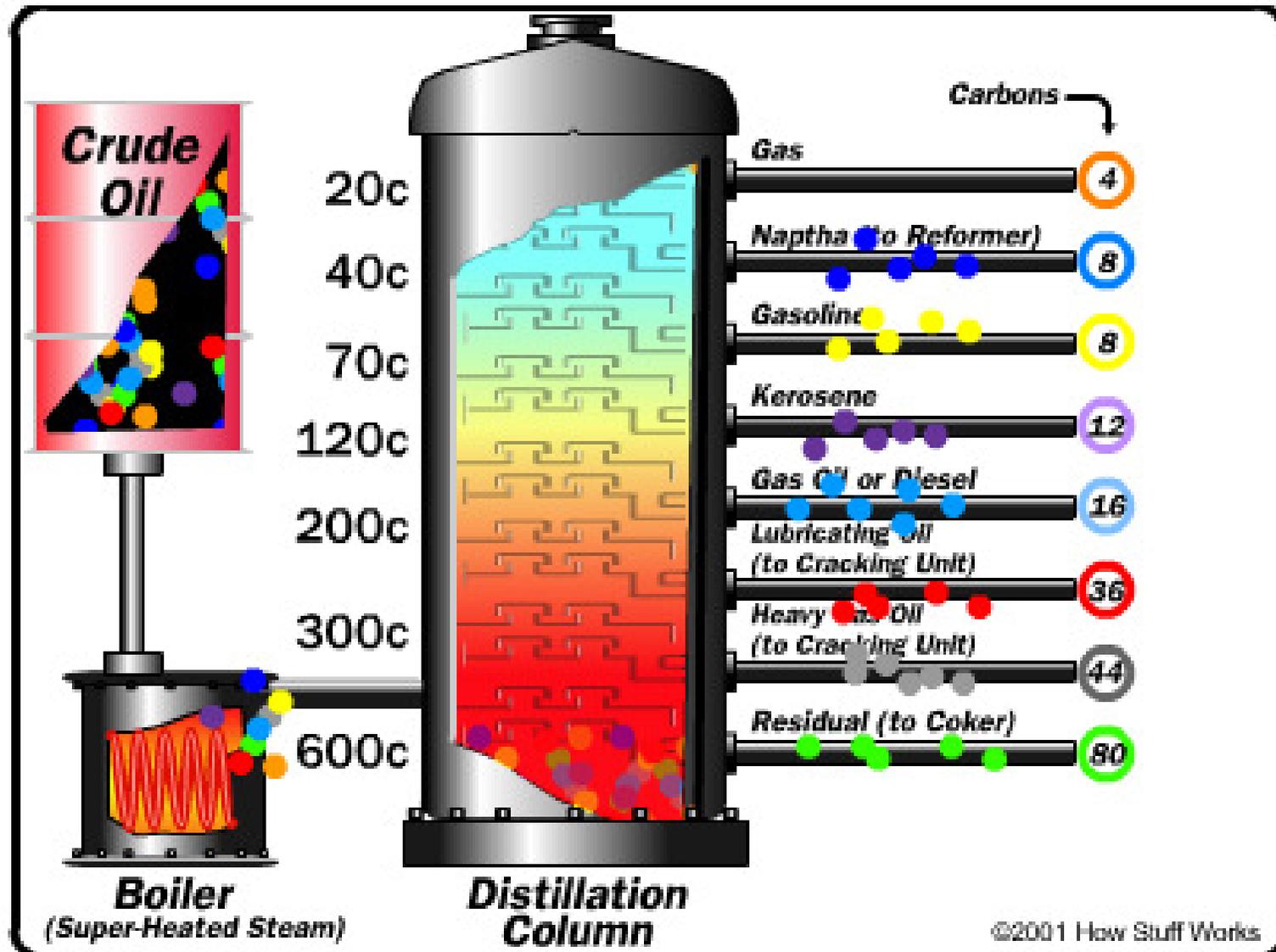


How are Petroleum Fuels made?

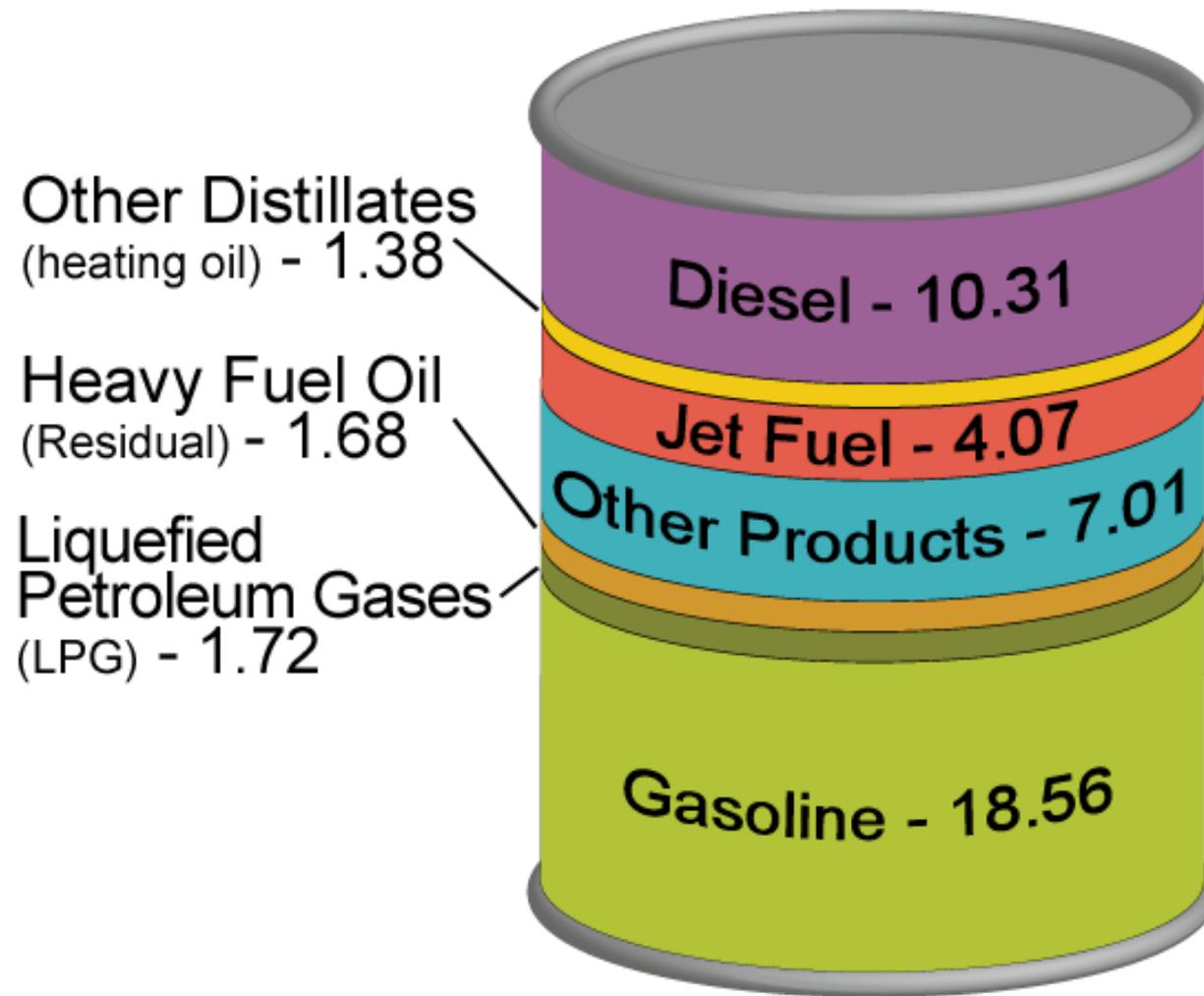
- Crude Oil is heated up to separate the complex mixtures of hydrocarbons into useable fuel products.
- Each petroleum derived product is distinguished by its boiling point.
- Diesel fuel undergoes a hydro-treating process to remove sulfur.



The Oil Refining Process



Products Made from a Barrel of Crude Oil (in Gallons)



From U.S. Energy Information Administration: Independent Statistics and Analysis
(http://tonto.eia.doe.gov/energyexplained/index.cfm?page=oil_home)

Refinery Products

| Product | Boiling Range Deg. C | Boiling Range Deg. F |
|-------------------------------|---------------------------------|---------------------------------|
| LPG | -40 - 0 | -40 - 31 |
| Gasoline | 30 - 200 | 80 - 400 |
| Kerosene, Jet Fuel, #1 Diesel | 170 - 270 | 340 - 515 |
| #2 Diesel, Furnace Oil | 180 - 340 | 350 - 650 |
| Lube Oils | 340 - 540 | 650 - 1000 |
| Residual Oil | 340 - 650 | 650 - 1200 |
| Asphalt | 540 + | 1000 + |
| Petroleum Coke | Solid | |

From: Schmidt, G.K. and Forster, E.J., "Modern Refining for Today's Fuels and Lubricants," SAE Paper 861176, 1986.

The petroleum fuel we use...

- In 2008 the U.S. petroleum consumption was 19.5 million barrels /day (820 million gallons/day).
- 8.989 million barrels/day of gasoline (378 million gallons/day)
- 3.945 million barrels/day of Diesel Fuel and Heating Oil (166 million gallons/day)
- 2/3 of U.S. petroleum consumption is for transportation and 2/3 of transportation consumption is gasoline.

From U.S. Energy Information Administration: Independent Statistics and Analysis
(http://tonto.eia.doe.gov/energyexplained/index.cfm?page=oil_home)

Who is using all the petroleum fuel...

Top Five Gasoline Consuming States, 2008

| State | Million Barrels/Day | Million Gallons/Day | Share of Total U.S. Consumption |
|------------|---------------------|---------------------|---------------------------------|
| California | 0.98 | 41 | 11% |
| Texas | 0.83 | 35 | 10% |
| Florida | 0.50 | 21 | 6% |
| New York | 0.36 | 15 | 4% |
| Illinois | 0.33 | 14 | 4% |

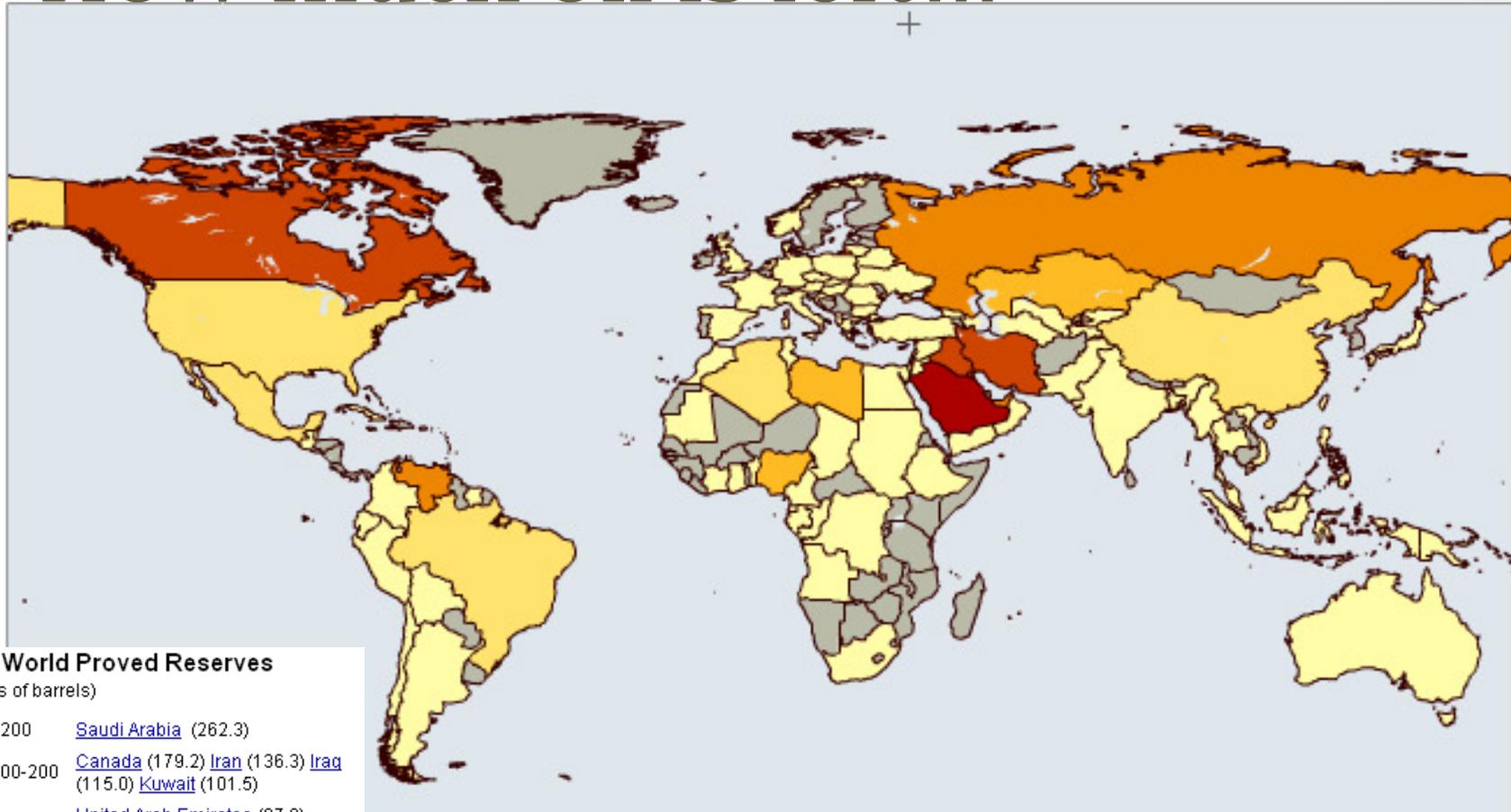
From U.S. Energy Information Administration: Independent Statistics and Analysis
(http://tonto.eia.doe.gov/energyexplained/index.cfm?page=oil_home)

How much petroleum does the world consume...

- Worldwide consumption of petroleum was 85.4 million barrels per day in 2008 (3.6 billion gallons/day).
- The three largest consuming countries were:
 1. The U.S. (19.5 million barrels per day)
 2. China (7.9 million barrels per day)
 3. Japan (4.8 million barrels per day)

From U.S. Energy Information Administration: Independent Statistics and Analysis
(http://tonto.eia.doe.gov/energyexplained/index.cfm?page=oil_home)

How much oil is left...



2008 World Proved Reserves
(billions of barrels)

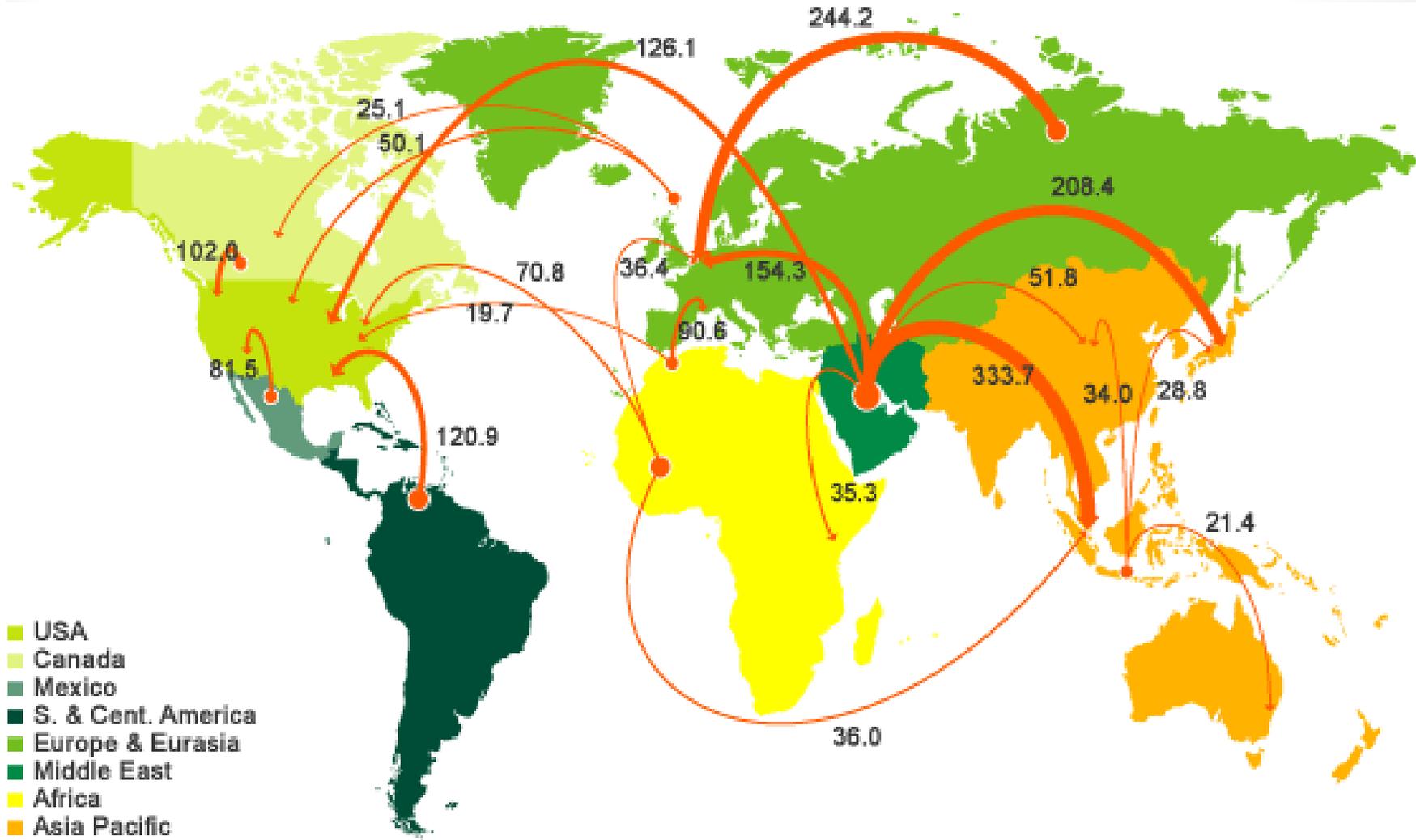
| | |
|----------|---|
| >200 | Saudi Arabia (262.3) |
| 100-200 | Canada (179.2) Iran (136.3) Iraq (115.0) Kuwait (101.5) |
| 50-100 | United Arab Emirates (97.8) Venezuela (80.0) Russia (60.0) |
| 25-50 | Libya (41.5) Nigeria (36.2) Kazakhstan (30.0) |
| 10-25 | United States (21.0) China (16.0) Qatar (15.2) Mexico (12.4) Algeria (12.3) Brazil (11.8) |
| <10 | 78 countries |
| 0 (none) | -2 countries |

(s) = Less than 500,000 barrels

From U.S. Energy Information Administration: Independent Statistics and Analysis

(<http://tonto.eia.doe.gov/country/index.cfm?view=reserves>)

Major Oil Trade Movements



From: http://www.ccs.neu.edu/home/gene/peakoil/map_major_oil_move_567x366.gif

Impacts of the Oil and Petroleum Based Industries

- Social
- Economic
- Environmental
- Cultural
- Medical

Petroleum Emissions and Byproducts

| Emission | Effect |
|------------------------------------|---|
| Carbon Dioxide (CO ₂) | Greenhouse Gas, Global Warming |
| Carbon Monoxide (CO) | Greenhouse Gas, Global Warming, Respiratory Illness |
| Sulfur Dioxide (SO ₂) | Acid Rain, Respiratory Illnesses, Heart Disease) |
| Nitrogen Oxides (NO _x) | Ground Level Ozone, Damage to Lungs |
| Volatile Organic Compounds (VOC) | Ground Level Ozone, Damage to Lungs |
| Particulate Matter (PM) | Smog, Asthma, Bronchitis, Cancer |
| Lead | Cancer |

How much oil is left...

Total World Oil Production : 85,472,000 barrels/day
Total World Oil Consumption : -85,534,000 barrels/day
- 62,000 barrels/day

Total World Oil Reserve: 1,342.2 Billion Barrels
Total World Oil Consumption: \div 85,534,000 barrels/day
15,692 Days = **43 Years**

Figures based on data from U.S. Energy Information Administration:
Independent Statistics and Analysis

(http://tonto.eia.doe.gov/energyexplained/index.cfm?page=oil_home#tab2)

Sustainability

- **Sustainability**- a process that can be *continued indefinitely without depleting* the energy or material resources on which it depends.
- **Ecosystem**- a grouping of plants, animals or other organisms that interact with each other and their environment in such a way as to perpetuate the grouping more or less indefinitely.
- **Principles of Sustainability:**
 - Ecosystems **recycle** their own **waste** and **reuse** the nutrients of all elements.
 - Ecosystems use **sunlight** as their energy source.
 - Consumer population size is maintained such that overuse of resources does not occur.
 - Biodiversity is maintained.





Renewable Energies

- Renewable Energies are replenished, they come back after they are used.
- All forms of renewable energy come from the sun (directly or indirectly).
- Biofuels are a type of renewable energy. They are made from plant or animal based materials.
 - Straight Vegetable Oil (SVO)- made from vegetables.
 - Biodiesel - made from fats and oils.
 - Ethanol - made from sugars.

Ethanol- What is it?

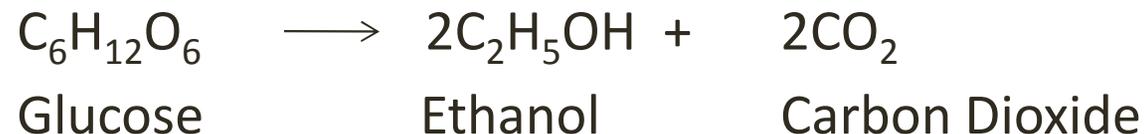
- A clear, colorless alcohol fuel made from the sugars found in grains, such as corn, sorghum, and barley, as well as potato skins, rice, sugar cane, sugar beets, and yard clippings.
- A renewable fuel because it is made from plants.



From U.S. Energy Information Administration: Independent Statistics and Analysis
(http://tonto.eia.doe.gov/energyexplained/index.cfm?page=biofuel_ethanol_home)

How Ethanol is made...

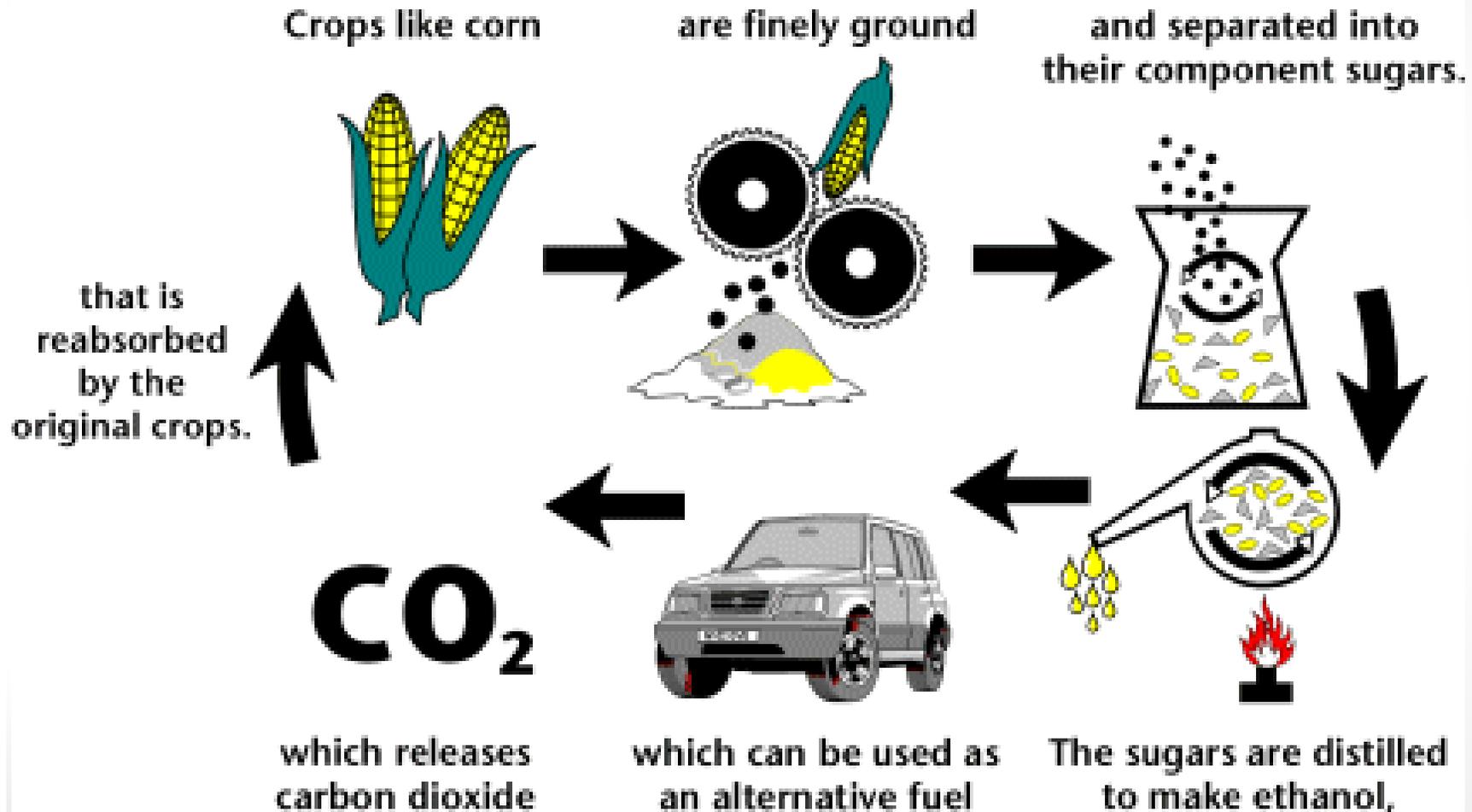
- Chemical process from other hydrocarbons
- Fermentation of sugars, followed by distillation to purify
- Overall reaction is:



- In the U.S. most ethanol is produced from corn.
- In other parts of the world sugar beets and sugar cane are common feedstock for ethanol.

From Bioethanol Presentation by Tim Turner and
From U.S. Energy Information Administration: Independent Statistics and Analysis
(http://tonto.eia.doe.gov/energyexplained/index.cfm?page=biofuel_ethanol_home)

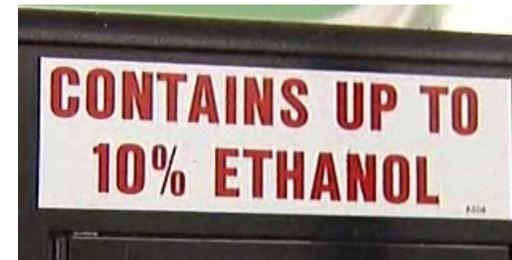
How Ethanol is made....



From: <http://www.nicholas.duke.edu/thegreengrok/greenoptions4.30>

How Ethanol is used?

- Directly in spark-ignition engines
 - E10- 90 % Gasoline + 10% Ethanol- All vehicles that run on gasoline can use E10 without changes to their engines. Over 99% of the ethanol produced in the U.S. is mixed with gasoline to make E10.
 - E85- 85% Ethanol + 15% Gasoline- an alternative fuel that is used mainly in the Midwest and South in specially designed Flexible Fuel Vehicles (FFV).
- As an alternative to Methanol in the biodiesel reaction process.



From Bioethanol Presentation by Tim Turner and
From U.S. Energy Information Administration: Independent Statistics and Analysis
(http://tonto.eia.doe.gov/energyexplained/index.cfm?page=biofuel_ethanol_home)

Advantages of Ethanol

- Can be easily made from local, renewable sources.
- Reasonably non-toxic to humans.
- Good fuel for spark-ignition engines.
- Good reactant to make biodiesel.



From Bioethanol Presentation by Tim Turner

Disadvantages of Ethanol

- Some bad combustion products- easily remedied.
- Low energy density compared to gasoline.
- Distillation is energy intensive.
- More expensive than methanol.
- More ethanol required than methanol to make biodiesel.
- Making ethyl esters is tricky.



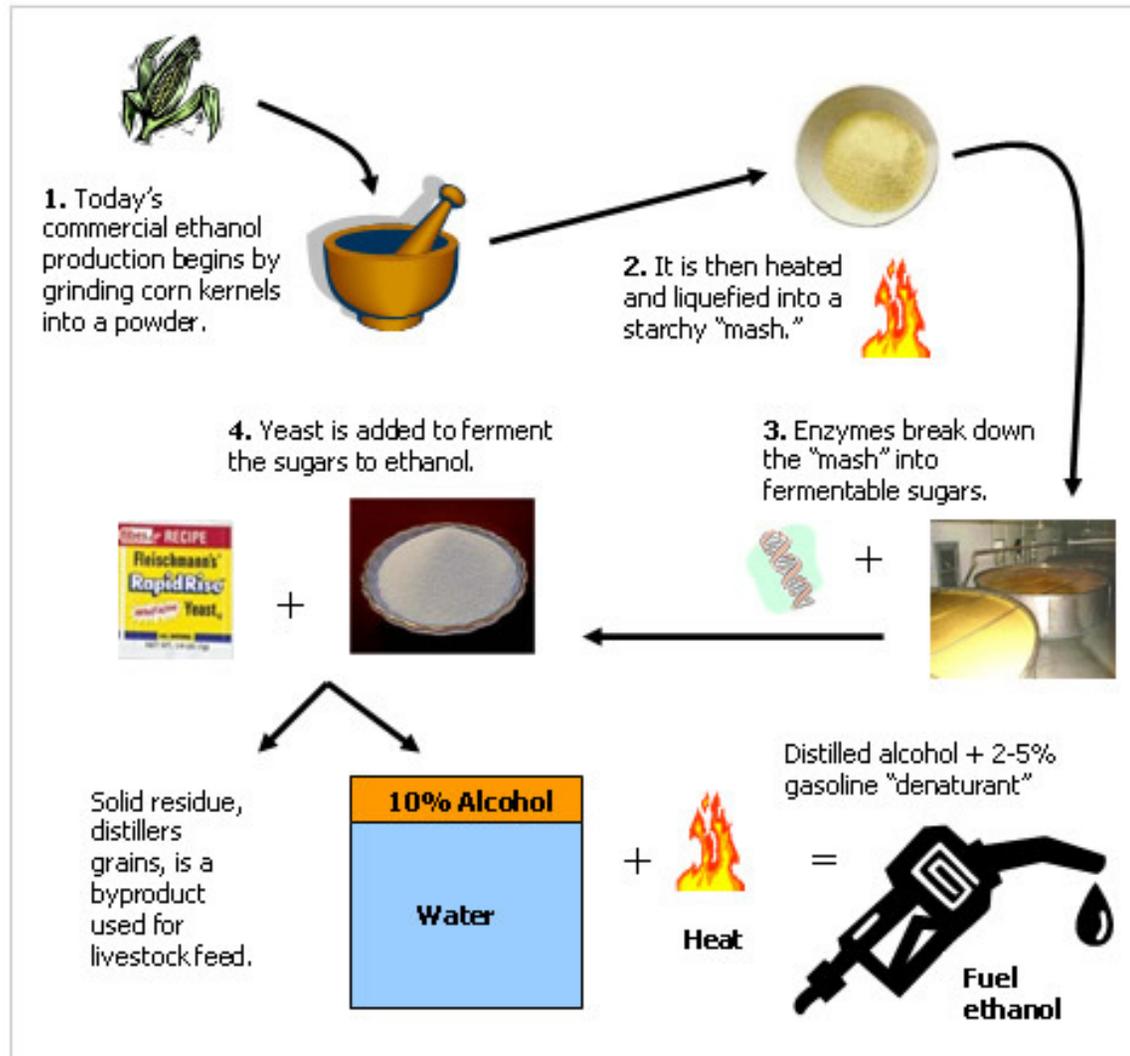
From Bioethanol Presentation by Tim Turner

3 Phases of Batch Production of Ethanol

- **Mashing-**
 - Convert plant materials (starches) to sugars
 - Dissolve starches in hot water
 - Convert starches to sugars using enzymes
- **Fermentation**
 - Convert sugar to ethanol using yeast or bacteria
 - An anaerobic process (sealed container)
- **Distillation**
 - Separating two or more liquids based on boiling/condensation points
 - Require heat or vacuum, or both.
 - Distillation columns produce higher quality ethanol

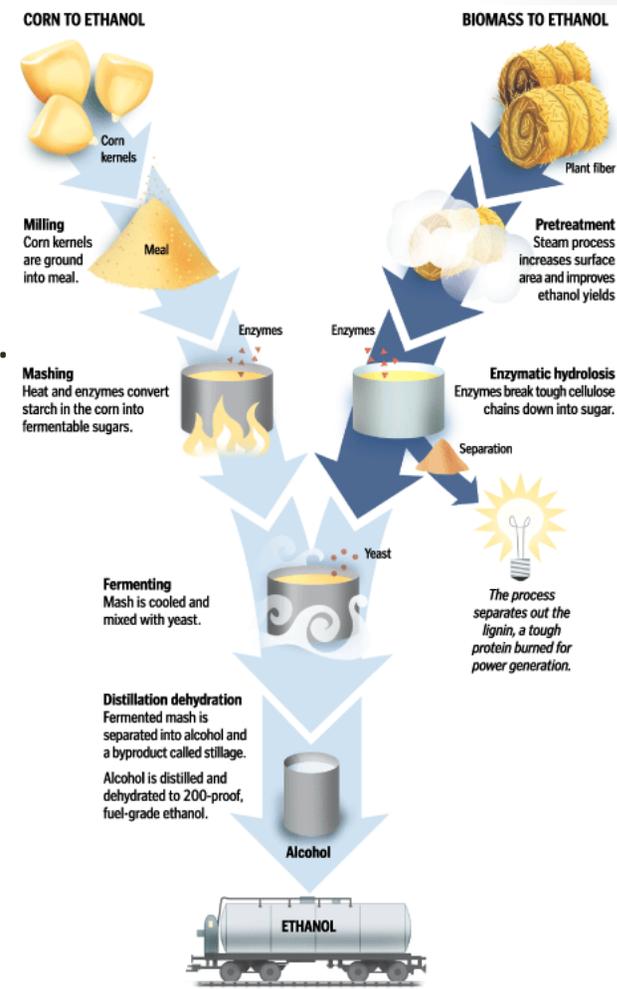
From Bioethanol Presentation by Tim Turner

3 Phases of Batch Production of Ethanol



Drawbacks of batch production

- Alcohol kills the yeast.
- Inefficient
 - Requires heating the entire batch to boiling.
 - Alcohol content of batch decrease over time.
 - At the end of the process:
 - Higher boiling temperature.
 - Lower yield per energy input.
 - Lower purity



From Bioethanol Presentation by Tim Turner

How to improve the ethanol production process

- Simultaneous mashing and fermentation
- Semi-continuous fermentation
- Continuous distillation
- Solar Thermal + Vacuum
 - Use solar collector to heat mixture to 140 – 150 degrees F.
 - Use vacuum pump to lower the system pressure so that the mixture boils at the current temperature.



From Bioethanol Presentation by Tim Turner

Modification to engine to run on Ethanol

- Enlarge injector nozzle openings or carburetor jets.
- Get a bigger fuel pump.
- Advance the spark timing.
- Replace rubber fuel lines, gaskets, etc.

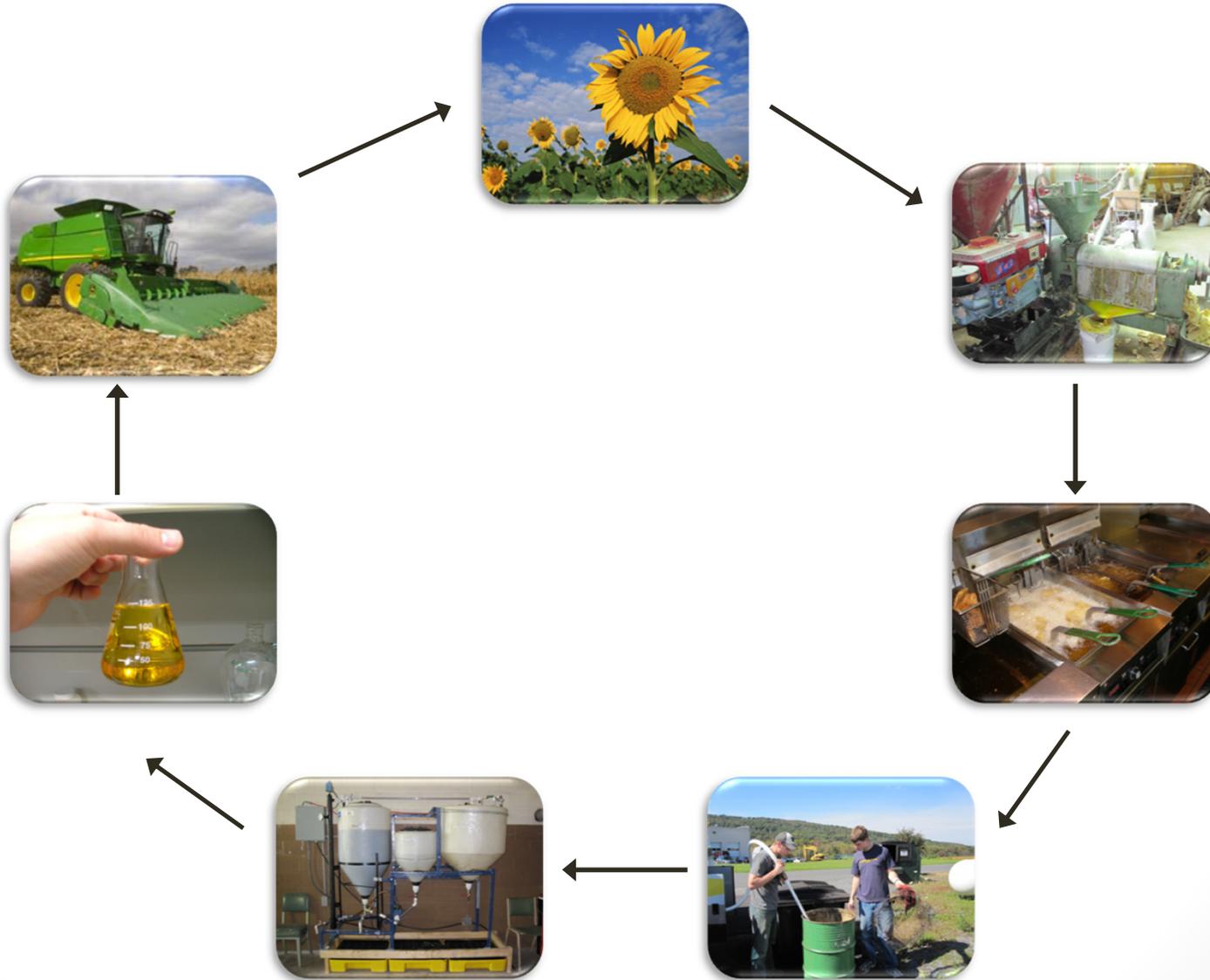


From Bioethanol Presentation by Tim Turner

What is Biodiesel?

An **environmentally friendly** alternative to petroleum base diesel fuel. It is produced by a reaction that occurs when fats and oils are combined with an alcohol and a catalyst. Its primary advantages are that it is an **agriculturally based renewable fuel** that is **non-toxic** and **biodegradable**. Biodiesel combustion produces **less carbon monoxide, unburned hydrocarbons and particulate emissions** than petro-based diesel. It is a closed carbon cycle fuel that emits **no net greenhouse gasses**. The carbon released during combustion of biodiesel fuel was previously removed from the environment by the oil producing plants.

From the field to fuel.



The Process at Messiah

College

Market Use
(Cafeteria Fryer)



Waste Product
(Collection)



Biodiesel
Production
(Small Scale
Processor)



Quality
Testing



End Use
(Campus Vehicles)



It all begins at the farm.



Seed Oil Pressing



French Fries!



Waste Vegetable Oil



Their waste is our fuel!



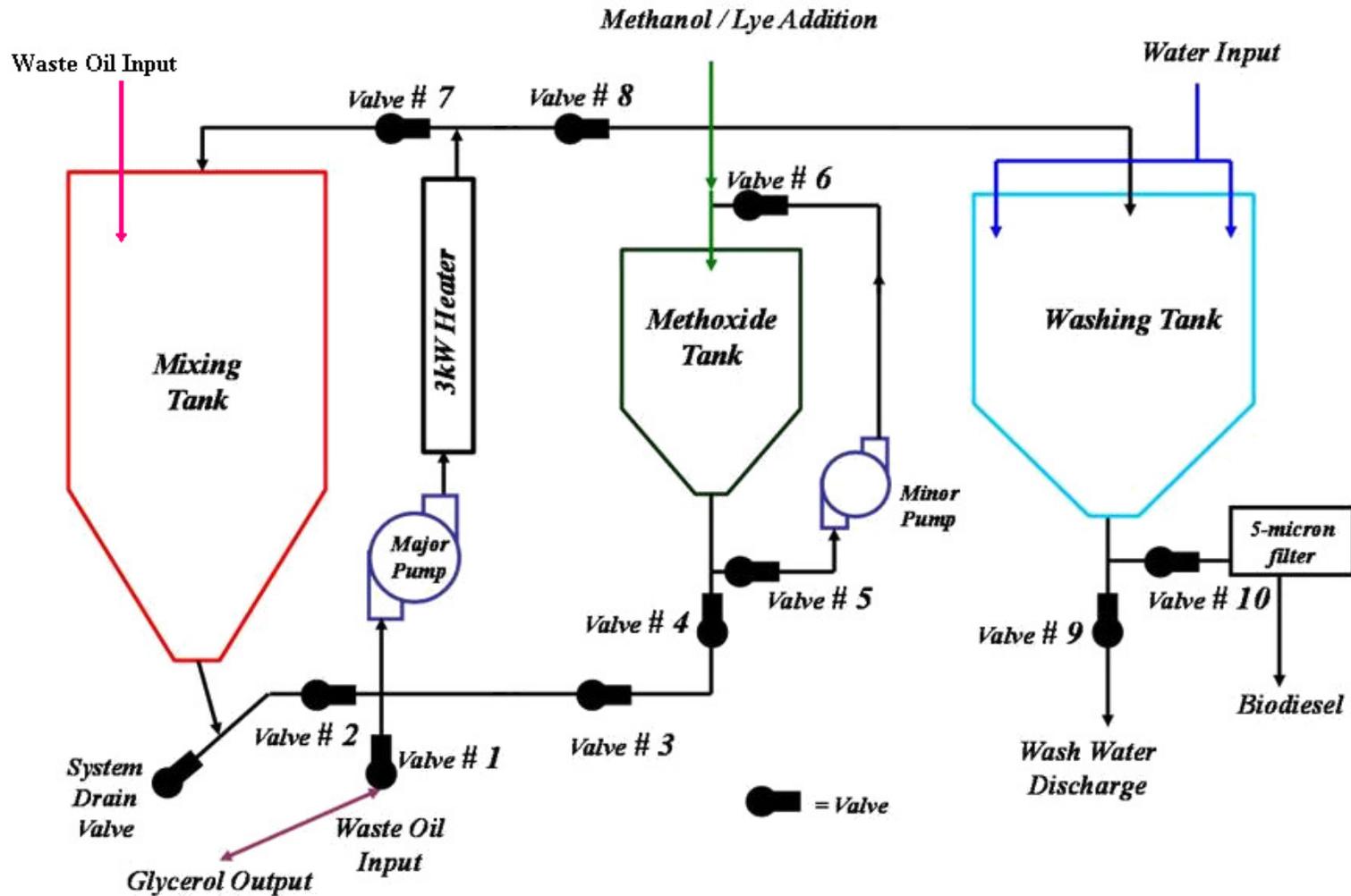
Storage and Filtration



Biodiesel Production

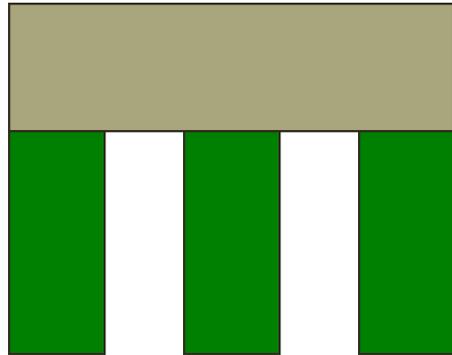


Our Process



Biodiesel Production...

TRIGLYCERIDE



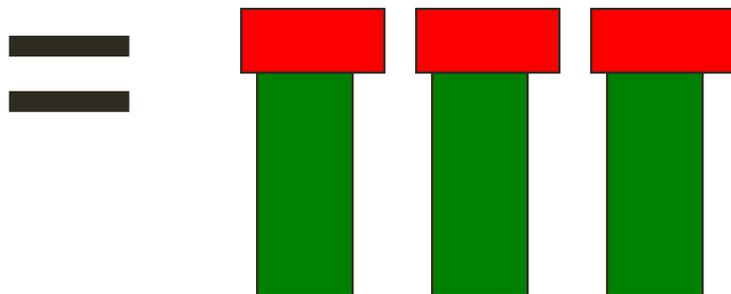
METHANOL



+

Catalyst

FREE ESTERS



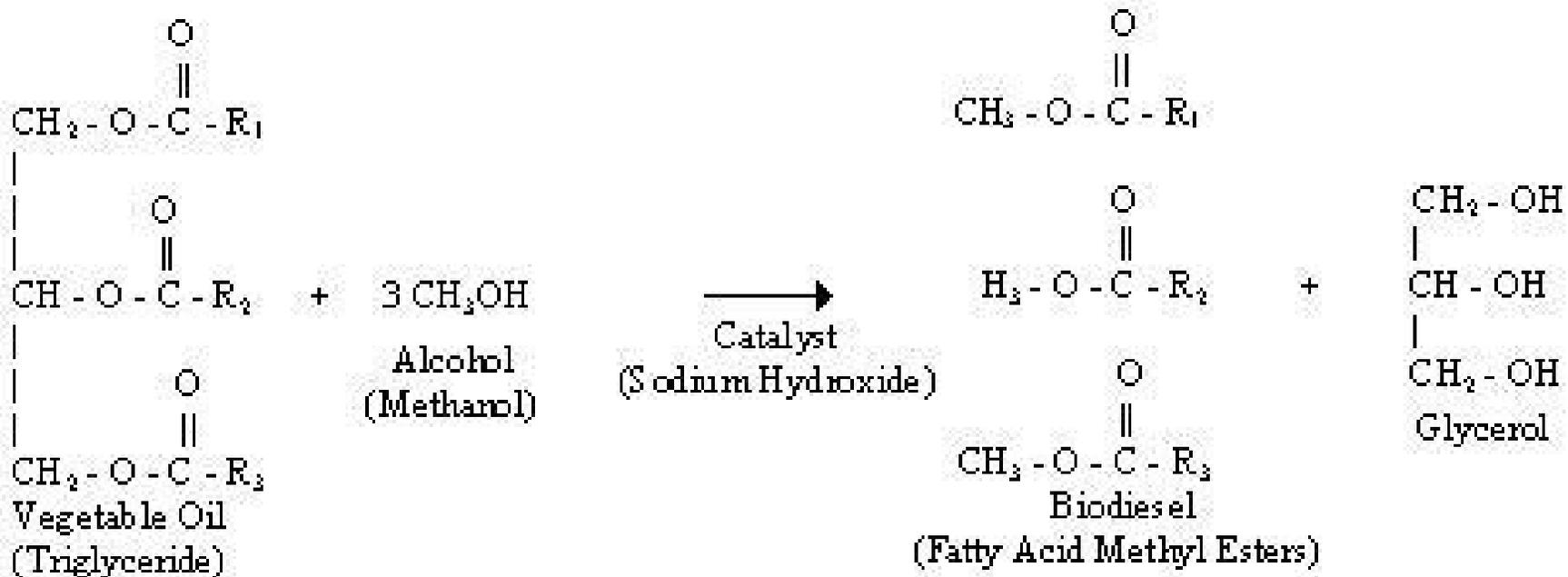
(BIODIESEL)

GLYCERIN

+

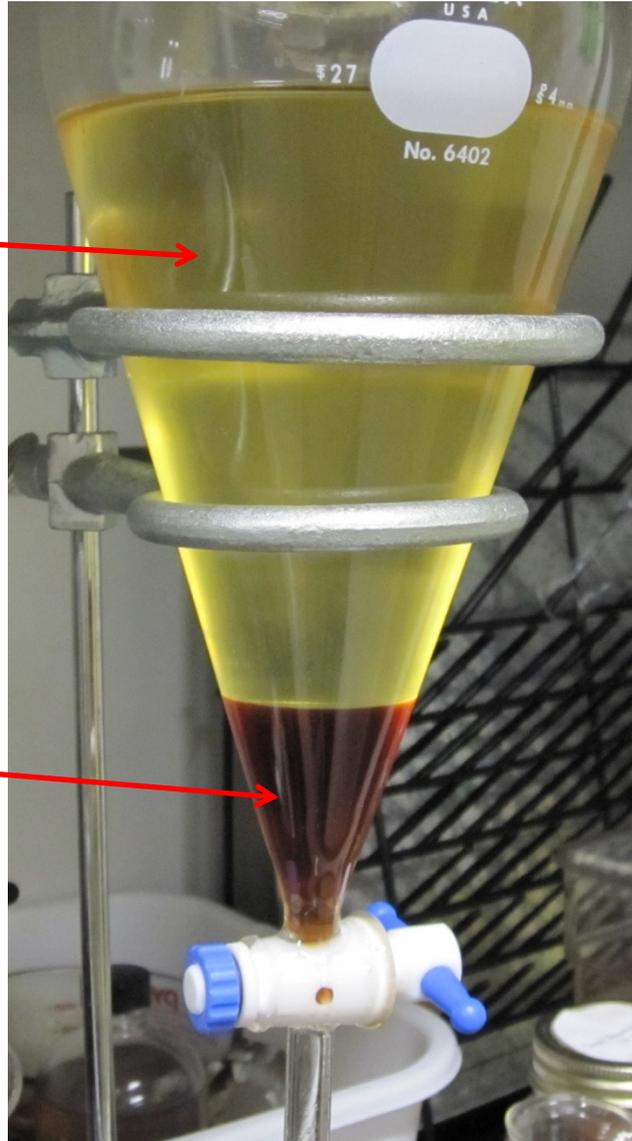


The Chemical Reaction...



Biodiesel and Glycerin

Biodiesel Layer



Glycerin Layer

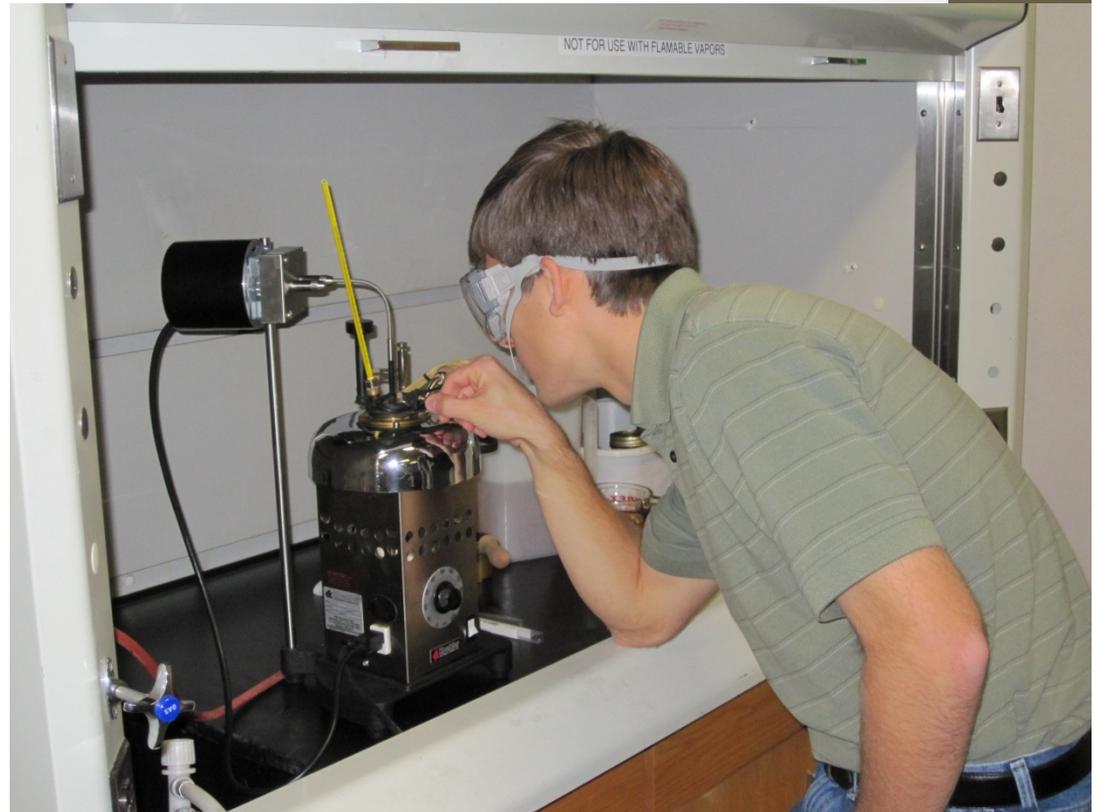
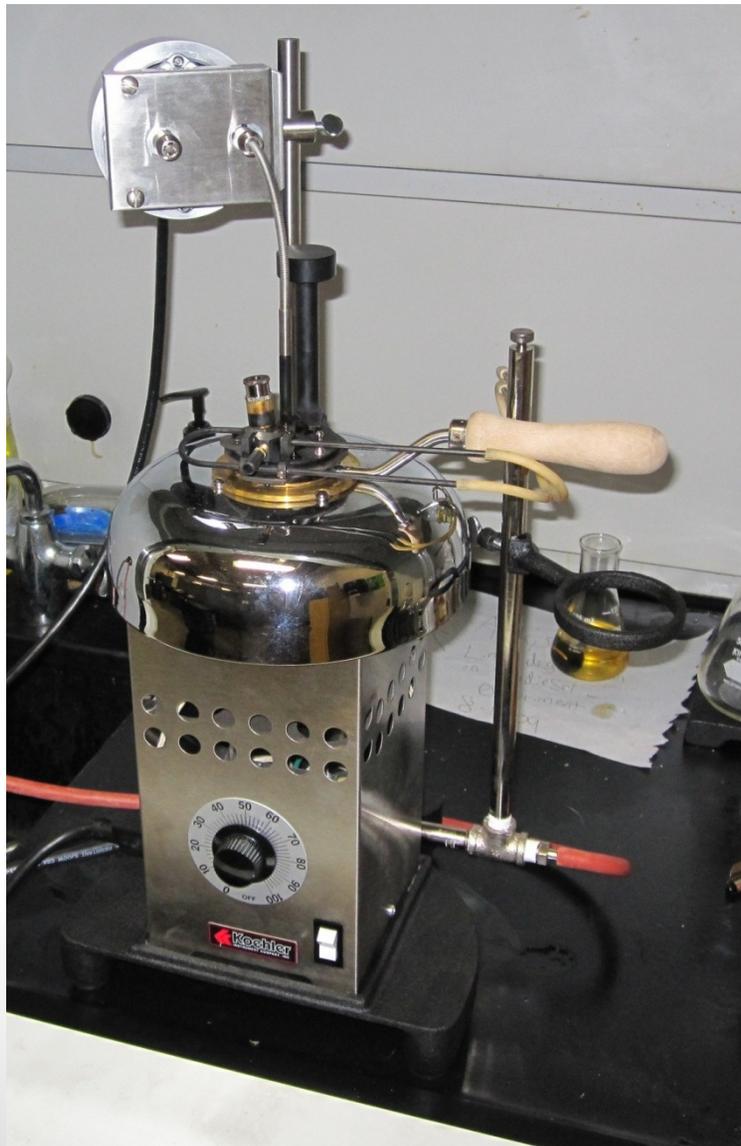
Methanol Recovery



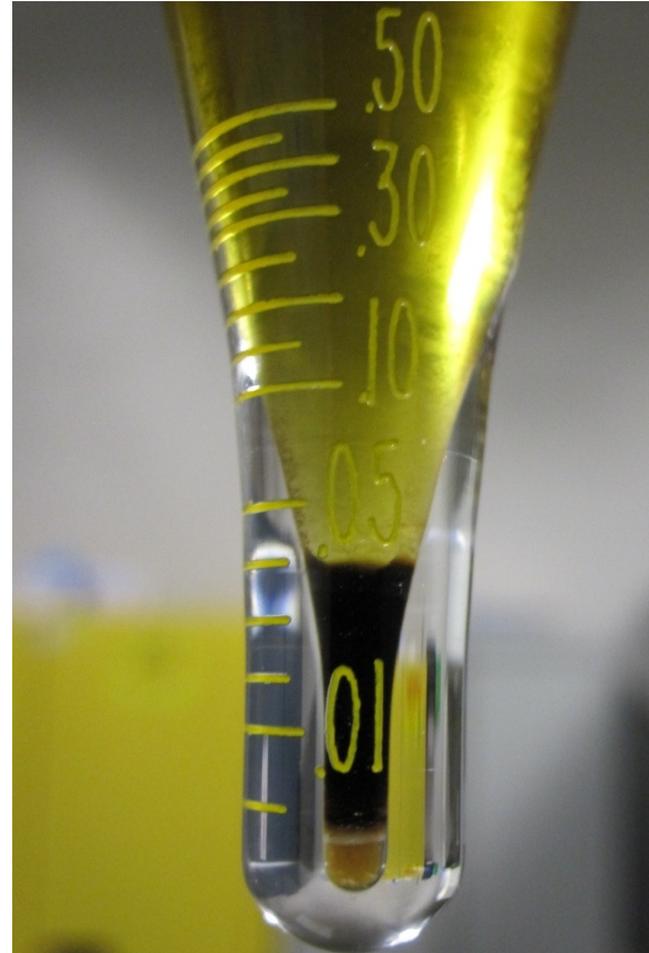
Quality Testing: ASTM D6751

| Property | Method | Limits | Units |
|-------------------------------|---------------------|---------------------------------|---------------------|
| Flash point, closed cup | D 93 | 130 min | °C |
| Water and sediment | D 2709 | 0.050 max | % volume |
| Kinematic viscosity, 40 ° C | D 445 | 1.9 – 6.0 | mm ² /s |
| Sulfated ash | D 874 | 0.020 max | wt. % |
| Total Sulfur | D 5453 | 0.05 max | wt. % |
| Copper strip corrosion | D 130 | No. 3 max | |
| Cetane number | D 613 | 47 min | |
| Cloud point | D 2500 | Report to customer | °C |
| Carbon residue | D 4530 | 0.050 max | wt. % |
| Acid number | D 664 | 0.80 max | mg KOH/g |
| Free glycerin | D 6584 | 0.020 | wt. % |
| Total glycerin | D 6584 | 0.240 | wt. % |
| Phosphorus | D 4951 | 0.0010 | wt. % |
| Vacuum distillation end point | D 1160 | 360 °C max, at 90% distilled | °C |
| Storage stability | To be determined | To be determined | To be determined |

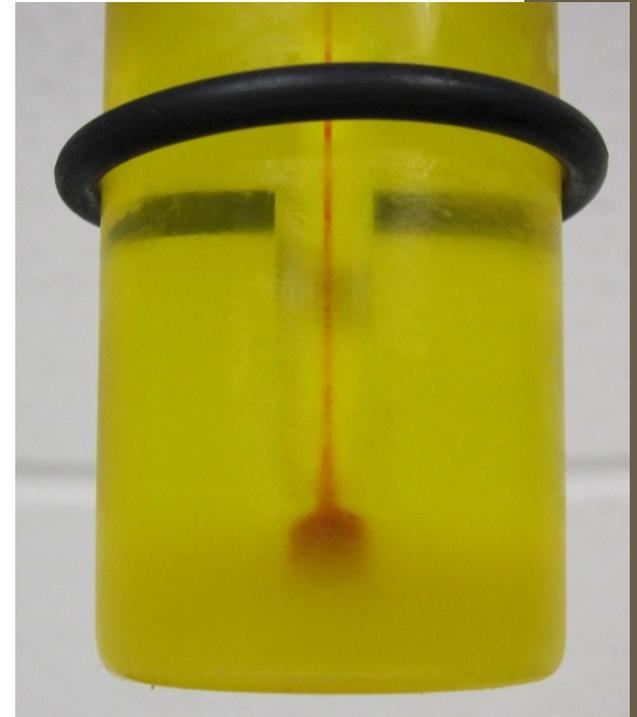
Flash Point Test



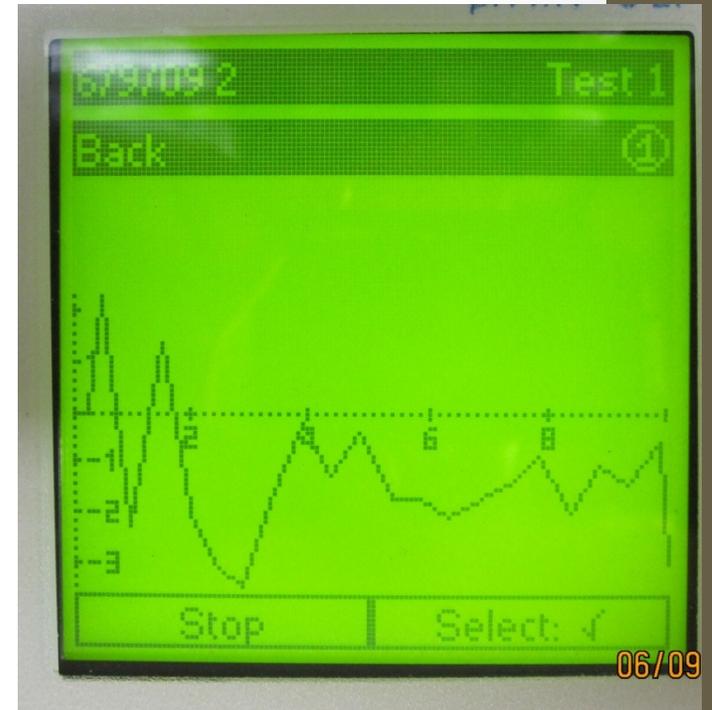
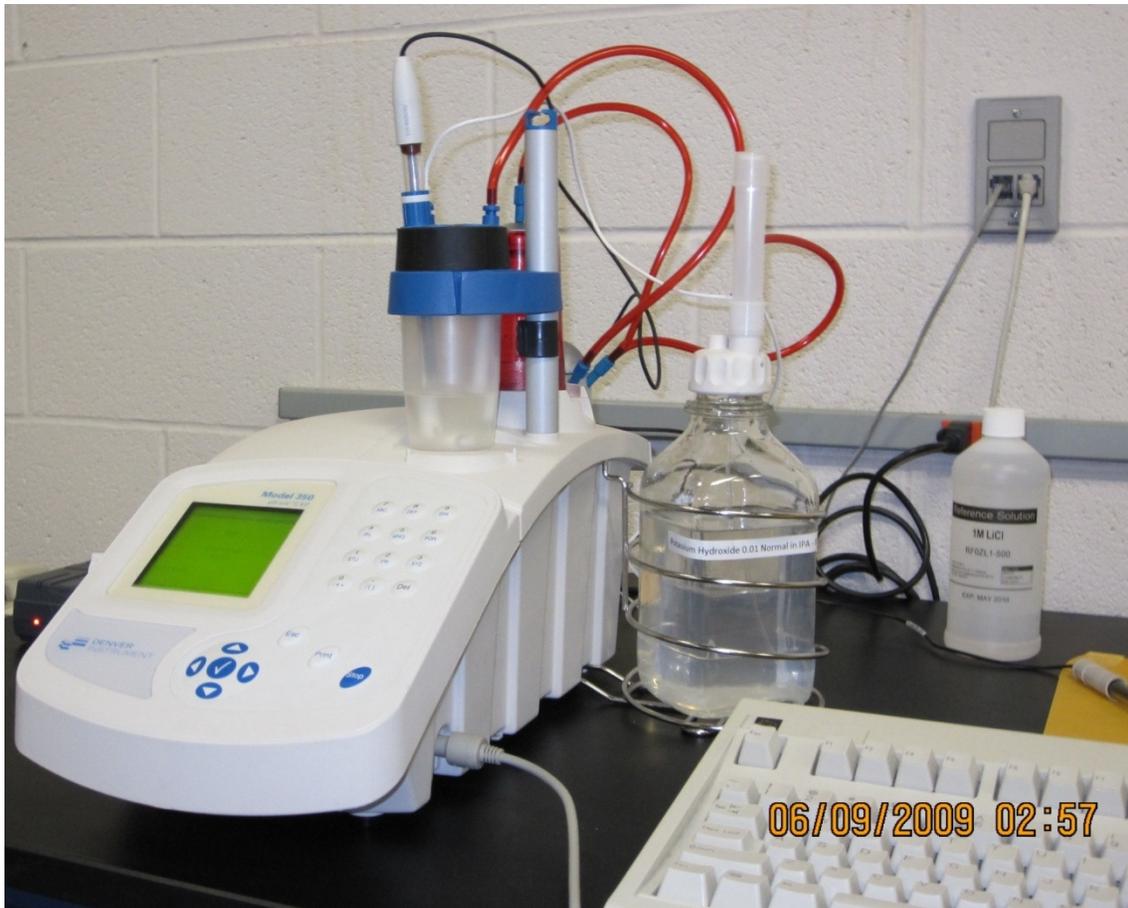
Water and Sediment Test



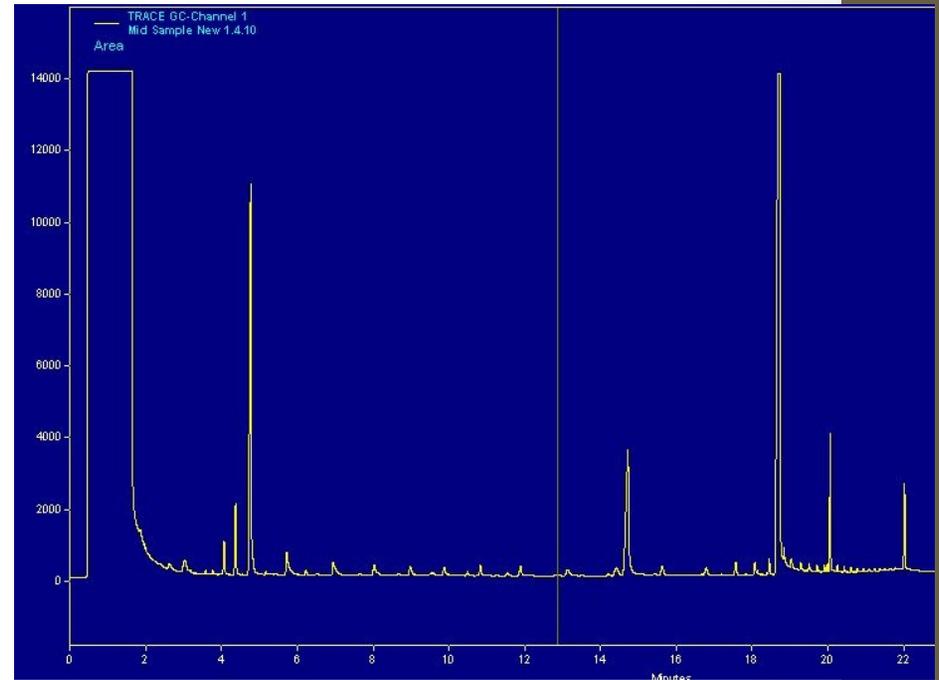
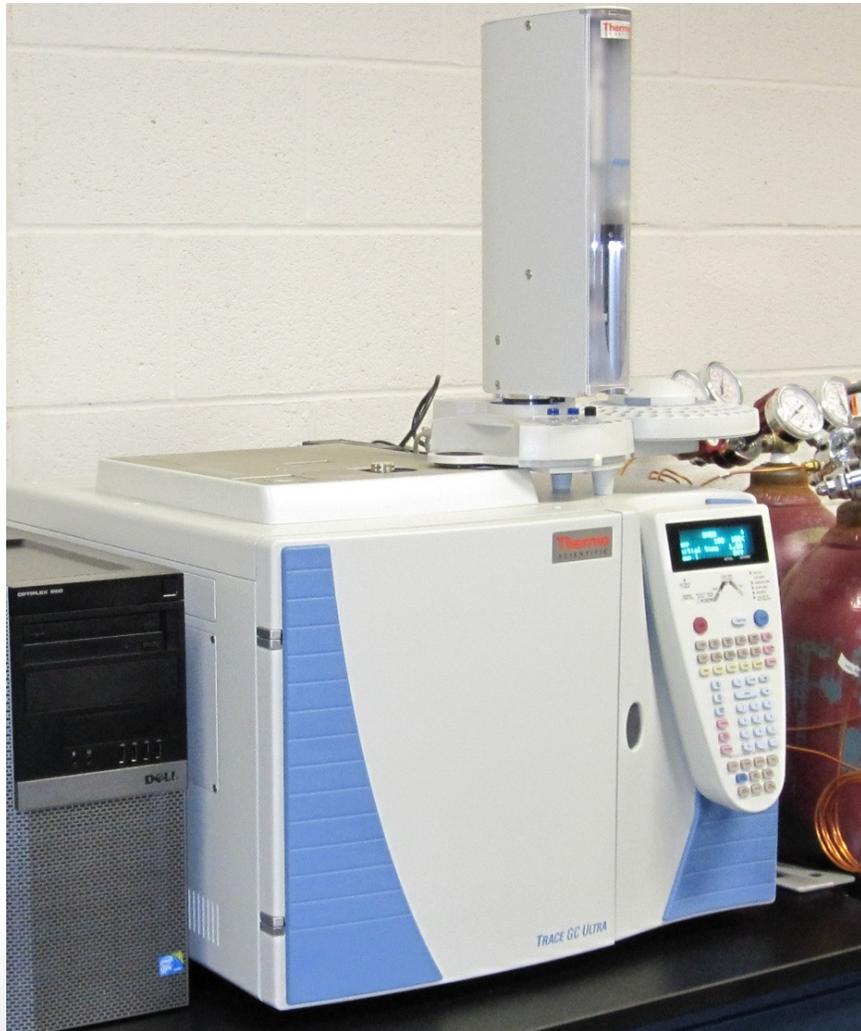
Cloud Point Test



Acid Number Test



Total and Free Glycerin Test



How We Use Biodiesel



Power Production



Heating Oil Supplement



Education



 Pennsylvania Farm Show 2009
January 10-17, 2009 • Farm Show Complex & Expo Center, Harrisburg, PA

Keeping Pennsylvania Growing



"The sun will no more be your light by day, ... for the Lord will be your everlasting light and your God will be your glory." Isaiah

MESSIAH COLLEGE

Carlisle greenfest
live green. save green.

MESSIAH COLLEGE

Alternative Feedstock: Jatropha



Burkina Faso Biodiesel



Pressing in Burkina Faso



Testing Reactor: Burkina Faso



Questions?

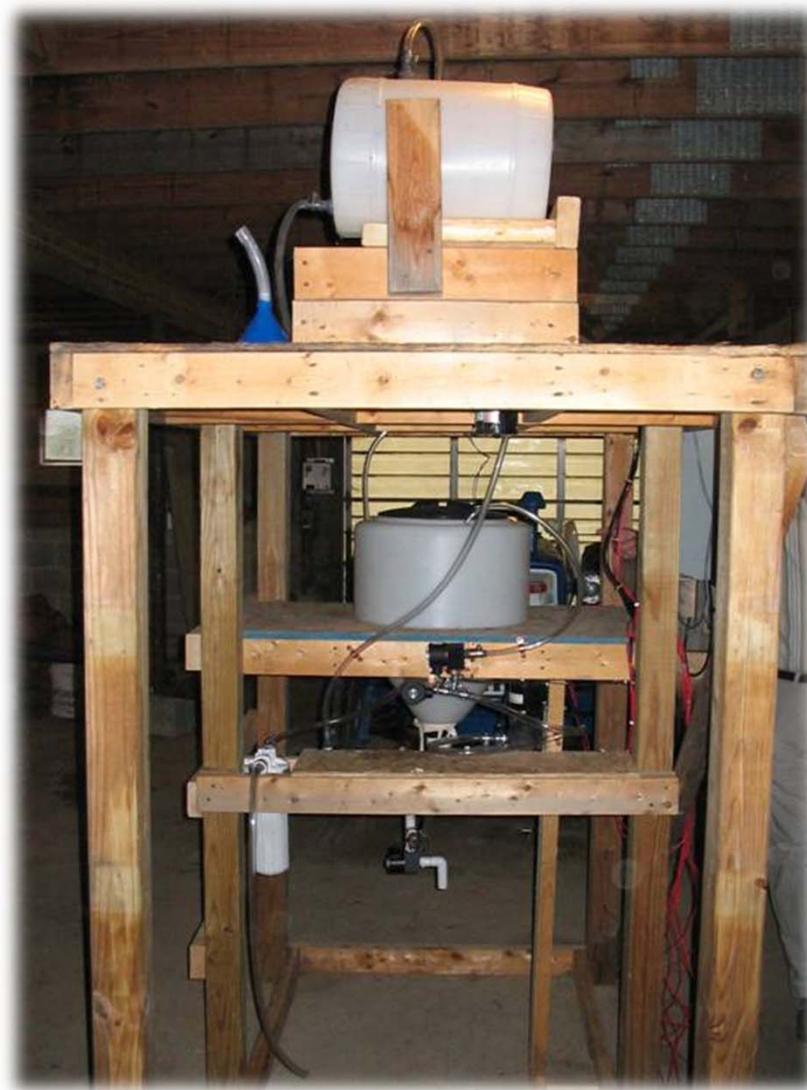
Messiah College Biodiesel Fuel Generation Project

DE-FG36-08GO88068

Appendix B. – Educational Materials

Appendix B.4.

History of Biodiesel at Messiah College Presentation



Early Biodiesel Processor Projects

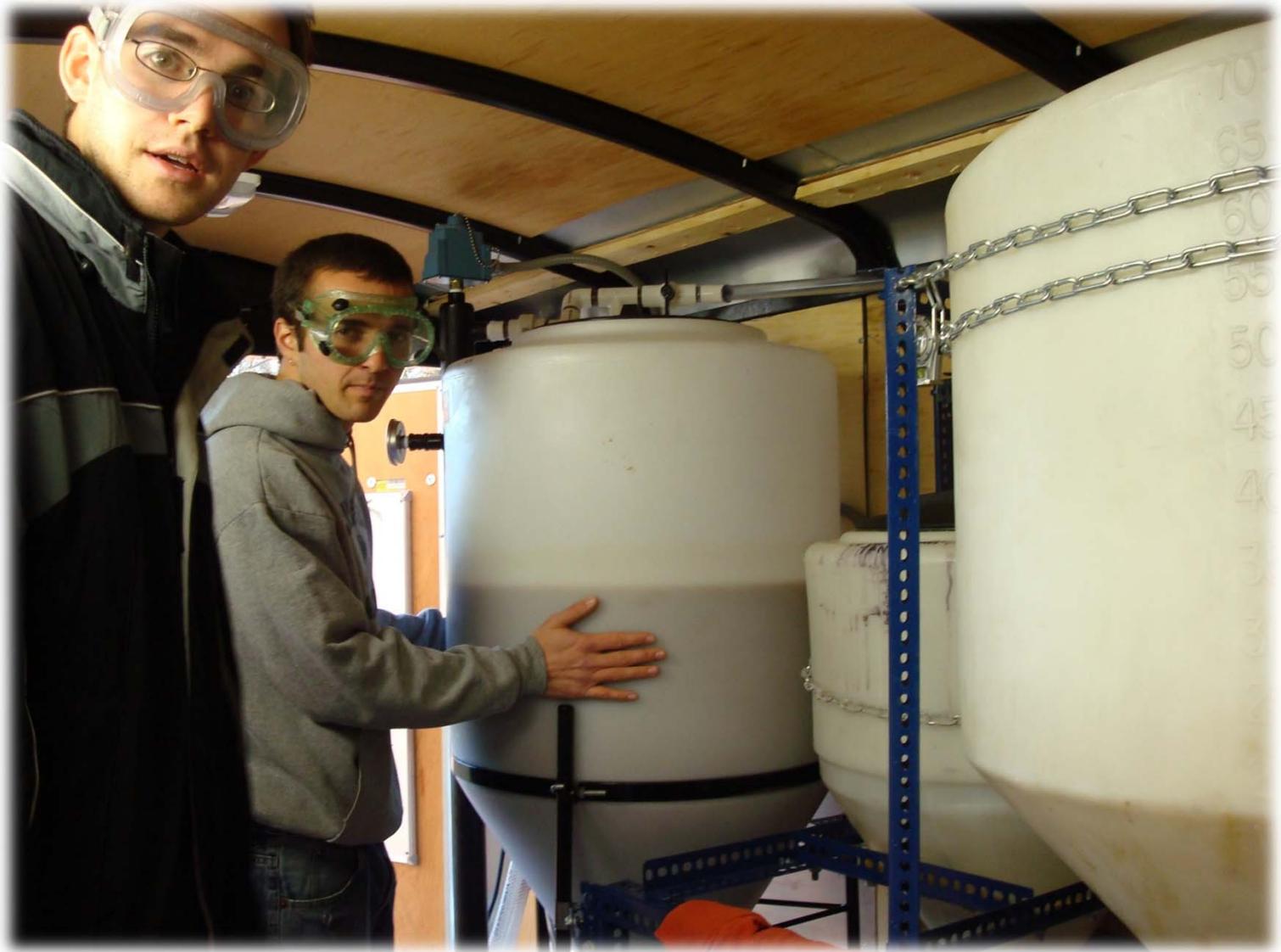


2005 PLC Processor



2007 PLC Processor





2008 Manual Processor





2008 Methanol Recovery System





2009 Manual Processor





2009 Manual Processor in Garage



2009 Methanol Recovery Test System





2010 Oil Purification and Processor





2011 Methanol Recovery System

Appendix C. – Articles and Publications

- C.1. – The Collaboratory Helps Fuel Pennsylvania’s Future
- C.2. – Biodiesel Fuel Production and Methanol Recovery
- C.3. – Messiah Engineering Students Educating the Community About Biodiesel
- C.4. – Automated Biodiesel Processor
- C.5. – College Grows Sunflowers for Food and Fuel
- C.6. – Flower Power: Messiah takes sunflowers from field to fuel
- C.7. – Sunflowers Key Part of Sustainability Project
- C.8. – Messiah College Used Flower Power to Save Money, Environment
- C.9. – Sunflowers Make an Impact
- C.10. – A Look into Messiah College’s Green Revolution: College Raising Bar for Sustainable Campuses
- C.11. – Fuel of the Future
- C.12. – Commercial Opportunities for Messiah College Biodiesel

Messiah College Biodiesel Fuel Generation Project

DE-FG36-08GO88068

Appendix C. – Articles and Publications

Appendix C.1.

The Collaboratory Helps Fuel Pennsylvania's Future



The Collaboratory Helps Fuel Pennsylvania's Future

The 93rd Pennsylvania Farm Show hosts a harvest of renewable energy, including Messiah's biodiesel project.

The Alternative Energy Harvest area of the Pennsylvania Farm Show is a relatively recent addition to the country's largest indoor agricultural display event. Here, at one end of the large Exposition Hall, students and staff from Messiah College's Collaboratory for Strategic Partnerships and Applied Research (the Collaboratory) set up a display of its small-scale biodiesel production process. While this is the first time that the Collaboratory has brought its biodiesel production trailer to the Farm Show, the team has been working over the last five years to perfect a process for converting waste vegetable oil from Messiah's dining facilities into biodiesel fuel for use in campus vehicles and as a substitute for petroleum heating oils.



A guided peek inside the trailer reveals that the basic process of creating biodiesel involves heating waste vegetable oil and the addition of methanol and either sodium or potassium hydroxide. A glycerol byproduct is allowed to settle out of the process and the resulting biodiesel is washed and filtered. Methanol is recovered from the glycerol byproduct to produce glycerin (glycerin can be used in soap and as a composting agent). The batch processor that the Collaboratory had on-hand at the Farm Show can produce 45 gallons of usable fuel at a time. In most cases, no modifications to diesel engines or fuel burners are necessary to use the biodiesel.

As can be gleaned from its name, a primary component of the Collaboratory's mission is partnering with organizations locally and world-wide to develop solutions which benefit disadvantaged people and care for the earth. Mike Zummo, an '06 Messiah College engineering graduate who manages the biodiesel project, explains, "We want to connect with members of the community who are interested in small scale [biodiesel] production to answer their questions as well as network with other local small scale producers." Since Pennsylvania's number one business is agriculture, the new frontier of growing and producing biofuels is no "small potatoes" for the farmers, businesses, and other partnering organizations within the Commonwealth. Exhibiting at the Farm Show allows students to connect with and help educate some of the Pennsylvanians that will benefit. Zummo says, "Participation in this project allows students to gain hands-on experience and apply the theories and principles learned in the classroom to a practical and socially relevant goal."

Following its initial success, the U.S. Department of Energy awarded the Collaboratory a grant for nearly \$500,000 last fall for further research and refining of the production process in efforts to meet the ASTM D 6751 Biodiesel specification while standardizing the process and equipment

design. The ASTM certification insures that the biodiesel meets certain established quality standards and that it can also be safely used in place of or blended with petroleum-based diesel fuel.



So, what special powers can biodiesel boast of that petro-based diesel fuel can't? Well, it's non-toxic and renewable, and it utilizes an organic product that was previously disposed of as waste. It also produces less carbon monoxide, unburned hydrocarbons, and particulate emissions than petro-based diesel. And, the production process has a positive net energy balance, which means, as Zummo points out, "that more energy is contained in the biodiesel produced than was required to produce the fuel." Not all alternative fuels can say that.





Rudolf Diesel would be proud of this turn of events with his legacy; his first engine operated on peanut oil and he continued to use and research biofuels until his death in 1913. If he were here today, he might even ask that the Collaboratory's trailer of biodiesel production equipment remain even after the rest of the Farm Show had packed up and left town—after all, the Pennsylvania Auto & Boat Show might just need a few gallons when they roll into the Exhibition Hall next week!

To Learn More:

[Collaboratory Biodiesel Research Project Pamphlet \(PDF\)](#)

[Collaboratory for Strategic Partnerships and Applied Research](#)

[U.S. Department of Energy Biodiesel Website](#)

Messiah College | One College Avenue | Grantham, PA 17027 | 717-766-2511

Comments or questions? Contact the WebMaster.

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Messiah College Biodiesel Fuel Generation Project

DE-FG36-08GO88068

Appendix C. – Articles and Publications

Appendix C.2.

Biodiesel Fuel Production and Methanol Recovery



ENGINEERING NEWS

Biodiesel fuel production and methanol recovery

by Mike Zummo

MARKET USE
(cafeteria fryer)



WASTE PRODUCTION
(collection)



BIODIESEL PRODUCTION
(small scale processor)



QUALITY TESTING



END USE
(campus vehicles)



Photos of current biodiesel recycling process at Messiah College by Mike Zummo '06

Researchers around the world are pursuing new forms of alternative energy to help fuel our world for the future, and Messiah College engineering students are doing their part. For the last six years, student teams from the Department of Engineering and the Collaboratory for Strategic Partnerships and Applied Research have been working to develop a process and processor systems for the production of biodiesel fuel.

Biodiesel is an environmentally friendly alternative to petroleum based diesel fuel used in diesel engines and as a fuel oil substitute. It is produced by a reaction that occurs when vegetable oils are combined with an alcohol and a catalyst. Its primary advantages are that it is an agriculturally based renewable fuel that is non-toxic and biodegradable. Biodiesel combustion produces less carbon monoxide, unburned hydrocarbons, and particulate emissions than petroleum based diesel. It is a closed carbon cycle fuel that emits no net greenhouse gasses.

The majority of the students' work has focused specifically on the conversion of waste vegetable oil into high quality biodiesel fuel. With great success, research and development teams have used the waste vegetable oil from campus dining

facilities and local restaurants to produce fuel for use in diesel powered vehicles and as a heating oil substitute. In September 2008, the College was awarded a Research and Development Grant from the United States Department of Energy to promote advances in small scale biodiesel production technology. This grant will provide the biodiesel research team with the ability to pursue American Society for Testing and Materials (ASTM) certification testing for biodiesel as well as extensive process and processor research and development



Summer 2009 interns Andy Derr '10 and Stephen Bray '10 work on methanol recovery.

opportunities. Currently the team is in the final stages of installing a biodiesel research and development testing lab in the basement of Frey Hall. This lab will house equipment that allows the team to perform the following quality tests required by the ASTM specification: total and free glycerin content by gas chromatography, acid number, cloud point, flash point, and water and sediment content. Upon completion of the testing laboratory, students will begin extensive testing of samples from the biodiesel processor to establish the quality of fuel being produced. Results of such testing will enable students to develop a small scale biodiesel processor that consistently produces ASTM certified biodiesel.

Another key aspect of the project involves working with the resulting waste streams. The glycerol byproduct of the biodiesel production process contains residual methanol which makes its uses limited. Implementing a methanol recovery system allows the methanol to be removed from the glycerol, leaving a safe glycerin byproduct that has many uses and provides recycled methanol for future production of biodiesel. Over the course of the last three years, student teams have developed a functional prototype methanol

continued on page 4

Biodiesel, continued from page 1

recovery system. In April 2009, the College was awarded a Seed Assistance Grant from the Innovation Transfer Network to provide funding for research, development, and further testing of the current methanol recovery prototype. Student researchers have been very successful in developing a safe and functional methanol recovery system which has produced 96% pure recovered methanol.

Messiah College engineering students are taking the principles and theories they

are learning in the classroom and applying them to the development of a more sustainable biodiesel production process. As they perform this applied research and development they gain a knowledge and understanding that they can then utilize to support local entrepreneurs and institutions interested in producing and using biodiesel. They can also take this knowledge to support our community partners abroad as they seek to establish sustainable sources of energy around the world.



Andy Derr '10 works on the biodiesel demonstration processor.

Messiah College engineering enters the world of "Rapid Prototyping"



Thin-walled duct



Gear tree



Flexible bellows

Advancements in computer aided design and modeling tools have dramatically streamlined the engineering design process. While these powerful tools allow us to create and analyze objects in the virtual world, there is still a need to produce actual physical prototypes for verification of form, fit, and function. Creating these detailed and dimensionally accurate prototypes is a time-consuming process requiring specialized fabrication skills that student engineers do not typically possess. While instructors do spend a good deal of time teaching students these skills, new manufacturing technologies that emerge will, at the same time, expand prototyping capabilities and shorten the learning curve. Rapid prototyping is one such emerging technology that has this potential.

Rapid prototyping, the means of creating a solid object directly from a computer model, is not new; in fact the technology has been commercially available since the mid 1980s. Not until recently, however, with the advent of a new class of smaller, less costly machines, known as 3D printers, has the technology come within reach of small commercial and educational users. The combination of maturing technology, lower equipment cost, and wide acceptance of 3D printing in industry make this an ideal time to jump into the world of rapid prototyping. With the help of a generous donation from Black & Decker Corporation, the engineering department recently took delivery of its first 3D printer from Z Corporation, Burlington, Mass.

Unlike traditional manufacturing technologies that shape an object by removing material from an existing solid, 3D printers build an object from the ground up, one thin layer at a time. There are a variety of methods used for 3D printing but all start by using software to "slice up" a computer model into thin (0.001"

to 0.010") cross sections. This data is then sent to the printer and printed out, one cross section at a time. The Z-310 printer that the department has ordered uses a plaster-based powder and liquid binder as the build media. To start the process, a thin layer of powder is rolled across a flat 8" x 10" build platform. A print head then traverses the build platform and dispenses binder onto the powder in the shape of the part cross section, much like printing ink on paper. In areas where the binder is dispensed, the grains of powder are fused together; all other regions on the build platform remain as loose powder. To start the sequence for the next cross section, the build platform drops one layer thickness, a new layer of powder is rolled out over the existing layer and the process is repeated using the next set of cross section data. The process continues fusing one layer on top of the next until the part is completed. Build times can take anywhere from one hour for small simple shapes to eight or more hours for large complex parts.

This 3D printing technology will enable students to create models with complex geometries that would have been extremely difficult or impossible with the department's previously existing capabilities. Even complex working assemblies can be created as one unit; the "gear tree" pictured above is one novel example of this capability. Making changes to a prototype also becomes easier with 3D printing technology. Instead of spending hours in the shop fabricating a new part, students simply alter the computer model, send it to the printer, start the print cycle and come back hours later to a finished part. Z Corporation technology also allows for the use of different building materials ranging from rigid materials suitable for use as sand casting molds to materials suitable for making the flexible bellows pictured above.

Whether it's creating a prototype motor coupling for the electric commuter vehicle, sand casting molds for the electric tricycle drive train, or helping students with complex geometry in Engineering Graphics, 3D printing technology will shorten the time to prototype and greatly enhance the overall learning experience related to practical project work by students here at Messiah College.

Messiah College Biodiesel Fuel Generation Project

DE-FG36-08GO88068

Appendix C. – Articles and Publications

Appendix C.3.

Messiah Engineering Students Educating the Community About Biodiesel

Kilo-Watt-hour Project Update

By Randy Fish

"It's been rainy for two days and the batteries have not fully charged, so take it easy on your power usage for today." Those were the instructions given to the 10 of us who traveled to Mahadaga, Burkina Faso, this summer by our missionary contact Matt Walsh '01.

But, what does that mean? Should I not use my fan tonight—its 95°? I bet Dr. Vader will use his. What does that mean for those at the clinic working off a different solar power station, also installed by Messiah? If the waiting room uses too much power, will nurses not be able to do their job?

What about students at the Center for the Advancement of the Handicapped (CAH) down the road using yet another Messiah installed solar power station?

If only there were a way to assign an amount of power to each room or each building and let the users figure out how

to ration themselves. Of course, you would want to be able to tell them how much power was left and shut them off if they went over their rationed amount. If only....

Well, we've just described the function of the KWhr project being designed by D. Allen '12, A. Dowling '13, leader K. Manieri '12, J. Martin '12 and N. Nichols '11. Along with students and faculty working on projects for disability resources (trike), energy (biodiesel) and education (summer enrichment program for CAH), Jon Martin and I delivered the first "working" prototype of the KWhr meter to our partners in Mahadaga.

Having the system in hand allowed us to iron out functional details for the expected first release in 2011. We were also able to plan out upgrades for the next generation.

After leaving Matt in Mahadaga, I visited with a missionary in Ouagadougou,



Burkina Faso, planning to start construction on a new Bible college outside the city. It turns out that they are also looking to run on solar power. Can Messiah engineering help? This new contact in Burkina Faso had also heard about the KWhr meter. "Will that be ready for us to use, as well?" he asked.

We are still writing code, and it looks like we are going to need to go into production already. Welcome to Messiah engineering.

Mobility, continued from page 1

strength God provides, so that in all things God may be praised through Jesus Christ. To him be the glory and the power forever and ever. Amen."

This year students we were able to help provide mobility for a 16 year old girl who had never traveled on her own before in her life, a tricycle for a man injured in a motorcycle accident and continued freedom for a 19 year old young man with cerebral palsy who is now running his own business thanks to the staff at the CAH and his tricycle. To God be the Glory....

WERC, continued from page 2

need testing to obtain further feedback. A "real workplace" test is planned for early in this school year. Procedures for this test are under development, including survey and feedback questions. The coaching manual will also be further revised. The current slate of students and faculty on this project appear on the Collab Comm Group wiki page at thecollaboratoryonline.org/wiki/Communications_Group.

Messiah Engineering Students Educating the Community about Biodiesel

by Mike Zummo



Junior mechanical engineering student Michael Hahn at the 2010 PA Farm Show

Producing, testing and using biodiesel have become common activities for the students, faculty and staff involved in the Biodiesel Research Project at Messiah College. Over the course of the last seven years, student project teams have worked to develop a

successful small-scale production system able to produce consistently high-quality biodiesel.

Through a grant received in 2008 from the U.S. Department of Energy, the project team was able to develop a cutting-edge biodiesel quality testing lab which has greatly increased the capacity for research and development in the area of small-scale production. Just this summer, student interns produced more than 500 gallons of high-quality biodiesel from waste vegetable oil collected from campus dining facilities. However, through all of this work, the team has discovered that biodiesel is not as commonly understood by most people as it is by team members, and the team wants to change that.

Educating the campus and local community about biodiesel, including what it is, how it is made and why it is important,

Biodiesel, continued on page 4

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ENGINEERING NEWS • FALL 2010 OUR MISSION

The mission of Messiah College is to educate men and women toward maturity of intellect, character and Christian faith in preparation for lives of service, leadership and reconciliation in church and society. Graduates of the engineering program will therefore be technically competent and broadly educated, prepared for interdisciplinary work in the global workplace. The character and conduct of Messiah engineering graduates will be consistent with Christian faith commitments. We accomplish this mission through engineering instruction and experiences, an education in the liberal arts tradition and mentoring relationships with students.

Biodiesel, continued from page 3



Senior Mechanical Engineering Students Jordan Beckler and Jeremy Miller guide a student in hands on biodiesel workshop.)

has become one of the key components of the work that the team is doing. This work has been going on for several years through team members attending different festivals and events to present their work with biodiesel.

The Pennsylvania Farm Show has been one of the most successful events

where the team has presented. At the 2009 Farm Show, the team displayed their mobile biodiesel processor and samples of waste vegetable oil, biodiesel, glycerin and many others materials associated with biodiesel production in the Renewable Energies Harvest Exhibit. They also distributed information about the biodiesel program, The Collaboratory for Strategic Partnerships and Applied Research, the Department of Engineering and Messiah College in general.

During the 10-day show, the team connected with more than 600 people about biodiesel and the work being done at Messiah College. A local cable news station filmed and aired a tour of the mobile biodiesel processor and a podcast was recorded about the importance of biodiesel by a Pennsylvania-based non-profit group promoting alternative energy.

Based on its past success, the team also attended this year's 2010 Pennsylvania Farm Show with a new and improved exhibit. They displayed a newly developed 5-gallon biodiesel process specifically

designed for educational demonstrations. Another addition was a multimedia presentation displaying the biodiesel production process and highlighting the testing capabilities of the quality lab.

The exhibit at the 2010 Farm Show was also extremely successful, and the team is currently preparing an exhibit for the 2011 Farm Show this coming January.



Senior Mechanical Engineer Student Jordan Beckler explains elements of titration to a student at a biodiesel workshop.)

Messiah College Biodiesel Fuel Generation Project

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Appendix C. – Articles and Publications

Appendix C.4.

Automated Biodiesel Processor



ENGINEERING NEWS

Automated Biodiesel Processor

By Mike Zummo

After almost three years of research, development and testing, the Biodiesel Research project has made a number of great strides in perfecting the process of small-scale biodiesel production. The student research team is currently collecting all of Messiah College's waste vegetable oil, purifying it through a centrifuge, converting the oil into biodiesel and processing all of the waste glycerin through a methanol recovery system. The culmination of all of this work has led to the development of an automated biodiesel processor that creates up to 25 gallons of biodiesel per batch with very minimal user input.

Senior electrical engineering student Philip Martinez '12 has spent the last year and a half working to develop the mechanical, electrical and software components of this new automated processor. Building on the work of several students before him, Martinez has created a functional prototype of a Programmable Logic Controlled (PLC) biodiesel production system. The PLC



Back: Jordan Beckler '10. Front, from left: Philip Martinez '12 and Michael Hahn '12 by the weather containment protecting the 55-gallon waste vegetable oil drum from rainwater.

processor performs all of the necessary reactions, washing, drying and waste removal steps required to make high-quality biodiesel with virtually no required user input along the way. After a user inputs the necessary reaction elements and turns the system on, the system is programmed to perform each step on its own. If that is not impressive enough, Martinez has also designed a number of safety features into the system that will completely shut the processor down in the case of overheating, over pressure, power interruption, heater failure or pump failure.

Continuing in his effort to make the PLC processor both safe and user friendly, Martinez spent many hours learning to use Solid Works to create a process schematic that he then engraved on the control panel using a CNC mill. This schematic allows the user to approach the system at any

The culmination of all of this work has led to the development of an automated biodiesel processor that creates up to 25 gallons of biodiesel per batch with very minimal user input.



Automated biodiesel processor with engraved schematic

Automated continued on pg. 2

Chair's Corner



This fall, some interesting people are showing up in the Engineering Department. When Dr. Gray looks out over the Introduction to Engineering class this year, he sees 74 new engineering students (about 18% female). Most of these eager faces are new to Messiah; students tell us that they came here

because they wanted to study at a school with a Christian world view and because of the strength of the program. Some are existing Messiah students changing to engineering from majors as diverse as biology, psychology and Spanish. While computer, electrical and mechanical concentrations attract the majority of these new students, a sizable and growing number tell us that they intend to choose one of our newer concentrations in biomedical or environmental engineering. At the end of September, we will host another group of interest-

ing people: the accreditation review team from ABET. Every six years, the department conducts a self-study and hosts a visit team as a part of the continuing accreditation process. The self-study has been conducted and the report has been submitted. Now it's time to meet with the review team. We enjoy interesting people. You are welcome to stop by anytime.

—Randy Fish, professor of electrical engineering

Automated continued from pg. 1

point and know exactly where it is in the processing cycle. Through a series of indicator and warning lights, even a relatively untrained operator can follow the schematic to determine exactly what the system is doing and if there are any problems. While the system is completely automated, each component does have a manual override switch that allows the user to take complete control if necessary.

Currently, the PLC processor is being tested to optimize system set points and to bring to light any problems in the control system. Over the course of the next two semesters, Martinez will be rigorously testing the system to continue to perfect its processing ability and safety. Though it is still in the development stage, this processor has successfully produced a number of batches of high-quality biodiesel.



Engineering Student Philip Martinez '12 works on the automated biodiesel processor control panel.

“Since the fall of 2010, engineering students have been collecting and processing all of the waste vegetable oil generated by Messiah College dining facilities into biodiesel. The goal of the automated processor is to reduce the time and labor required to convert this oil into biodiesel.”

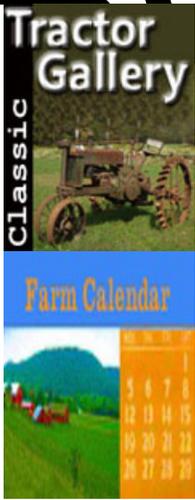
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Appendix C. – Articles and Publications

Appendix C.5.

College Grows Sunflowers for Food and Fuel



College Grows Sunflowers for Food and Fuel

Like

2

10/15/2011 10:00 AM
By Chris Torres Staff Writer



GRANTHAM, Pa. — Here in the Keystone State, you would be hard pressed to find sunflowers dotting the landscape.

While visually appealing, there aren't many farmers able to find a suitable use for them.

Researchers at Messiah College are hoping to change that, though.

This summer, five acres of sunflowers were grown as part of a project to study the feasibility of using sunflower oil as a food and biofuel source.

Mike Zummo, biodiesel project manager, said the goal is to use sunflower oil in the school's dining facilities, collect the waste and convert it into a biodiesel.

The school has been working on biofuels research for the past 10 years, but Zummo said a \$492,000 grant awarded to the school by the U.S. Department of Energy in 2008 enabled researchers to apply what was learned in class and to get an actual project off the ground.

"It's a decision that we came to last fall," Zummo said. "We knew what we were capable of with biodiesel production. We knew what our students were interested in and wanted to take a chance with this sunflower trial to see if it were something that could take off."

The money enabled the school to purchase materials along with a small seed press that will be used to press the oil from the sunflower seed.

The school worked with area farmer Lynn Wingert on obtaining seed and planting the crop.

Wingert, who rents ground around the Mechanicsburg area and grows mostly cash grain, is also a Pioneer seed salesman. He was able to get sunflower seeds for the project and helped plant the seeds on five acres of ground that he had been farming, but is owned by Messiah College.

The crop was harvested last week with a New Holland corn combine.

"We'd like to potentially show scalability, to show what we've done on five acres and be able to extrapolate that on 10 or 15 acres,"

Messiah College Biodiesel Fuel Generation Project

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Appendix C. – Articles and Publications

Appendix C.6.

Flower Power: Messiah takes sunflowers from field to fuel

ZOOM IN

CLOSE-UP



NEWS

Flower Power: Messiah takes sunflowers from field to fuel

Five acres of sustainability serve campus in myriad ways



"Really, the Sunflower Project and the biodiesel project are one and the same ... a truly full-circle process of sustainability."

—Mike Zuzano '08, the Collaboratory's biodiesel project manager



Take 10 years, 25 pounds of seed, planted, five acres of land and a lot of passion. What do you get? One of Messiah's newest sustainability ventures, the Sunflower Project. Using the seeds from five acres of sunflowers that were planted this summer, students and faculty involved in sustainability groups on campus and the Collaboratory's biodiesel project will press oil for the College's cooking needs. That oil will then be recycled into biodiesel that will operate campus tractors.

"Really, the Sunflower Project and the biodiesel project are one and the same," said Mike Zuzano '08, the Collaboratory's biodiesel project manager. Demonstrating how, as a campus, Messiah can use resources to meet multiple needs, the project is what Zuzano calls "a truly full-circle process of sustainability."

Though the project is just coming to fruition, with its first harvest occurring this fall, the plan dates back a full decade with the advent of the Collaboratory's biodiesel project. The advances and successes in creating biodiesel in these 10 years led to conversations between Zuzano and Messiah's sustainability coordinator Craig Daken about where the project could possibly go next. "Last year, Mike Zuzano and I were talking and saying, 'We've got the biodiesel part figured. Now where do we get our oil from?' That started a series of conversations

about growing oil-bearing crops and who would grow that for us," said Daken.

After being approached by Zuzano and Daken about the project, local farmer Lynn Winger agreed to become the primary caretaker for the fuel crop. "The sunflowers were planted right over last year's residue in central Oregon," said Winger.

The wet weather this fall in Grandhuz pushed back the harvest date from the end of September to the beginning of October, which also opened the door for more damage from birds. However, Daken reports that birds were partly kept in check by an unexpected fire resistance. "Two Cooper's hawks moved in and were guarding the fields for us," he said. With no serious crop loss due to insects or animals, and only minor thinning of plants in one acre due to poorly drained soil, the projected yield of oil from the sunflowers will be nearly 500 gallons.

Daken reports that they will also be looking for ways to use all parts of the plants, not just the seeds. Prosoak, the by-product that remains after oil is pressed from the seeds, is one option. "Farmers are interested in it as a feed supplement for livestock," Daken said. Since this is the project's pilot year, many aspects are in the experimental stage, which allows for some hands-on problem solving and brainstorming for students.

"As a student who is passionate about the



environment, it is important for me to know that my college supports what I am studying in class, so much so that they are taking action," said Kristin Lister '12, an environmental science major and president of Earthkeepers, Messiah's environmental club. "What has been most exciting for me is the opportunity to be involved in these changes and to see the enthusiasm other students have for getting involved in what the College is doing."

Students were more directly involved with it through the Environmental Issues Interns course," said Daken. "One of the course professors overseeing the pressing of the seeds and the delivery of the oil." The Environmental Issues Interns course students worked closely with the Collaboratory and engineering students who designed and constructed the pressing system.

With plans to continue the crop and, hopefully expand to cover all of the College's cooking oil needs, one thing Daken says that students will be researching is how to maintain the nutrient content of the soil. Planting the same crop over and over can deplete the soil of certain nutrients and wear it out, so crop cycling and soil renewal is key. One option being considered is composting. "We currently have a student doing an honors project on vermiculture, which is using worms to compost food waste, and that

could be applied to this field," said Daken. An initial harvest was conducted by hand Oct. 1, with 30 students coming out to the fields and gathering enough seed to fill one 55-gallon drum—an amount that allowed for a first oil pressing. The rest of the seeds were harvested later that week by combine.

Paul Nickerson '13, a member of Earthkeepers who co-managed the Community Garden this summer, notes that the project is one that opens the door for many disciplines to get on board. "One of the most interesting parts of the Flower Power project for me has been to see how numerous fields of study have managed to work together to promote and achieve this project," said Nickerson. "From biology and engineering, to chemistry and public relations, there is not a field of study that cannot be utilized in forwarding sustainability on campus."

—Allyson Long '12

Watch the video of the Sunflower Project at www.messiah.edu/watch?v=0h10wF-J5Hc.



The acres of sunflowers were planted this summer, and volunteers harvested the seeds by hand Oct. 1. The seeds were then pressed into oil, which was used for cooking at Little Nelson Dining Hall. The Collaboratory's Biodiesel Group then collected the used sunflower oil to be used in diesel maintenance vehicles on campus.

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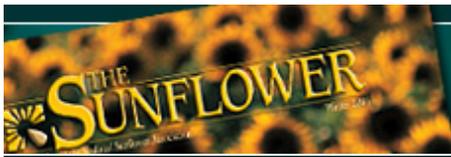
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Appendix C. – Articles and Publications

Appendix C.7.

Sunflowers Key Part of Sustainability Project


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SUNFLOWER MAGAZINE

Sunflower Key Part of Sustainability Project

November 2011

The “food vs. fuel” debate has become increasingly important in today’s marketplace. A unique project at a liberal arts college in Pennsylvania is taking it in a notably different direction: to “food and fuel.”

Sustainability is not only an obligation toward the environment, it’s an investment in the future. At Messiah College in Grantham, Penn., the concept is being translated into the “Sunflower Power Project.” In this trial year, five acres near campus (above photo) were planted to sunflower and recently harvested. The seeds will be pressed and the oil used in the campus dining services. The used oil will then be converted into biodiesel for use on campus.

This pilot project is a joint venture with the Office of Sustainability, Biodiesel Projects and the on-campus dining facility. Mike Zummo, Messiah College’s Biodiesel Project manager, says the goal is to create a full-cycle energy plan that begins with sunflower and ends with biodiesel.

“It’s about creating a closed loop. We produce the product, process it, use it for food, and then bring it back around and turn it into biodiesel that we use on campus as a source of fuel for the press (for next year’s crop) and other areas on campus,” Zummo says. “We’re really getting double the value with our oil in food and fuel.”

With the help of a local farmer, Zummo and Craig Dalen, the college’s sustainability coordinator, arranged for the five-acre plot to be planted to sunflower. Lynn Wingert has been renting and farming the college’s land for almost 40 years, so he knew the territory. But this was his first-ever venture into sunflower. He says he’s always been open to trying new things over the years, but admits he went into this optimistically, but with “more than a little trepidation.”

As any farmer can relate, the sunflower crop has unique challenges that need to be addressed. Wingert, an independent sales representative with Pioneer, tapped various resources, with most of the information coming from the National Sunflower Association website. He consulted with his company’s supply people to come up with a short list of hybrids that would work for the area, and they decided on an ExpressSun® variety.

Wingert says his main concern at first was weed control. The field had been no-till corn for 15 years. Despite clean fields going in, Wingert wasn’t sure what special considerations would come with the sunflower. He applied some fertilizer early in the season with the pre-emergence herbicide package of Dual II Magnum, Prowl H2O and Gramoxone, plus another boost of starter fertilizer at planting. He used his corn planter to plant the sunflower in 30” rows with a target population of 20,000.

Aside from a sharp learning curve when it came to planting sunflower, Wingert says

made from a steep learning curve when it came to planting sunflower, Wingert says that the unusual 2011 weather pattern in the area was probably the most limiting factor. An extremely wet spring caused planting to be pushed back to mid-May. A few days after planting, a heavy rain took a toll on the stand. This was followed by 100-degree summer heat. In the early fall, just when the plants looked mature, excessive rain to the tune of 18 inches over a 20-day period hit the area, delaying harvest significantly.

Despite the challenges, the field was harvested on October 7, using a standard grain head, which all involved recognize was certainly not the ideal. "It was a bit frustrating," says Wingert about the harvesting process. "But we all knew that for five acres, we couldn't invest in specialized equipment. From the information we gathered, everyone said it could be done with a grain head, but just to expect more loss."

Wingert says the project is likely to expand next year, and he's happy to continue his involvement in new and innovative approaches. He's heard rumblings "though the grapevine" of some farmers in the area considering sunflower. "I see the double-crop option as a possibility in this area," he says.

This is likely due to the high visibility of the project, both from a campus and a community standpoint. The field is located right at the campus entrance near the college president's house (photo). "I joked with the guys (Zummo and Dalen) that maybe we should hide the field over the hills somewhere since this was our first try," Wingert says, concerned that the trials and struggles would be on display. But much to everyone's surprise, the field turned into a sort of billboard advertisement for the project.

"At the very sight of sunflower, we generated awareness across the community with the crop," adds Dalen. "We are not an ag school, but you ask anyone on campus about the sunflower project and they will know about it."

Total yield of the five-acre field was around 7,500 lbs. Now that the seeds are safely in the gravity wagon, work for Zummo begins to unfold. This year's crop will go through cold-press equipment on campus and then be used in the dining hall. It is estimated that one acre of sunflower will garner about 100 gallons of cooking oil. "It's within our reach," he says, "to someday reach 30 acres with this project to take care off of all on-campus dining hall needs." The used oil will then return to Zummo and his biodiesel team and be converted into fuel to power things on campus — including the presses for next year's crop — coming full circle.

With help from a U.S. Dept. of Energy research grant, he has been converting cooking oil into biodiesel on a small scale for more than 10 years. For over a year, they have been collecting the dining services waste oil (about 3,000 gallons of vegetable oil) and putting that through a settling process. After sediment and water are removed, the oil is sent through a centrifuge to spin out more of the heavy sediment to get cleaner oil. The oil is then further processed to generate the biodiesel.

Zummo and Dalen are also looking into utilizing another by-product of the crush — the sunflower "cake" — for local farmers to feed to their livestock.

Future of the Project

In recent years, sustainability has grown from a mere suggestion to a world-wide revolution. It is the capacity to endure or survive. This capacity, directly or indirectly, depends upon our natural environment. More recently, sustainability has emerged as a result of significant concerns about the environmental and economic consequences of rapid population growth, economic growth and consumption of our natural resources.

"This project was collaboration more than anything that involved the campus, the community and the farm," Dalen says. "We've created a connection for the

community and the farming sector,” Dalen explains. “We generated attention for the sustainability project; but we also used this as an opportunity to learn, foster stewardship and build community.”

For long-term goals, both Dalen and Zummo say they look to further educate the students to put the project in their hands to take out into the world. Part of the reason Zummo pushed for on-campus oil presses was born from a visit to a small country in West Africa. His vision is to prove the concept of using oil for cooking and fuel, and then take that knowledge to other underdeveloped countries.

Dalen agrees, saying his hope is that the project’s reach goes far beyond sustaining the campus needs for healthy cooking oil and fuel oil. One of Dalen’s major goals was to involve the students in a hands-on approach. That included a small group of students who hand-harvested a few rows in order to foster ownership in the project. This group was made up of students who are sustainability studies majors and some from Dalen’s Environmental Issues course, which fulfills a requirement for general education. Putting a call out through student organizations enlisted other volunteers and a number of students who came out to help because they were interested in the project.

“This is about student education, community building and inspiring others with these types of projects,” Dalen says. “This is a developmental model.”

— **Sonia Mullally**

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Appendix C. – Articles and Publications

Appendix C.8.

Messiah College Used Flower Power to Save Money, Environment



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York County

Messiah College uses flower power to save money, environment

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Jonathan Bert and 131 others

Posted: Jul 21, 2011 10:12 PM EDT
Updated: Jul 22, 2011 12:17 PM EDT

By Al Gnoza - bio | email

GRANTHAM, Pa. (WHTM) - Messiah College has been around for over 100 years, but they're trying something for the first time this year. The school is a couple of months into what they are calling, "The Sunflower Power project."

The project is Messiah's latest effort in a commitment to sustainability, which is the discipline of using natural resources without destroying the ecological balance of an area.

The school is growing five acres of sunflower plants. They plan to squeeze the oil out of the sunflower seeds, then use that oil to cook with at the campus dining hall. They will then use the used cooking oil to fuel campus vehicles.

"From the field we hope to get about 500 gallons of oil and from that we'll lose some in the cooking process," said Mike Zummo, Messiah's Biodiesel Project Manager. "We'll probably get about 400 gallons we can make bio-fuel with."

The college has been recycling its cooking oil for the last three years to power some of its vehicles and heat some of its buildings. This, however, is the first time the school will grow the fuel source.



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 - Police: Woman took pickup from t baby behind**
 - 'Suspicious man' was watching Er children, officials say**
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Messiah College Biodiesel Fuel Generation Project

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Appendix C. – Articles and Publications

Appendix C.9.

Sunflowers Make an Impact

Sunflowers Make an Impact

By [Nick Tay](#)

Student Writer

Published: Wednesday, September 28, 2011

Updated: Wednesday, September 28, 2011 18:09

Over recent years, sustainability has grown from a mere suggestion to a campus-wide revolution at Messiah College and now the college is taking another step forward by introducing the "Sunflower Power Project."

This pilot project is a joint venture between the Office of Sustainability and the Collaboratory. It is headed by Craig Dalen, Messiah College's Sustainability Coordinator, Mike Zummo, Biodiesel Project Manager for the Collaboratory, and Student Project Director Dawnique Shury. Dalen says they plan to create an energy "full cycle" (an energy cycle that produces clean energy and produces no harmful byproducts), that begins with sunflowers and ends with biodiesel fuel.



The sunflowers are grown on a five-acre plot close to President Kim Phipps' house. Dalen estimates that "one acre of sunflowers will yield 100 gallons of cooking oil, which will help offset the 3,000 gallons needed per year at Lottie."

Once the sunflowers are mature, they are harvested and their seeds are pressed for oil using machines run by the biodiesel. Lottie then uses the oil for cooking, especially deep frying.

"Our goal is to have all the cooking oil in Lottie be from sunflower oil and then recycling the same oil into biodiesel," Shury said.

The other by-product of the seed pressing process – the sunflower shell "cake" – is not forgotten either. The protein-rich shells will be given to local farmers as feed for their livestock.

Dalen said that after use the used cooking oil is ready to be converted to biodiesel, which is sent to Zummo and his biodiesel team. Once in biodiesel form, the oil is mixed with other fuels and is ready to power the "gators" (golf carts) on campus -- and the machines that will press next season's sunflower seeds.

While Dalen said that he and Zummo are in charge of the project right now, he believes that student involvement is key.

"We're here to prove that this pilot project works," he said. "However, the students are the ones responsible for the future of the sunflower project."

Currently, a number of students are involved in the program: several sustainability majors are in the process of writing federal grant proposals, while others plan to take sunflower seeds and grow them in their rooms .

Messiah College Biodiesel Fuel Generation Project

DE-FG36-08GO88068

Appendix C. – Articles and Publications

Appendix C.10.

A Look into Messiah College's Green Revolution: College Raising Bar for Sustainable
Campuses



Home > News > Cumberland County

A Look Into Messiah College's Green Revolution

College Raising Bar for Sustainable Campuses

Comments 1 Share 3 Recommend 42



7/26 Messiah College Goes Green
7/26 Messiah College Goes Green
(2:08)



Eric Gemmell
6:39 p.m. EDT, July 26, 2011

- Topics**
- Colleges and Universities
 - Agricultural Research and Technology
 - Elections

Grantham (Cumberland County) — From sunflower seed oil to solar thermal heating systems, Messiah College is rapidly embracing the green revolution.

Over the course of the summer, several major renovations have transformed the Cumberland County campus.

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Adjacent to the college president's home, five acres of sunflowers dot the landscape.

According to Sustainability Project Manager, Mike Zommo, it's been a project in the making for ten years.

"It's taken up to this point to grow a crop this big and to harvest the seeds."

Zommo said the seed oil will be used two fold on the campus; the seed oil will be used in the cafeteria for cooking, then recycled and used for bio-fuel.

Across campus, Messiah's sustainability efforts continue. Crews are applying the finishing touches to a solar thermal heating system on a college dorm.

Craig Dalen, Messiah Sustainability Manager said, "When you turn on the shower or the sink you get hot water heated by the sun."

Jennifer Gardner, a Messiah Sophomore said the campus' green momentum is "Fantastic."

She added, "Sustainability is becoming predominant and necessary."

Messiah's green movement has spawned so much interest they have now students can major in Sustainability.

You can see all of Messiah's current and past sustainability project by visiting [Green@Messiah](#).

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Beth Lorow at 9:21 AM July 28, 2011

We appreciate FOX's interest in Messiah College's sustainability efforts. There were a few errors in the story, however, that I would like to correct on behalf of the institution.

Mike Zummo (not Mike Zommo) is the College's Biodiesel Project Manager, not Sustainability Project Manager. Also, the student referenced and quoted is Jamie Gardner, not Jennifer Gardner, and the new major is actually called "Sustainability Studies."

Messiah College is located in Grantham which is a village in Upper Allen Township, Cumberland County. Grantham is not a township, as indicated in the video.

Messiah College Biodiesel Fuel Generation Project

DE-FG36-08GO88068

Appendix C. – Articles and Publications

Appendix C.11.
Fuel of the Future

Fuel of the Future

By [Morgan Lee](#)

Published: Wednesday, February 16, 2011

Updated: Wednesday, June 29, 2011 11:06

When Craig Dalen and Mike Zummo collaborate they cannot stop talking. The Sustainability Director and Biodiesel Grant Manager, respectively, discuss a future of big, bright yellow sunflowers on Cemetery Hill that produce sunflowers seeds to produce oil to fry food in Lottie Nelson, whose leftover oil becomes biodiesel, which fuels campus gators roving around on campus. Even as they have this conversation, they realize that as they speak and dream, they are beginning to reap a harvest that was planted almost a decade ago. Around ten years ago, several engineering students used their senior project to begin researching ways to make biodiesel from vegetable oil--gas prices had begun to increase, and an interest in alternative energy sources was steadily rising. Given the accessibility of vegetable oil, it seemed like a natural progression to work with this compound.

The students worked out a process where vegetable oil would be collected and filtered to rid it of any food contaminants. First it would be tested to determine the free fatty acid content or acidity of the oil. The percentage of this content determined how much catalyst needed to be added in order to break down the vegetable oil. From there, the oil would be heated at 140 °F and mixed with methanol and potassium hydroxide, which acted as the catalyst.

Oil is a triglyceride compound meaning for every one glycerin molecule, there are three fatty acid also attached. The methanol breaks this bond, separating the glycerin from the fatty acid chains, creating methyl esters aka biodiesel. Learning how to not only create but also perfect this process was not accomplished overnight.

"Really, it took us around five to ten years before we finally got to the point where we were really good at making biodiesel," said Mike Zummo, the Biodiesel Grant Coordinator. "There was no strong student structure and so every year we would have to start over."

In order to make the project more sustainable, the project later became an

engineering project and also moved to the Collaboratory. In 2008, Messiah College received a federal grant from the Department of Energy for its work in biodiesel. Zummo was hired in order to manage the research of the half million dollar grant and also began tackling other related projects.

Recently, students have been working to extract the useable methanol remaining in the glycerin after it has been separated from the fatty acid methyl ester.

"Methanol recovery is a critical part of the biodiesel process and we're really ahead of the curve when it comes to doing it because we've had a good processor," said Zummo. "We're head and shoulders above everyone else."

The college currently uses vegetable oil collected from Dining Services and Baker's Diner to convert into biodiesel this spring will begin to fuel campus gators. But in an effort to become even more sustainable and cut further down on costs, such as money that Dining Services spends annually on vegetable oil, students have also begun to do research on pressing crops and extracting oil from seeds, looking for what type of processes need to happen for the oil to be usable in the kitchen.

The sunflower project is a natural progression, where the plants generating the oil themselves would be grown on campus. Zummo and Dalen have brainstormed planting several acres of Messiah College property with sunflowers whose seeds would be harvested to make cooking oil which would later be transformed into biodiesel.

"We could potentially make 100 gallons of oil per acre of sunflowers," said Zummo.

Although soybeans would also be able to do the job, they would lack the beauty of these flowers. "Imagine, on Cemetery Hill, late summer and early fall, all the sunflowers welcoming people back to campus and seeing the fields," said Dalen. "We get a high yield of oil and aesthetics."

In the future, if this system is successfully implemented, Dalen and Zummo have ideas to one day warm oil-heated buildings such as Hoffman and the Agape Center with it as well. Zummo would also like this to go abroad.

"We one day want to take project abroad to communities in Africa that have no ways of producing energy and teach them how to make biodiesel fuels and power vehicles," said Zummo.

Simultaneously, both Dalen and Zummo have been focused on bringing awareness to what the biodiesel program is doing.

"My goal and vision is to share this through education both here on campus but also

with the local community. I've personally shared with Harrisburg and Mechanicsburg schools and we're working with Milton Hershey right now," said Zummo. He continued, "We want to share what we learn and not just benefit ourselves."

"It's the type of project you sit back and ask why aren't more people doing this?" said Dalen.

Mike Zummo welcomes anyone interested in learning how to make biodiesel or wanting a tour of the facilities to email him at MZummo@messiah.edu.

Messiah College Biodiesel Fuel Generation Project

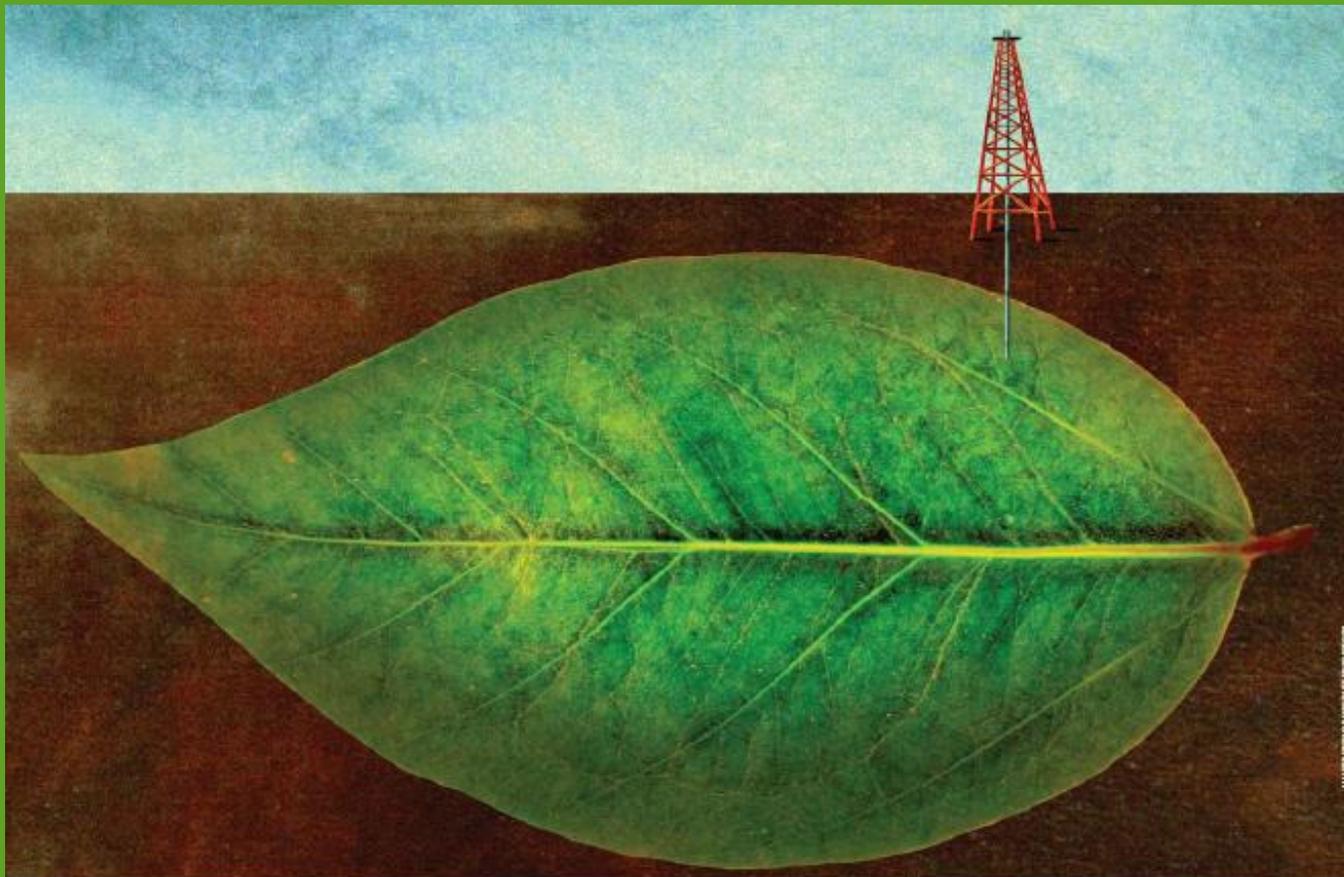
DE-FG36-08GO88068

Appendix C. – Articles and Publications

Appendix C.12.

Commercial Opportunities for Messiah College Biodiesel

MESSIAH COLLEGE BIODIESEL RESEARCH PROJECT



Innovation Transfer Network
Engineering Forum



6/24/11

A FULL CIRCLE PROCESS



FEEDSTOCK CROPS



SEED OIL PRESSING



COOKING APPLICATION



RECYCLING WASTE TO FUEL



STORAGE AND FILTRATION



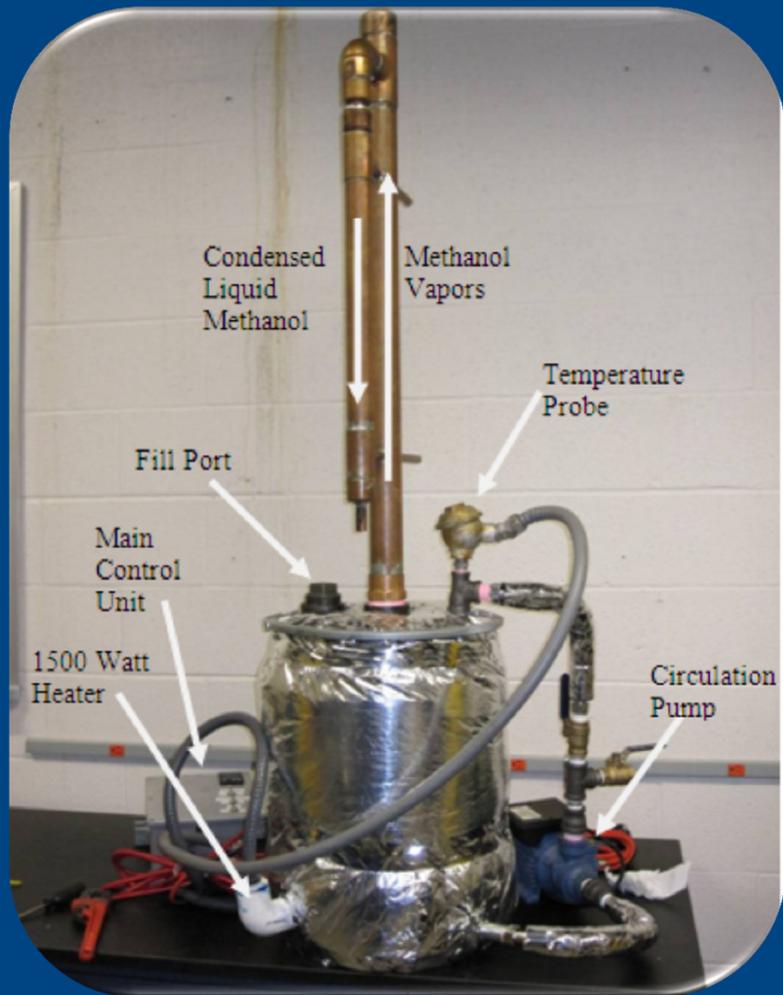
BIODIESEL PRODUCTION



PROGRAMMABLE LOGIC CONTROLLED (PLC) PROCESSOR



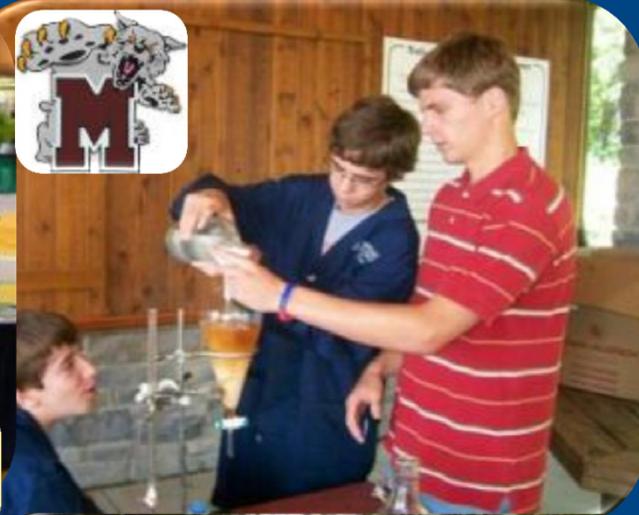
METHANOL RECOVERY



COMPLETED METHANOL RECOVERY SYSTEM



EDUCATION



QUESTIONS?

Messiah College Biodiesel Fuel Generation Project

DE-FG36-08GO88068

Appendix D. – Manuals and Reports

Appendix D. – Manuals and Reports

- D.1. – 2009 Methanol Recovery Report
- D.2. – 2009 Methanol Recovery Seed Assistance Grant Report
(Includes 2010 Methanol Recovery Manual)
- D.3. – 2010 Biodiesel Final Report
- D.4. – 2010 Biodiesel Production Manual
- D.5. – 2011 Centrifuge Testing Report
- D.6. – 2011 Methanol Recovery Report
- D.7. – 2012 Biodiesel Production Manual
- D.8. – 2012 Centrifuge Manual
- D.9. – 2012 Methanol Recovery Manual
- D.10. – 2012 Pressing Manual
- D.11. – 2012 Automated Processor Manual
- D.12. – 2010 Biodiesel Quality Testing Final Presentation
- D.13. – 2010 Methanol Recovery Final Presentation

Messiah College Biodiesel Fuel Generation Project

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Appendix D. – Manuals and Reports

Appendix D.1.

2009 Methanol Recovery Report

Biodiesel: Methanol Recovery

Project Final Report



Jacob Munson

Biodiesel: Methanol Recovery Project

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Biodiesel: Methanol Recovery Project Abstract

This project aims to make biodiesel production more sustainable and economically viable by recovering unused methanol from waste glycerin. We constructed a reflux column still to boil off, then condense, methanol from waste glycerol. Purity of the recovered methanol is critical to its reuse in biodiesel production. The remaining glycerin was tested as a fire starter and soap product. Further research and experiments will create a methodology to recovering methanol and its recycling.

Introduction

The biodiesel team has always been committed to sustainability and energy conservation. A byproduct of the biodiesel production process is glycerin with waste methanol in it. This combination of liquids has to be disposed of as a hazardous material due to methanol's toxicity. The biodiesel team decided to build a methanol recovery system to extract methanol from waste glycerin. To date, a methanol recovery system has been built and tested; several gallons of methanol have been recovered. The future acquisition of testing equipment will lead to the analysis of both biodiesel made from recovered methanol and the recovered methanol itself.

Background

Context

Many backyard biodiesel producers are unaware of the harmful side effects that disposing of glycerin with methanol in it can pose. Ground water contaminations as well as soil contamination both result from such disposal. Our biodiesel team built this project as an informative medium and model example to the commitment of sustainability. Not only does this project seek to inform many biodiesel producers, it proves that such consciousness is possible on a relatively small budget. Recovering methanol is both environmentally friendly and economical friendly since recovered methanol can be reused in the biodiesel production process.

Technical

Methanol has a boiling point of approximate 150°F and glycerin boils at around 550°F. By heating the two liquids we can turn the methanol molecules into vapors, and then condense them back into liquid methanol. Much of the information collected was from websites related to building stills for moonshine, or grain alcohol. As stated above many small scale biodiesel producers either are not performing methanol recovery or are not compiling and documenting the work that is being done. From this it was hard to obtain large amounts of information from the biodiesel community on such recovery from a biodiesel viewpoint.

In Appendix A, a paper is included that compares the benefits and shortcomings of different still types. At the end of this paper several books and websites are referenced as they may be useful for the continuation of the project. Further websites are compiled in Appendix B, listing their subject and corresponding hyperlink.

Narrative

In the beginning of the year the biodiesel team members helped me in completing the overall build of the system. Several parts had to be fabricated which delayed system completion slightly. We ran into problems in heating our glycerin because of too small of a heating element. We replaced our 2000 watt heating element with a larger 4500 watt which did work after we sorted through some problems with the generator.

Another problem that we encountered was the removal or drainage of glycerin once methanol has been extracted from it. The glycerin becomes extremely thick and unmanageable to move once it cooled. We lost several days cleaning out the pump and system because we let the glycerin cool and it was too much for our pump to handle. An additional issue was the usage of the generator, which lead to some heavy lifting and commuting at inconvenient times.

However, we did recover three different batches of methanol with 88, 93 and 85 percent purity respectively. These purities were measured using a hydrometer, measuring specific gravities and comparing that to known specific gravities at a precise temperature. We also reused some of the waste glycerin to make soap. In doing so we established that the methanol recovery system built is a low cost solution to the environmental hazards that glycerin/methanol disposal poses. Furthermore, we had a proof of concept that biodiesel can be made from recycled methanol using a mixture of recovered and pure methanol to produce a batch of biodiesel.

I learned that projects are sometimes very evolutionary. Many of the objectives set out to be accomplished in the beginning of the year were challenges to actively seek solutions for. Also there were added objectives as the project moved along. I learned that projects are sometimes very flexible to the outcome as long as larger consideration takes place.

Project Plan

Phase Analysis

For the specific system I built we are in the testing phase as an overlapping research phase into the methanol and glycerin from the system occurs. Design, implementation and , manufacturability and prototyping have been completed. However, since this project has received a grant it is hard to say what phase the project will take. I know there has been discussion of another recovery system built which may lead more of the same phases being repeated for a different outcome. Even so, the research and testing that I have already done will prove valuable, as well as pursued to the fullest in the future.

Schedule

In Appendix C is the project planning article for the 08-09 school year. Much of the analysis of recovered methanol was unable to be performed due to the late acquisition of the gas chromatograph. Also the testing of the methanol recovery system did not happen after winter break until the weather became warmer. However, the purity of the recovered methanol was tested and waste glycerin was used to make soap which was not originally in the schedule. Also we ran into troubles of the pump and glycerin thickness resulting in the loss of several days otherwise used to recover methanol. Since the recover process takes 4-12 hours depending on the batch size it was sometimes hard in finding large blocks of time to dedicate to recovery. But some research was done on the biodiesel end using 90-93% pure methanol to produce biodiesel.

Resource Analysis

The biodiesel team is currently acquiring a gas chromatograph that can analyze chemicals and their make-up. This will prove an extremely valuable resource in the future for changing

input variables and determining the resulting output changes. Also a flash point tester is being acquired which will serve as a second method of testing methanol purity. The flash point tester will also serve as a method to determine if our glycerin is below certain toxicity level, making it safe for disposal. There is much research and analysis to be done on methanol and its effect of the biodiesel production process that this equipment can do.

Budget

In Appendix D is an Excel sheet that provides costs of the material used in the project. Also I did some cost analysis and discovered it will take approximately 130 gallons of glycerin to be processed before the system pays for itself. This analysis does not take into account the production cost of biodiesel used to run the system. The research we are doing in methanol recovery has led to a \$10,000 grant specifically for methanol recovery research.

Future Work

Since this project was first implemented this year there is lots of future work that need to be continued to make the project more sustainable and practical. The biodiesel team has decided to switch to a potassium hydroxide to mix with methanol which could lead to some experimentation of glycerin reuse. This year I did test its reuse as a soap product which did prove successful. Contacts within the college need to be established as to the possible college distribution or use or large scale soap. Another option is the glycerin is used for aid in composting through the community garden. The community garden has expressed an interest and such an option will need to be pursued further.

Cooling the condenser column was an issue that has potential creative solutions. Options like using wash water from the biodiesel cleaning process, or collected rain water could serve as resolutions. Another alternative would be using biodiesel as the coolant or another alcohol or oil based coolant to keep the condenser column cool. A less considered option would be a larger radiator to extract more heat from the cooling water. A similar problem is the draining of methanol free glycerin, with methanol extracted glycerin becomes extremely viscous which is rather unmanageable to pump and drain out our system. This issue will need to be addressed

Running 8 or 10 gallon batches of glycerin in 55 gallon processor is a waste of both energy and space. So over the summer a smaller scale methanol recovery system is going to be built. This would serve as a more flexible option since only small amounts of glycerin are being produced at the moment. Not only would it be more flexible it could lead to faster recovery times and serve as a better gauge for adjusting input variables and analyzing their results.

With the acquisition of testing equipment by the biodiesel team there is large amounts of analysis that needs to be performed on recovered methanol and biodiesel made from reused methanol. Gas chromatography can be performed to get a better idea of the contaminants in the recovered methanol. Furthermore a minimum purity level of methanol needs to be established in order for biodiesel production. A possible method for methanol refinement needs to be researched to see if recovered methanol can be made more pure. If this is not possible then the lifecycle or how many times recovered methanol can be used for biodiesel production should be tested.

Conclusion

In conclusion, the methanol recovery project has proven to be an exciting start up project that holds much promise in the future. The biodiesel team needed a low cost solution to refine waste glycerin and recover out high purity methanol. Even though the system is built there is a large amount of research that needs to be done to determine the effectiveness of such a system. The project is in no way near completion as this year was a good proof of concept to the potential methanol recovery can hold. The continuance of this project will lead the biodiesel team to be more sustainable, both environmentally and economically.

Still Type Selection for Methanol Recovery from Waste Glycerol

By: Jacob Munson

Whenever any vapor is being brought out of solution, the act of distillation is usually involved. There are several main types of stills that can be used depending on the use of the distillate. The largest factor in choosing a still is how much distillate is wanted to be recovered from the other liquid, also how pure that distillate is when the process is complete. This is why the still type selection is an important part of the methanol recovery project. Since we are going to be recovering methanol that will be used again we want a high purity recovery as well as a process that will only take one step, versus several refining steps.

Extracting this methanol from waste glycerol is an important step in making the biodiesel process more eco-friendly and safe. Glycerin that has waste methanol suspended in it is more volatile to handle and the methanol within this glycerin can be detrimental to the environment. This is why I am conducting careful research in the area of what type of still can offer the most pure distillate recovery. In the end this recycling will mean that not as much methanol will have to be used. Also the use of glycerin for composting will have less environmentally harmful effects.

The pot still is often the most well known and common type of still used for vapor recovery. It entails a pot over a heater and a pipe running out of the top of the pot that eventually coils through some type of cooling mechanism. When two or more liquids are heated, the one with the lowest boiling point will vaporize, travel up the pipe, condensate at the cooling coil and come out of this pipe as the refined semi-pure liquid it is. The trouble with this type of still though is that it does not offer a highly pure distillate. The recovered liquid can then be

processed again to make a finer distillate, but this of course takes more energy, time and does not provide an extremely pure distillate, unless the cycle is ran several times.

A better suited still for our application is a reflux still. This method of distillation puts packing or internal trays in the recovery column. Rising vapors of the distillate have to pass through this packing before they are condensed again. Effectively this process vaporizes the liquid seeking to be recovered, as it passes through the large amount of surface area in the packing of the packing column, the vapors are then condensed back into liquid form via a fitted cooling pipe on this packed column. As the condensed liquid trickles back down the column it is met by more pure vaporized particles of the same substances. This is like constantly redistilling the liquid until only the pure distillate is left to pass through and recovered. A highly detailed version of this method can be found at www.moonshine-still.com.

I plan on using the information and adapting the build plans listed on this website to construct a reflux still suited for the methanol recovery project. The pictures on the website detail a smaller version of a still that will be suitable for the Biodiesel teams application. Along with plans the website has a list materials, and instructions on how to build and implement a still. The website is going to be a useful resource when building of this project actually starts.

Since we are going to be reusing the recovered methanol, the purity of distilled methanol will be important. According to King in his book on Separation Processes 95 mol % liquid methanol can be refined into 97.9 mol % vaporized methanol during the recovery process. Since we will be using high quality methanol this can serve as a gauge as to the possibility of refinement quality. Further research can be done in this area in a book called Purification of

Laboratory Chemicals by Perrin and Armarego. It would be possible that a team in the future could research recovery purity specifications on methanol in waste glycerin.

The next phase of research will include acquiring materials that will be needed to build a reflux still. Finding a cheap but effective packing material will be a critical step to ensuring that the distillate is pure. The website mentioned above also listed several useful suggestions and resources for other suitable materials in building a methanol recovery still. Also diagrams in Biodiesel Basics and Beyond by Kemp will serve further as a starting point to our build. In this book several other safety factors are included that are not on the website. The book refers to the distillation process specifically for methanol recovery from waste glycerol. Research done so far is a step in the right direction to starting the methanol recovery project.

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Kemp, William. *Biodiesel Basics and Beyond*. City: Aztext Press, 2006.

King, C. *Separation Processes*. New York: McGraw-Hill, 1980.

Perrin, D. and W. Armarego. *Purification of Laboratory Chemicals*. New York: Pergamon Press, 1988.

Rousseau, Ronald. *Handbook of Separation Process Technology*. London: J. Wiley, 1987.

"Building a World Class Home Distillation Apparatus." 22 Aug. 2001. 1 Nov. 2007
<<http://moonshine-still.com/>>.

Methanol Recovery Project Websites

Biodiesel Background Information

<http://journeytoforever.org/biodiesel.html>

Methanol Recovery System

http://journeytoforever.org/biodiesel_make2.html#methreclaim

Separating Glycerin/FFAs

http://journeytoforever.org/biodiesel_glycsep.html

Glycerin/ Soap Making & Other uses

http://journeytoforever.org/biodiesel_glycerin.html

Methanol Recovery System Links

<http://www.santbani.k12.nh.us/biodiesel/Methanol%20Recovery%20System.ppt>

<http://www.b100.org/presentations/MethanolRecovery/index.htm>

Methanol Recovery Condenser How To

<http://www.liferesearchuniversal.com/condenser.html>

Biodiesel Library

<http://www.b100.org/presentations/MethanolRecovery/index.htm>

http://journeytoforever.org/biodiesel_processor5.html#methcondens

Still Building Outline

www.moonshine-stil.com

Methanol Purity Graph

<http://www.make-biodiesel.org/methanoltest/>

Soap Making Information

<http://www.eaudrey.com/glycerin.htm>

Methanol Recovery Project Planning Article

Jacob Munson

Goals/Deliverables

Methanol Recovery System Completion

Goal:

Complete build of the methanol recovery system by 11-07-08. With the assistance of Collaboratory and Biodiesel team members plumbing, heating element, and pump wiring shall be completed to make the system operational.

- | | |
|--|-----------|
| - Complete steel plumbing of methanol recovery system | 10-27-08 |
| - Assemble steel fittings and brass valves | 09-25-08 |
| - Take measurements of steel plumbing | 09-25-08 |
| - Model steel plumbing/methanol recovery system for tube fabrication | 10-06-08 |
| - Obtain fabricated tubing from Andrew Derr | 10-20-08 |
| - Assemble fabricated tubing | 10-23-08 |
| - Modify heating element to heat to desired 220°F | 11-3-08* |
| - Obtain wire schematic if necessary | 10-27-08* |
| - Obtain additional hardware if necessary | 10-30-08* |
| - Complete modification and test | 11-3-08* |
| - Wire pump to make operational | 11-07-08* |
| - Obtain wire schematic if necessary | 10-27-08* |
| - Obtain additional hardware if necessary | 10-30-08* |
| - Complete wiring and test | 11-07-08* |

* denotes expected Collaboratory team member(s) involvement due to colder weather and decrease in biodiesel production

Proof of Concept

Goal:

Prove that the methanol recovery system works as intended by checking the pump circulation and liquid temperature. If modifications are necessary they will be carried out to make the system operational; research may be necessary if larger problems arise.

- | | |
|---|-----------|
| - Test methanol recovery system at Professor Erikson's Farm | 11-14-08* |
| - Obtain permission from Professor Erikson | 11-10-08 |
| - Test system | 11-14-08* |
| - Ensure pump is circulating liquid properly | 11-14-08* |
| - Ensure liquid reaches appropriate temperature | 11-14-08* |
| - Make modifications if necessary | 11-14-08* |

* denotes expected Collaboratory member(s) involvement due to colder weather, i.e. decrease in biodiesel production

Testing and Refinement**

Goal:

Perform ASTM standardized tests to ensure that the recovered methanol is 90% or higher in purity. Research and refinement may be necessary to improve quality of tested methanol via gas chromatograph. Over the course of 4 months these tests will be performed to collect valid data and ensure the process is sustainable.

- Ensure testing equipment necessary is available 11-17-08
 - Consult Chemistry department if not available through Biodiesel 11-17-08*
 - Set up dates for spring semester testing 11-20-08*
 - Research outside party if testing equipment unavailable on campus 11-20-08*
 - Contact company and set up dates for spring semester testing 11-24-08*
- Run methanol recovery system 12-04-08 – 03-09-09
 - Test recovered methanol for purity 12-11-08 – 03-16-09
 - Analyze results 12-11-08 – 03-16-09
 - Research and change process if necessary 12-15-08 – 03-19-09

Documentation and Sustainability

Goal:

Write a 3-5 page detailed report that includes the instructions of how to operate the methanol recovery system and perform the necessary tests. Pictures will be included to provide visual aid to written instructions. ASTM literature will also be listed for quality assurance of testing.

- Inquire to Collaboratory/ Biodiesel student(s) interested in continuing work 03-23,26-09
 - Familiarize interested student(s) on how to run system 04-02,16-09??
 - Supervise student(s) complete independent running of system ??
 - Supervise student(s) complete independent testing of methanol ??
- Write an instruction manual for system operation and distillate testing 05-01-09
 - Write rough draft and meet with advisor for correction 04-30-09
 - Revise and complete final draft 05-01-09
 - Compile resources 04-23-09
 - Photographs, include comments 04-23-09
 - ASTM literature for testing purposes 04-23-09

*- denotes if necessary

** - denotes this is a step that will be repeated and refined, my hope is that one batch of recovered methanol can be tested before Christmas break. Testing and refinement of system will be continued through the spring semester.

?? - denotes flexible schedule/dates based on the students comfort level and quickness to understand procedures/testing

Major Resources

Gas Chromatograph

For testing and refinement phase of project the gas chromatograph will be used in testing the purity of refined methanol to ensure it can be reused in biodiesel production. It is the Biodiesel teams hope that such a gas chromatograph will be obtained with DOE Grant money before winter. If not the Chemistry department may have to be consulted or an outside company that conduct such tests so that the testing and refinement phase can move forward in the spring.

ASTM Testing Literature

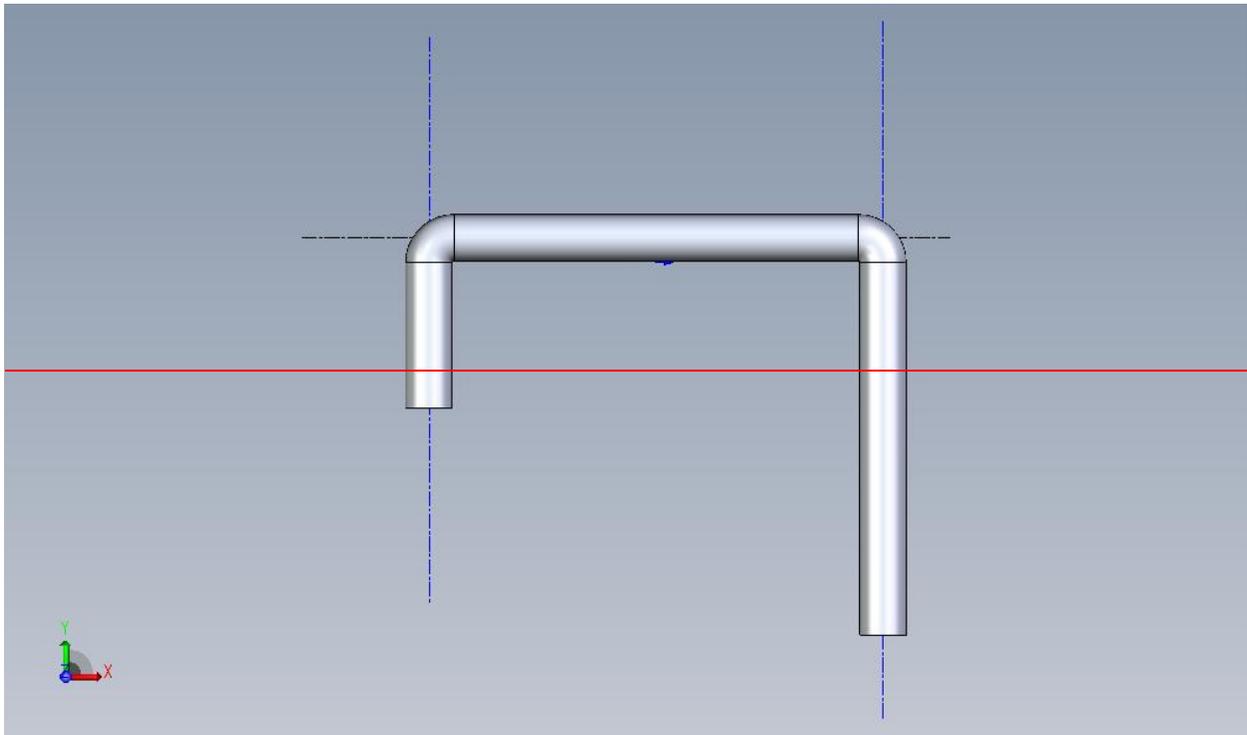
ASTM testing literature will be vital to the assurance in quality of recovered methanol distillate. Such testing literature will be obtained either at the library, or online sources when necessary.

Appendix D

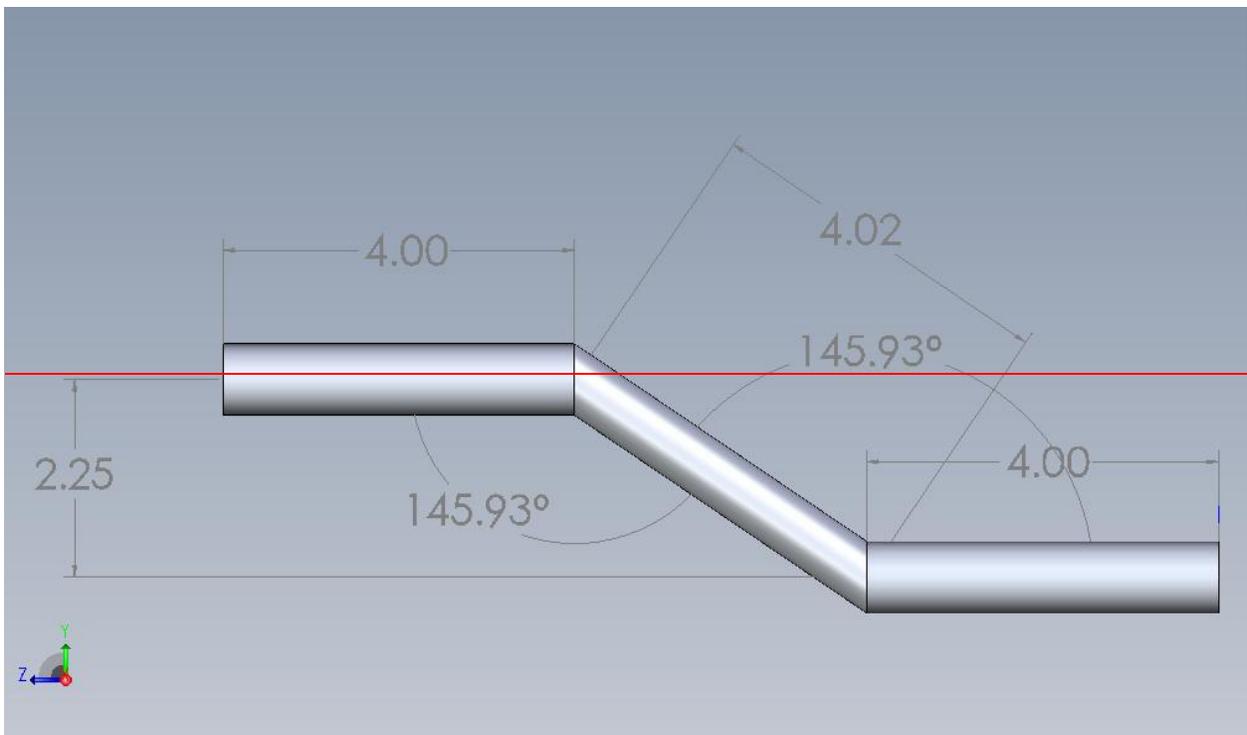
| Material | Size | Quantity | Total Price | Price | Supplier |
|------------------------------------|--------------------------------|----------|-------------|----------|---|
| Copper Fittings | | | | | http://www.mscdirect.com |
| Tee | 2" x 2" x 1 1/2" | 1 | \$ 16.03 | \$ 16.03 | http://www.mscdirect.com |
| Cap | 2" | 1 | \$ 5.71 | \$ 5.71 | http://www.mscdirect.com |
| Elbow (90) | 1 1/2" | 1 | \$ 6.07 | \$ 6.07 | http://www.mscdirect.com |
| Reducing Coupling | 1 1/2" x 1" | 1 | \$ 5.72 | \$ 5.72 | http://www.mscdirect.com |
| Cap | 1 1/2" | 2 | \$ 6.20 | \$ 3.10 | http://www.mscdirect.com |
| Tee | 1 1/2" x 1 1/2" x 1 1/2" | 2 | \$ 19.66 | \$ 9.83 | http://www.mscdirect.com |
| Reducing Coupling | 1" x 1/2" | 1 | \$ 2.91 | \$ 2.91 | http://www.mscdirect.com |
| Adapter | 2" Threaded to Slip | 1 | \$ 16.73 | \$ 16.73 | Lowes |
| | | Shipping | ? | | |
| | | Total | \$ 79.03 | | |
| Copper Pipe | | | | | |
| 2" | 3' | 1 | \$ 43.62 | \$ 43.62 | http://www.rcrdistributors.com |
| 1 1/2" | 3' | 1 | \$ 26.88 | \$ 26.88 | http://www.rcrdistributors.com |
| 1" | 2' | 1 | \$ 9.20 | \$ 9.20 | http://www.rcrdistributors.com |
| 1/2" | 2' | 1 | \$ 4.80 | \$ 4.80 | http://www.rcrdistributors.com |
| | | Shipping | \$ 12.00 | | |
| | | Total | \$ 96.50 | | |
| Pumps | | | | | |
| Glycerin/Methanol Recirculation | N/A | 1 | \$ 39.99 | \$ 39.99 | http://www.northerntool.com |
| Water Recirculation | N/A | 1 | Free | | Dave Hostetter |
| | | Shipping | \$ 11.65 | | |
| | | Total | \$ 51.64 | | |
| Steel Fittings | | | | | |
| Closed Nipple | 1" | 2 | \$ 2.58 | \$ 1.29 | Lowes |
| Closed Nipple | 3/4" | 2 | \$ 1.72 | \$ 0.86 | Lowes |
| Tee | 3/4" | 2 | \$ 2.94 | \$ 1.47 | Lowes |
| 90° Elbow | 3/4" | 1 | Free | Free | John Meyer |
| Coupling | 3/4" | 1 | \$ 1.09 | \$ 1.09 | Lowes |
| Coupling | 2" | 1 | \$ 3.73 | \$ 3.73 | Lowes |
| Reducing | 1" to 3/4" | 2 | \$ 4.34 | \$ 2.17 | Lowes |

Appendix D

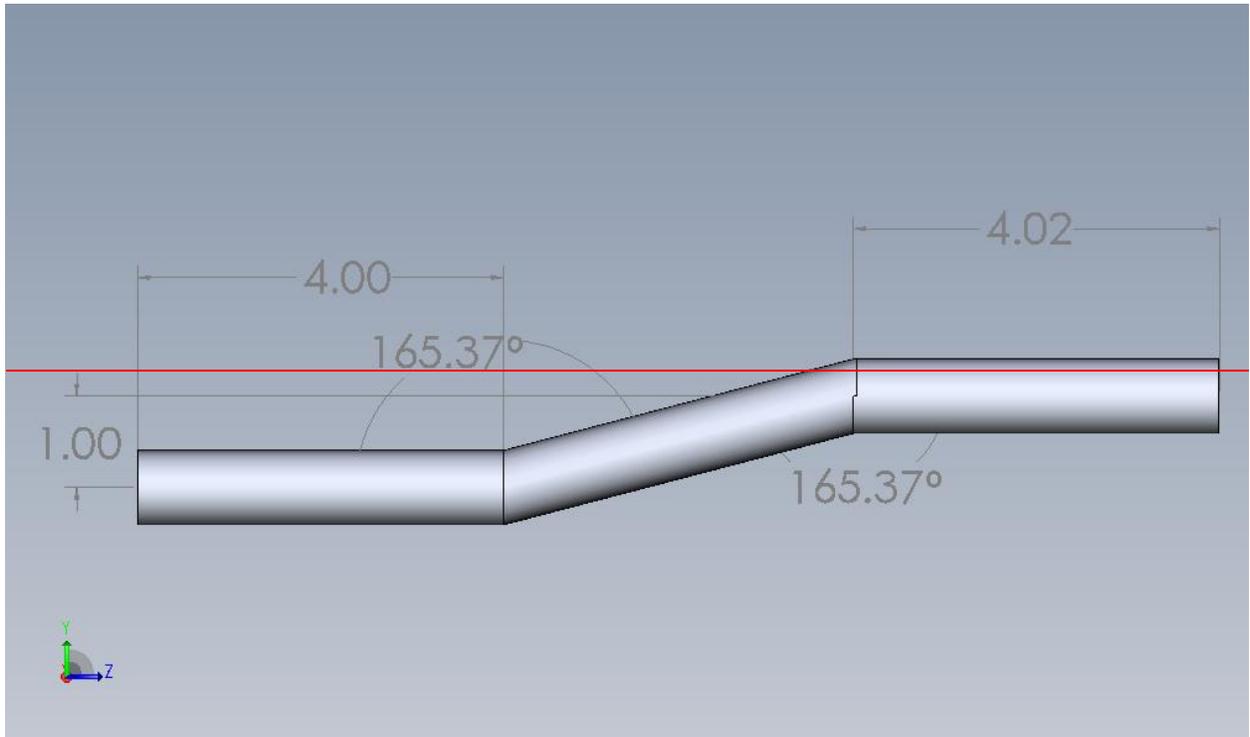
| | | | | | |
|--------------------------------|-----------------------------|-------|-----------|----------|----------------|
| Coupling | | | | | |
| Adapter | 3/4" Threaded to Pipe | 6 | Free | Free | Andy Derr |
| | | Tax | \$ 0.98 | | |
| | | Total | \$ 17.38 | | |
| | | | | | |
| Miscellaneous | | | | | |
| Stainless Steel Hose Clamps | 1/2" | 3 | Free | | Biodiesel Team |
| Steel Mesh Screen | 1" x 1" | 2 | Free | | Biodiesel Team |
| Drum | 55 gallon | 1 | Free | | John Meyer |
| Heater Core | 4500 W | 1 | Free | | Biodiesel Team |
| Bucket | 5 gallon | 2 | Free | | Robert Munson |
| 5 Gallon Bucket Lid | N/A | 1 | Free | | John Meyer |
| 5/8" ID Vinyl Tubing | 10' | 1 | \$ 10.76 | | Lowes |
| Ball Valve | 3/4" | 3 | \$ 58.89 | \$ 19.63 | Lowes |
| Tubing | Fitted | 2 | Free | | Andy Derr |
| Generator Plug | N/A | 1 | \$ 23.25 | \$ 23.25 | Lowes |
| 10-3 Wiring | 10' | 1 | \$ 17.30 | \$ 1.73 | Lowes |
| Fiberglass Insulation | Roll | 2 | \$ 25.16 | \$ 12.58 | Lowes |
| Raschig Rings | Bag (1/2L) | 1 | \$ 16.02 | | |
| | | Tax | \$ 8.12 | | |
| | | Total | \$ 159.50 | | |
| | | | | | |
| Project Total | | | \$ 404.06 | | |



Top Fabricated Tubing



Middle Fabricated Tubing



Bottom Fabricated Tubing

Messiah College Biodiesel Fuel Generation Project

DE-FG36-08GO88068

Appendix D. – Manuals and Reports

Appendix D.2.

2009 Methanol Recovery Seed Assistance Grant Report
(Includes 2010 Methanol Recovery Manual)

Methanol Recovery System Prototype Development and Testing Final Report

| | |
|--|---|
| Recipient Name: <u>Michael Zummo</u> | Faculty/ <u>Staff</u> /Student (circle one) |
| Title: <u>Biodiesel Project Manager/Principle Investigator</u> | |
| Higher Education Institution: <u>Messiah College</u> | |
| Address: <u>One College Ave., Grantham, PA 17055</u> | |
| Phone: <u>717-766-2511 x 3170</u> Fax: <u>717-796-5222</u> | |
| E-mail: <u>mzummo@messiah.edu</u> | |
| Project Title: <u>Methanol Recovery System Prototype Development and Testing</u> | |
| Amount Received: <u>\$10,000</u> | |

Please fill out the following chart where applicable. This information is required in order for ITN to comply with the State's (DCED) grant funding process.

| Measure | | |
|-------------------------------|----------------------------------|----------------|
| Patents | | |
| • Provisional Patent Filed | Owner: | Date Filed: |
| • Provisional Patent Awarded | Owner: | Date Received: |
| • Software Copyrights Filed | Owner: | Date Filed: |
| • Software Copyrights Awarded | Owner: | Date Received: |
| Trademarks | | |
| • Trademarks Filed | Owner: | Date Filed: |
| • Trademarks Awarded | Owner: | Date Received: |
| Technology Development | | |
| • Prototype Developed | Anticipated Release: 2011 | |
| • Product Developed | Anticipated Release: | |
| • Product Commercialized | To Whom: | Date Released: |
| Funds Leveraged | | |
| • Venture Capital | Amount: | |
| • Private Equity | Amount: | |
| • Grants | Amount: | Type: |
| • Other Sources | Amount: | Source: |
| New Start-Up or Spin-Out | Name: | |

A. Financial Report

The objectives of this project were to use the funds awarded through the Seed Assistance Grant to perform additional research, testing and development on the existing methanol recovery system prototype with specific goals of verifying the quality of recovered methanol and glycerin. The findings of this research and testing were then to be used to modify and improve the prototype to increase performance and safety as well as reduce energy requirements. To achieve these objectives a full time student intern, Stephen Bray, was hired to work with the project manager to conduct the research, testing and development of the prototype over the course of the summer. The student intern was contracted to perform 400 hours of work being paid \$10 per hour. The original budget for the student intern also provided funds to cover on-campus summer housing for the student. However the student chose to find other housing off campus and was therefore paid an additional \$4 per hour for a total of \$14 per hour before taxes. The wages for the student intern, including all necessary taxes, totaled to \$6096.22, making up the majority of the allotted funds for this project. Pay stubs for the student intern documenting hours and pay can be found attached in *Appendix A: Item Nos. 1A-F: Student Intern Pay Stubs*.

The remaining portion of the funds were used to cover equipment and supply costs associated with the research, testing, and development of the prototype system. The initial objective of the project was to determine the purity and quality of methanol and glycerin being recovered by the existing prototype. The main focus with regard to the purity of recovered glycerin was to determine the remaining methanol content and verify that enough methanol had been removed to create a non-hazardous substance. Flash Point testing using a Pensky-Martens Flash Point Tester (Appendix B., Photo No. 1) and following ASTM Test Method D 93 was used to determine the remaining methanol content of recovered glycerin. The Pensky-Martens Flash Point Tester had been previously purchased by the Biodiesel Research Project for biodiesel testing and no additional equipment was require to perform glycerin flash point testing. To establish the appropriate methods for determining the purity of methanol four different ASTM Standards were purchased from ASTM International, totaling \$149.00. Receipts for these standards can be found attached in *Appendix A: Item No. 2A and 2B: ASTM Receipts*. Based on these standards, the most cost effective and practical method for testing methanol purity was to determine specific gravity by means of a hydrometer. A hydrometer set (Appendix B., Photo No. 2) was then purchased from Fisher Scientific to be used for testing methanol purity. The hydrometer set cost \$280.01 and the invoice for this purchase can be found attached in *Appendix A: Item No. 3: Fisher Scientific Invoice*.

Testing then began using the original prototype system to recover methanol and purify glycerin. It was quickly decided that a new prototype capable of handling smaller batches of glycerol should be develop. The original prototype (Appendix B., Photo No. 3) required a minimum of 40 gallons of glycerol to operate properly. To produce this much

glycerol required several batches of biodiesel to be processed and would only allow the methanol recovery system to be run every two to three weeks. A sample this large also required approximately 8 hours to perform a full recovery cycle. The new prototype (Appendix B. Photo No. 4) is a smaller unit that can operate with as little as 10 gallons of glycerol, which is approximately the result of one batch of processed biodiesel. This allows the system to be run more often for testing and research and the total time for full recovery has been reduced to approximately 4 hours. The costs associated with the development of the new prototype totaled \$172.05 and receipts for these purchases can be found attached in *Appendix A: Item Nos. 4-6: Prototype Development Invoices and Receipts*.

As the new prototype was develop, it was determined that the addition of electronic controllers for the heater and chiller would increase overall performance of the system as well as improve its safety. Controllers were installed to both monitor and manage the temperature of the glycerol in the system and the cooling water flowing through the column head. These controllers allow the operator to set the minimum and maximum temperatures and the system will then maintain these set points throughout the recovery cycle. This increases performance by allowing the system to be more stable and consistent during recovery and increases safety by ensuring that the system cannot over heat and that cooling water cannot freeze. The initial controller was purchased from Automation Direct but was not the correct model and was returned. Automation Direct credited the College for the cost of the controller totaling \$79.50, see *Appendix A: Item Nos. 7A and 7B: Prototype Development Invoices and Receipts/Automation Direct* for records of this transaction. The correct controller was then selected and also purchased from Automation Direct. The total for these activities was \$234.67. The invoices and receipts associated with these purchases can be found attached in *Appendix A: Item Nos. 7-9: Prototype Development Invoices and Receipts*.

The final phase of this project was to develop a data acquisition system to be used to monitor temperatures of several different components of the methanol recovery system prototype. By monitoring these temperatures and then determining the quality of the recovered methanol and glycerin, the optimal system settings can be determined to produce the highest qualities of both methanol and glycerin. The data acquisition system selected to perform this research was an 8-channel thermocouple data logger system produced by Measurement Computing. The specific system selected is a wireless module that includes all necessary software to monitor and record temperature. This data logger system was purchased from MicroDAQ.com. The initial purchase made was a data logger only capable of reading a single thermocouple. This system did not meet our needs and was returned and exchanged for the 8-channel model. MicroDAQ.com credits the college for the cost of the data logger totaling \$246.00, see *Appendix A: Item Nos. 10A and 10B: Monitoring System Invoices and Receipts/MicroDAQ*. The overall total for these activities was \$678.93. A laptop dedicated to the data acquisition system was also

purchased from LAM Systems, Inc. totaling \$892.00. The invoice for these purchases can be found attached in *Appendix A: Item Nos. 10-13: Monitoring System Invoices and Receipts*.

| Expenditure | Amount |
|--|-------------------|
| <i>Summer Intern</i> (Wages and Taxes) | \$6,096.22 |
| <i>Testing</i> (ASTM Standards and Hydrometer Set) | \$429.01 |
| <i>Prototype Development</i> (Plumbing and Controllers) | \$406.72 |
| <i>Monitoring System</i> (Data Acquisition and Laptop) | \$1,570.93 |
| Total: | \$8,502.88 |

Table 1. Summary of Expenditure of Funds
(See Appendix A. for copies of pay stubs, invoices and receipts.)

B. Technical Progress Report:

Project Activities and Outcomes

The three main objectives for the Methanol Recovery Prototype Development and Testing Project as funded by the Seed Assistance Grant were to:

1. Determine the average quality of glycerin and the purity of recovered methanol obtainable using the existing prototype methanol recovery system.
2. Modify the existing prototype to improve the purity of methanol and the quality of glycerin recovered and reduce the energy required for the process.
3. Develop an operation and safety manual for the existing methanol recovery process and processor prototype.

The initial work of this project was to determine the methods by which the quality of glycerin and the purity of methanol would be tested. It was quickly decided that the quality of the glycerin would be assessed based on the remaining methanol content after the methanol recovery process was completed. This assessment was performed using a Pensky-Martens Closed Cup Flash Point Tester (Appendix B., Photo No. 1) and following the *ASTM D 93 Standard Test Methods for Flash Point by Pensky-Martens Closed Cup Tester*. This equipment and method were previously purchased for biodiesel testing. The flash point of glycerin is directly correlated to the methanol content remaining in the glycerin; the higher the methanol content, the lower the flash point. Establishing the flash point of the glycerin allowed its hazard rating to be categorized. A flash point of 140°F allows the glycerin to be considered minimally hazardous and a flash point temperature above 200°F renders the glycerin no longer hazardous. The average flash point of the glycerin processed in the original prototype was 100°F.

Selecting the method to test the purity of recovered methanol required research into several ASTM standard test methods. The first standard researched was *ASTM E 346-08 Standard Test Methods for Analysis of Methanol*. This standard provided a general guide to the test methods required to determine certain properties associated with methanol. Of the methods presented in *ASTM E-346-08*, further research was done into *ASTM D 891 Standard Test Methods for Specific Gravity, Apparent, of Liquid Industrial Chemicals*, *ASTM D 1613-06 Standard Test Method for Acidity in Volatile Solvents and Chemical Intermediates Used in Paint, Varnish, Lacquer, and Related Products*, and *ASTM E 203-08 Standard Test Method for Water Using Volumetric Karl Fischer Titration*. Based on cost, feasibility and practicality, *ASTM D 891 Standard Test Methods for Specific Gravity, Apparent, of Liquid Industrial Chemicals: Test Method A- Specific Gravity, Apparent, By Means of a Hydrometer* was selected to assess methanol purity for the purposes of this project. The specific gravity of recovered methanol was determined using a hydrometer then using a chart that plots measured specific gravity and temperature adjustments, to determine the percent methanol content. The specific gravity of methanol is typically between 0.871 at the low end of purity ($\approx 67\%$) and 0.775 at the high end of purity ($\approx 99\%$) based on temperature. The average purity of methanol recovered from the original prototype was 85%. At this point Objective No. 1 was completed.

Based on the data collected from the glycerin and methanol testing described above, design changes were made to the original prototype to produce higher purity methanol and better quality glycerin. The first modification to the system was to reduce the overall batch size from 40 gallons to 10 gallons. The current biodiesel processor produces approximately 10 gallon of glycerol from each batch of biodiesel. The smaller system can now be run after a each batch of biodiesel is made, rather than waiting to accumulate glycerol for the larger design and allowing less methanol vapor to evaporate from the glycerin between runs. The smaller batch system (Appendix B., Photo No. 4) allows the size of the heating element to be reduced from 3000 Watts to 1500 Watts which reduces the overall energy required to heat the glycerol. The heating element in the new prototype is submerged directly in the main glycerol storage tank rather than mounted in line with the plumbing. This allows the main storage tank to be heated to reduce the viscosity of the glycerol for easier pumping. The size reduction also allows for shorter overall run times and more precise control of the system. The reflux column head from the original prototype was also used on the new system. The new prototype is fully insulated to minimize heat loss over the course of the recovery cycle which aids in reducing the total energy required by the system.

Initial testing of the new prototype produced recovered methanol with an average purity of 95% with several batches approaching 98% purity. The average flash point of the remaining glycerin was 170°F, with an overall high of 230°F.

The next phase in the development of the new prototype involved the addition of a chiller system to maintain the temperature of the cooling water. The original prototype required that the user change the cooling water after it had been heated above a specific temperature. Water was being changed several times each hour requiring substantial involvement of the user and large amounts of water. The original prototype used approximately 40 to 50 gallons of water for each recovery cycle. The addition of the

chiller system allowed for the system to use approximately 15 gallons of water for dozens of recovery cycles. This chiller system is a modified air conditioning system that flows the warmed cooling water over a chilled radiator to reduce the overall temperature. This system reduces user input because time no longer needs to be spent changing cooling water. The chiller system does require more energy than the previous method but it saves hundreds of gallons of water. Several recovery batches were run using the new chiller system and the resulting methanol purity was slightly improved because of the more uniform temperature of the cooling water flowing through the column head to re-condense the methanol vapor.

To further reduce the amount of human input and increase overall consistency and safety, electronic controls were added to the system. The original prototype required manual control of the heaters by the user and if left unattended the system had the potential to overheat. It also required manual monitoring of the cooling water temperature to determine when the water should be changed or more recently when the chiller system should be started. The addition of two electronic controller units allows the user to set the maximum temperature for the heaters on one controller as well as the maximum temperature of the cooling water on another controller. Using these controls, both the heater and the chiller unit will turn on and off as necessary when set point temperatures are reached. Final programming of these controllers is currently being completed and testing and modification to the newly controlled system will begin shortly.

The final phase in the development of the new prototype will be the installation of a temperature monitoring system. The purpose of the monitoring system will be to measure the temperatures at key locations on the methanol recovery system such as several points along the column head, in the head space of the heating and storage tank, and at different levels in the glycerol storage and heating area. The monitoring system consists of a wireless thermocouple data acquisition system linked to a laptop with data recording software. This will allow constant temperature readings and analysis from several, different, critical points on the methanol recovery system. With this data it can then be determined which parameters produce the highest purity methanol and highest quality glycerin. The methanol recovery system can then be modified, the controllers can be re-programmed and a precise procedure can be developed to produce the optimum results. This concludes the activities associated with Objective No. 2.

The final objective of this project was to develop an Operation and Safety Manual for the Methanol Recovery System prototype. A manual was developed for the original prototype and is currently being updated to reflect the changes made to the new prototype system. A draft of this manual can be found in Appendix C.

Project/Research Next Steps

The next step for the Methanol Recovery System Prototype Testing and Development project is to install and employ the monitoring system to collect temperature data from the prototype and determine correlations between that data and the output of the system. While collecting the temperature data, the purity of the methanol and quality of glycerin recovered from the system will be tested simultaneously. From these data sets correlations will be drawn between the critical system temperatures and purity of the

methanol and quality of the glycerin recovered. This will allow for the identification of critical areas in the methanol recovery system that will require modifications and specific improvements. These modifications and improvements will not only improve the overall performance of the system but will also improve safety and reduce energy requirements. Once these critical temperatures and areas are established, further work can be done with the methanol recovery system to reduce the amount of necessary human input.

The final desired outcome of reducing overall human input is a fully automated methanol recovery system that is integrated into the biodiesel production process and is capable of detecting potential issues and adjusting the process accordingly. The initial plan is to accomplish this goal through the use of a Programmable Logic Controller (PLC). A PLC would allow automated control of the system through the use of different logic commands. These logic commands will be directly related to the output of multiple sensors placed on the methanol recovery and biodiesel systems. Some possible controls for the PLC include run time, maximum temperature of glycerin, optimal temperature for cooling water, and monitoring head pressure to prevent possible failures. The PLC will also be capable of controlling the transfer of a batch of glycerin from the biodiesel processor to the methanol recovery system and beginning the recovery cycle without any human input. This will make the process of methanol recovery a seamless part of the biodiesel production process.

Both the monitoring system and the PLC incur substantial costs for a small scale methanol recovery system however, the data and control that will be produced from these systems provide invaluable benefits. For the purposes of this project, the monitoring system was purchased through the funding from the SEED Assistance Grant. In the near future the monitoring system will be installed and research will begin to determine the most critical temperatures and components of the methanol recovery system. The future plan to incorporate this prototype into a biodiesel processing system through the use of a PLC will be funded by external sources. Contact has been made with an outside partner that has offered to fund the purchase and installation of a PLC and all necessary sensors for this project. As research is completed on the current prototype and developments are made, work will continue to create a synchronized biodiesel and methanol recovery system.

Stage of Development, Challenges, Issues and Concerns Related to Commercialization

In accordance with the table above, this project is still in the Technology Development stage. Currently two prototype Methanol Recovery Systems have been developed and tested but additional research, testing and development must be done before a prototype can be released for further expansion into a marketable system. It is anticipated that a complete prototype can be design and tested by 2011 and then be released for development into a marketable system.

Presently no major challenges or concerns have been faced with this project. However, as the project continues to move forward, there are questions about sources of future funding for additional research and development of the prototype. Ideally the project will require an industry partner or funding source to continue to develop and move toward

commercialization, however no such partner or source has been found. There are also questions regarding patents associated with this project and the requirements necessary to move this project toward commercialization. Once a completed prototype has been developed it will be necessary to pursue a patent on the system to potentially pursue a marketable product however this is not something the project manager or institution has experience with. There is a great need for a small scale methanol recovery system that can safely and effectively be used in the small scale biodiesel market. The goal of this project is to continue working to develop a complete system that can be marketed to the small scale biodiesel industry.

Appendix B:

Photos



Photo No. 1: Pinsky-Martens Flash Point Tester



Photo No. 2: Hydrometer Set



Photo No. 3: Original 40 Gallon Methanol Recovery System Prototype

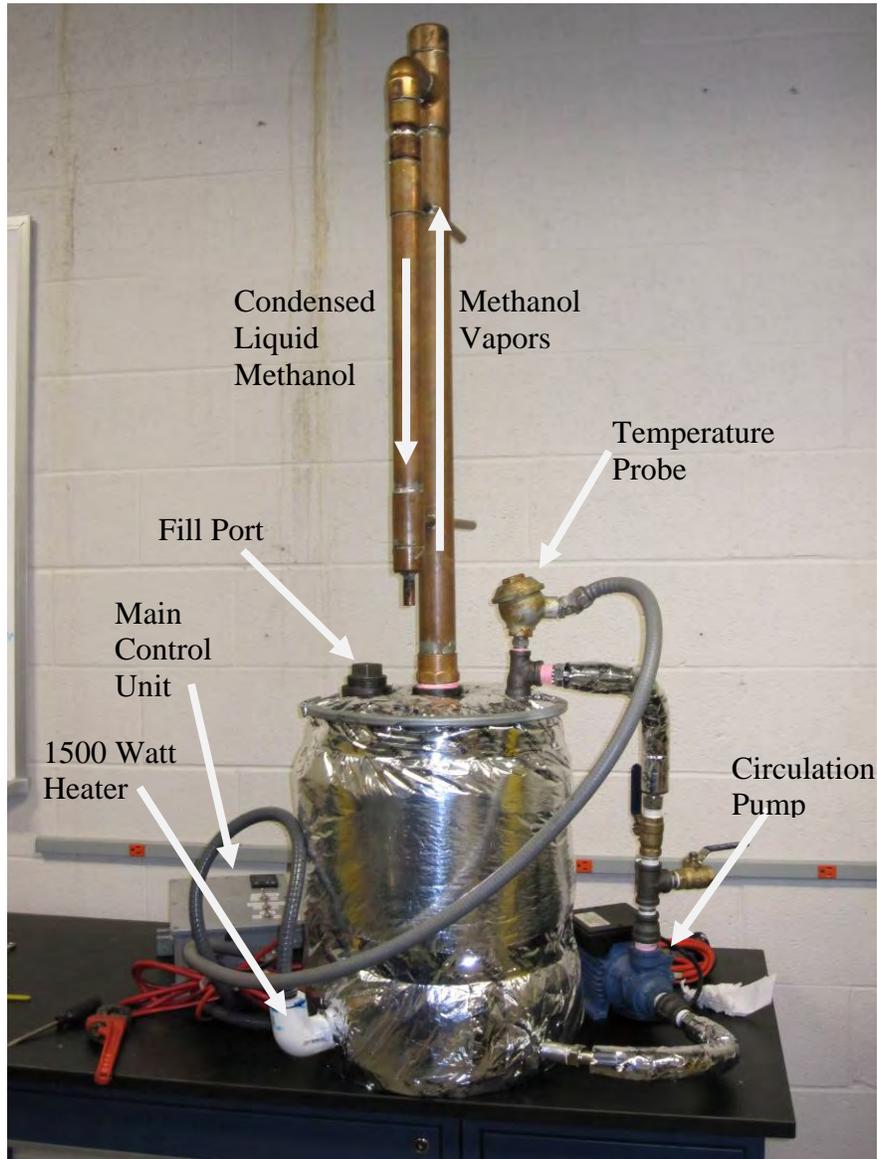
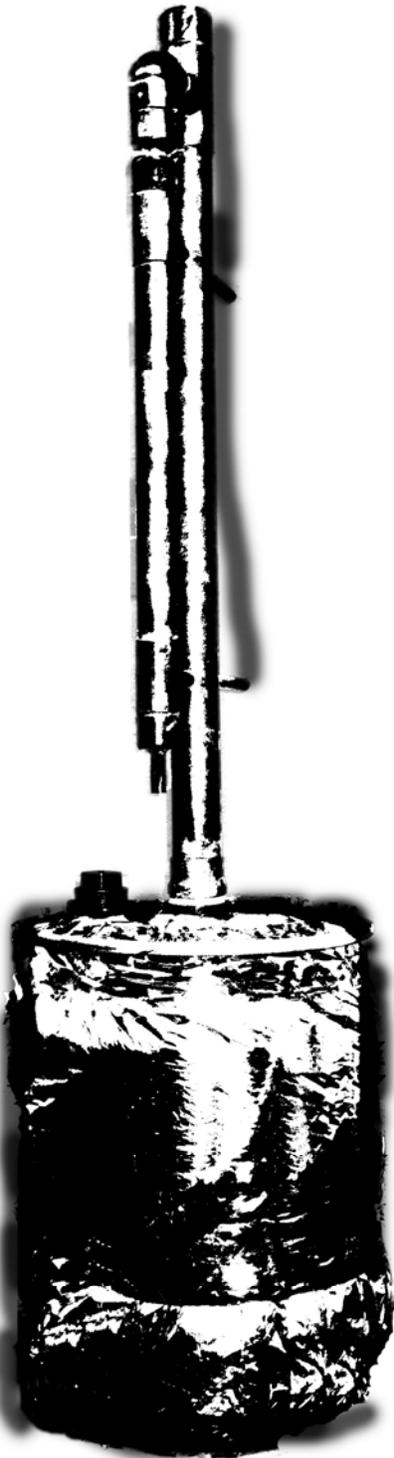


Photo No. 4: New 10 Gallon Methanol Recovery System Prototype

Appendix C:
Methanol Recovery System
Operation and Safety Manual

Methanol Recovery Operation and Safety Guide



Abstract

For the last six years student teams have been working in conjunction with the Department of Engineering and the Collaboratory for Strategic Partnerships and Applied Research to develop a process and processor systems for the production of biodiesel fuel. The majority of this work has focused specifically on the conversion of waste vegetable oil into high quality biodiesel fuel. With great success, research and development teams have used the waste vegetable oil from campus dining facilities to produce fuel for diesel powered campus vehicles and as a heating oil substitute. A byproduct of the biodiesel production process is a glycerin substance with a high methanol content known as glycerol. The glycerol byproduct of the biodiesel production process contains hazardous levels of methanol which makes it very difficult to find any use for it. Implementing a methanol recovery system allows the methanol to be removed from the glycerol, leaving a safe glycerin byproduct that has many uses and provides recycled methanol for future production of biodiesel. Students, faculty and staff at Messiah College are diligently committed to promoting renewable energy technologies and establishing sustainable sources of fuel to reduce dependence on petroleum based sources. Over the course of the last three years, student teams have developed a functional prototype methanol recovery system.

In the chemical process of creating biodiesel, a waste product known as glycerol is generated. Glycerol is a combination of methanol, the alcohol used to convert vegetable oil to biodiesel, and glycerin, a compound that occurs from the conversion of the free fatty acids in the oil. Glycerol is a waxy substance that consists of glycerin with methanol suspended throughout it. Because the methanol is suspended in the glycerin, this normally harmless and potentially useful compound must be treated as a hazardous waste. The issues of appropriately and safely handling, storing and/or disposing of this unrefined byproduct are currently the largest problem in commercial biodiesel production. Without a methanol recovery process, producers must pay to have their glycerol removed. In the case of small scale producers, the glycerol product is often improperly handled and disposed of, introducing large amounts of hazardous methanol into the environment. This proves to be an inhibition to sustainability in the biodiesel industry. The idea of methanol recovery is fairly simple but the implementation has not been studied enough to be understood completely.

Through the work of several engineering students, our team has developed a system by which we can remove the methanol from the waste glycerin. Our process boils the methanol out of the glycerin and re-condenses it into liquid form using a reflux still. This leaves a glycerin substance that can be used for fertilizer, hand soap, and potentially livestock feed and methanol that can be re-used in the biodiesel production process.

Safety Equipment and Considerations

The process of recovering methanol from glycerol involves heating the glycerol to the boiling point of methanol, capturing the methanol vapor in the reflux column and re-condensing the vapor into a liquid. Methanol and methanol vapor are extremely dangerous and create inhalation, eye and skin irritation, fire and explosion hazards. For specific safety consideration regarding methanol please see the Methanol Material Safety Data Sheet in Appendix A prior to using methanol in any way.

The individual(s) working with the methanol recovery system should be equipped with all of the following safety measures:

*Chemical Resistant Goggle- **Required***-Goggles should cover the entire region around the eyes so that no liquid or vapor and make contact. Safety glasses are not acceptable eye protection when working with the methanol recovery system.

*Protective Gloves-**Required***- Latex or nitrile gloves are acceptable. Ensure that there are no holes in the gloves allowing chemicals to interact with the skin. If gloves are ripped replace immediately with new ones.

*Long sleeve shirt, long pants and closed toed shoes- **Required***- Clothing should be worn that protects the skin from being exposed to hazardous chemicals. Shorts, t-shirts, sandals, flip flops or any other form of clothing that leave skin exposed to possible contact is unacceptable.

*Respirator- **Optional***- If methanol recovery is being performed in a confined space or any area that does not have adequate ventilation a respirator is recommended. However methanol recovery should always be performed in an area that is well ventilated and has close access to outdoor fresh air.

The space that houses the methanol recovery system should be equipped with all of the following safety measures:

*Eye Wash Station- **Required***- An emergency eye wash station should within close proximity of the methanol recovery system. If any hazardous material makes contact with the eye flush immediately at the eye wash station for 15 minutes and then seek medical attention.

*Sink or Water Source- **Required***- A sink or water should be in within close proximity to the methanol recovery system. If any hazardous material makes contact with the skin, rinse the area immediately with soap and water and remove any clothing or safety apparatus that may also have come in contact with the material. If the area exposed becomes irritated seek medical attention.

*Fire Extinguisher(s)- **Required***- One or more fire extinguisher should properly mounted in easily accessible locations near the space that houses the methanol recovery system.

All personnel operating the system should be trained to operate these fire extinguishers. Fire extinguisher should be inspected and if necessary replaced on a regular basis.

Ventilation- Required- The area that houses the methanol recovery system should be well ventilated and have a steady source of fresh air. It is recommended that methanol recovery be performed in a space that opens directly to fresh air such as a garage with the door open. Methanol vapors are heavier than air and will settle to the lowest point of the space being used. A fan should be used to maintain air flow in this region.

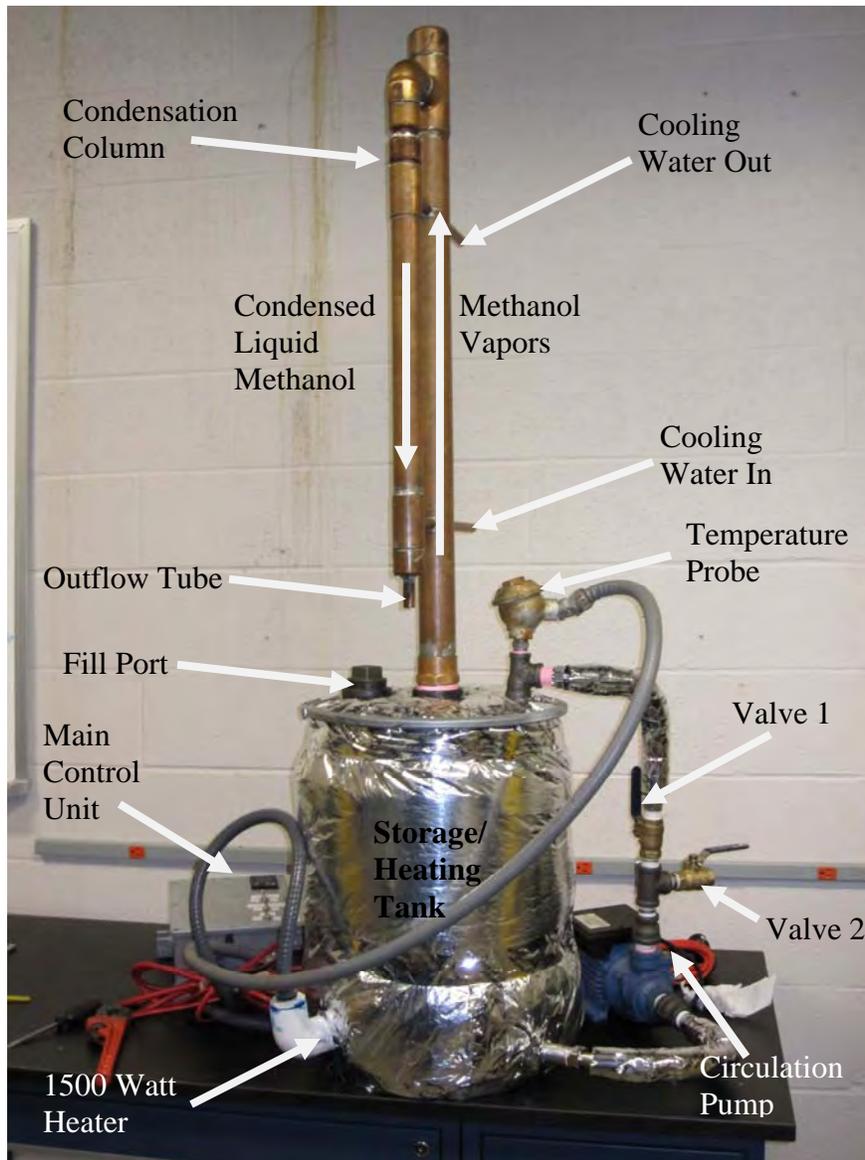
Other issues to be considered when working with the methanol recovery system or methanol in any form are:

Insure that there are no open flames, sparks, arcs or other types of ignition sources in the space that will house the system. Methanol and methanol vapor are extremely flammable!

All storage containers for glycerol, methanol and glycerin should be of the correct type and properly labeled with Hazardous Material labels. Glycerol and methanol should be stored in an approved flammables cabinet at all times when not being used.

If leaks are observed in the methanol recovery system immediately turn the system off and allow it to cool. Do not attempt to run the system until the leaking substance has been identified and the leak has been properly repaired.

System Overview



Recovering Methanol

The process of recovering methanol is timely but ultimately rewarding. Begin the process by acquiring and using all necessary safety equipment as listed above.

Step 1: Checking the valve positions.

The first step in beginning methanol recovery is the addition of the glycerin to the methanol recovery unit. Before this can happen, the user should check to ensure the proper valve position. In this system a valve is closed by the valve handle is perpendicular to the valve body and is open when valve handle is parallel with the valve body. Valve 1 is the flow control valve to the pump. Valve 2 is the outlet valve (See Figure 1 for valve locations). In order to fill the unit, Valve 1 should be open and Valve 2 should be closed. This will ensure that when the glycerol is added it will not run of the system through Valve 2.

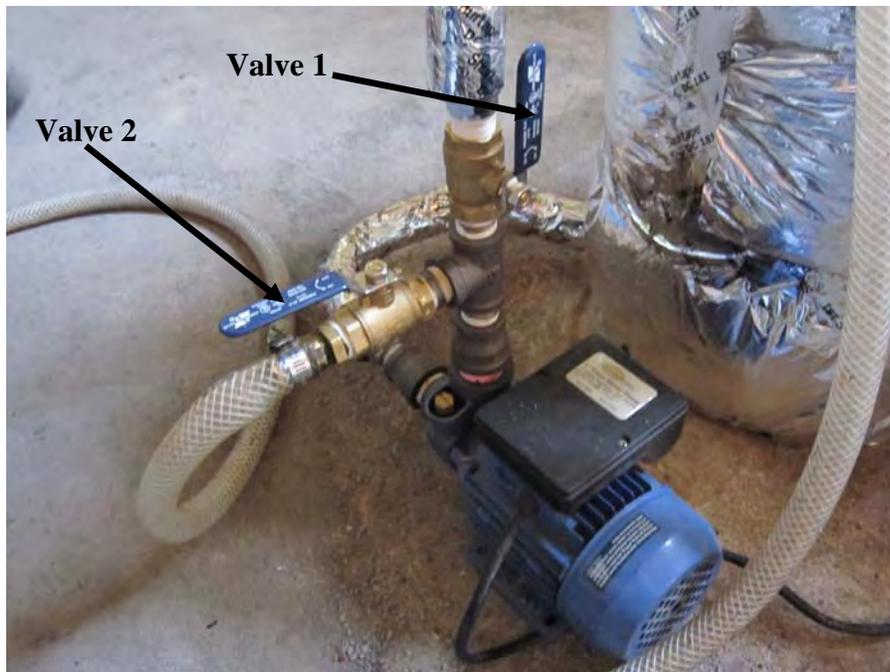


Figure 1. In this image both Valve 1 and Valve 2 are open.

Step 2: Adding the Glycerol

After the user has verified that the valves are in the proper positions, they can begin to add glycerol to the system. This can be done by removing the plug from the filling port on the lid of the system, inserting the funnel then pouring the glycerol from its storage container (See Figure 2.).



Figure 2. Location of filling port.

Once the glycerol is completely transferred into the system and the filling port plug has been replaced, the user can begin the recovery process.

Step 3: Powering the System

The actual recovery process begins by providing power to the unit. This can be misleading because there are two similarly colored cords coming from the main control unit. The Main Power Cord (which provides power for the main heater, circulating pump, and thermocouple) is the orange and black cord coming out of the right side of the main control unit (See Figure 3.). This cord can be plugged in to a regular 120 V, 20 Amp power outlet. The Secondary Power Cord is used to power an extra 1500 W heater that can be used to heat the glycerol. If this heater is required the Secondary Power Cord must be plugged into a 120 V, 20 Amp power outlet that is independent of the Main Power Cord.

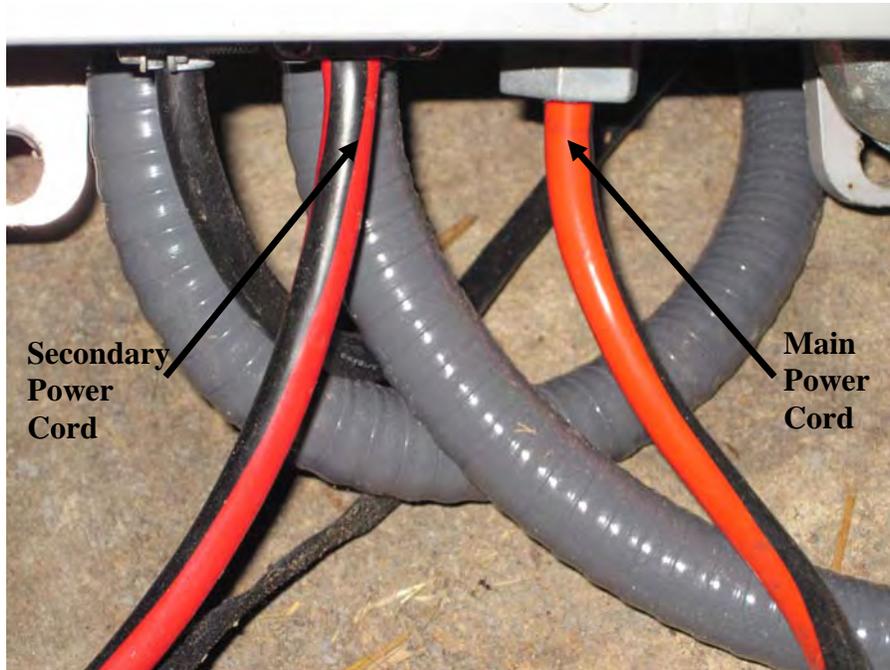


Figure 3. Power Cords from Main Control Unit

Step 4: Starting the Main Heater and the Pump

After plugging in the Main Power Cord, the user can turn on the Circulation Pump (labeled Pump) and Main Heater at the Main Control Unit (See Figure No. 4). Depending on the storage location of the glycerol, it may be cold and viscous making it difficult to pump. If this is the case the glycerol will need to be heated for a few minutes to become less viscous so that it can be pumped. To do this turn off the Circulation Pump but leave the Main Heater on for 5 minutes and then turn the Circulation Pump on again. Continue to do this until the pump begins to move the glycerol. Once the pump is running and glycerol is flowing through allow it to continue running and monitor the temperature of the glycerol. The temperature can be read from the Thermocouple Temperature Controller in degrees Celsius. If the area that houses the methanol recovery system is capable of providing a second independent 120 V, 20 Amp, then the Secondary Power Cord can be plugged in (See Figure 3. above) and the Secondary Heater can be initiated to heat the glycerol to the required temperature more rapidly.

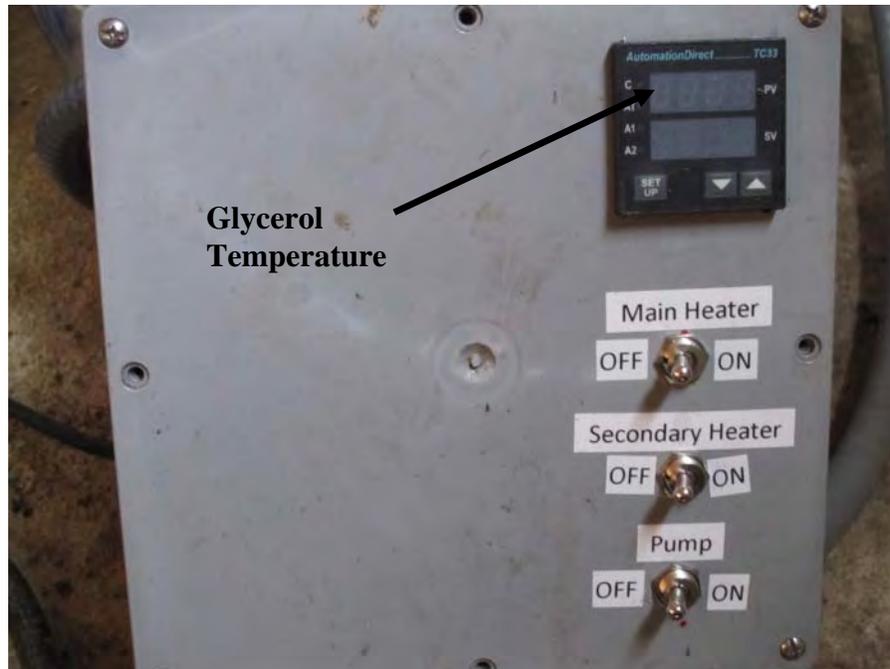


Figure No. 4: Main Control Unit

The glycerol is then circulated and heated until it reaches the point at which the methanol will begin to boil out of the glycerol. This begins to occur at 60°C and will continue upwards to 160°C. There is nothing to be done until the methanol condensation occurs on the Outflow Tube of the Distillation Column (See System Overview above). A hose should be attached to the Outflow Tube to direct the recovered methanol into a proper storage container. As the temperature of the glycerol approaches the boiling point of methanol, the flow of recovered methanol from the Outflow Tube will increase.

Step No. 5: Starting the Cooling Water

Once there is a steady flow of condensed methanol, the circulation pump for the cooling water can be turned on to begin flowing cool water through the Condensation Column. This process is started by plugging in the submerge pump in the cooling water tank. This will initiate flow of cooling water through the Condensation Column and allow the methanol to condense and flow more easily. This also aids in recovering higher purity methanol.

Step 6: Completing the Recovery Cycle

The glycerol is heated until the flow of recovered methanol from the Outflow Tube ceases. When this occurs the heater(s) and circulation pump can be shut off allowing the glycerin to begin to cool. A sample of the glycerin should be taken to the lab to perform a Pensky-Martens flashpoint test. *CAUTION: The glycerin will be very hot when removed from the system.* Perform this test in accordance with ASTM D93. If the sample flashes at a temperature greater than 60°C, the methanol recovery process is complete and the glycerin may be removed from the system. If the sample does not flash above 60°C, the methanol recovery process should continue until the flash point of the glycerin reaches 60°C or greater.

At the completion of the recovery cycle the container storing the recovered methanol should be immediately sealed and stored in an approved flammables cabinet. A sample of the recovered methanol should also be taken to the lab to perform Specific Gravity testing in accordance with ASTM D 891 using the hydrometers. This testing will establish the purity of the recovered methanol.

Step 7: Removing Glycerin

To remove glycerin from the system, close Valve 1 and open Valve 2 (See Figure 1. Above). When the valves are in the appropriate positions the circulation pump can be started to pump the glycerin out of the system. Place an appropriate storage container at the end of the outlet hose attached to Valve 2 to collect the glycerin. This process should be done while the glycerin is still warm. If the glycerin is allowed to cool it will become more viscous and difficult to pump out of the system. Use caution if the glycerin is removed quickly after the process is complete because it will still be very hot. After all of the glycerin has been pumped from the system, the glycerin storage container(s) can be stored in a safe area for further processing or disposal and the system can be completely shut down until the next recovery cycle is performed.

Appendix A:
Methanol Material Safety Data Sheet

Material Safety Data Sheet

Methyl Alcohol, Reagent ACS, 99.8% (GC)

ACC# 95294

Section 1 - Chemical Product and Company Identification

MSDS Name: Methyl Alcohol, Reagent ACS, 99.8% (GC)**Catalog Numbers:** AC423950000, AC423950010, AC423950020, AC423955000, AC9541632, AC423952**Synonyms:** Carbinol; Methanol; Methyl hydroxide; Monohydroxymethane; Pyroxylic spirit; Wood alcohol; Wood naptha; Wood spirit; Monohydroxymethane; Methyl hydrate.**Company Identification:**Acros Organics N.V.
One Reagent Lane
Fair Lawn, NJ 07410**For information in North America, call:** 800-ACROS-01**For emergencies in the US, call CHEMTREC:** 800-424-9300

Section 2 - Composition, Information on Ingredients

| CAS# | Chemical Name | Percent | EINECS/ELINCS |
|---------|----------------|---------|---------------|
| 67-56-1 | Methyl alcohol | 99+ | 200-659-6 |

Hazard Symbols: T F**Risk Phrases:** 11 23/24/25 39/23/24/25

Section 3 - Hazards Identification

EMERGENCY OVERVIEW

Appearance: clear, colorless. Flash Point: 11 deg C. Poison! Cannot be made non-poisonous. Causes eye and skin irritation. May be absorbed through intact skin. This substance has caused adverse reproductive and fetal effects in animals. **Danger! Flammable liquid and vapor.** Harmful if inhaled. May be fatal or cause blindness if swallowed. May cause central nervous system depression. May cause digestive tract irritation with nausea, vomiting, and diarrhea. Causes respiratory tract irritation. May cause liver, kidney and heart damage.

Target Organs: Kidneys, heart, central nervous system, liver, eyes.

Potential Health Effects

Eye: Produces irritation, characterized by a burning sensation, redness, tearing, inflammation, and possible corneal injury. May cause painful sensitization to light.**Skin:** Causes moderate skin irritation. May be absorbed through the skin in harmful amounts. Prolonged and/or repeated contact may cause defatting of the skin and dermatitis.**Ingestion:** May be fatal or cause blindness if swallowed. May cause gastrointestinal irritation with nausea, vomiting and diarrhea. May cause systemic toxicity with acidosis. May cause central nervous system depression, characterized by excitement, followed by headache, dizziness, drowsiness, and nausea. Advanced stages may cause collapse, unconsciousness, coma and possible death due to respiratory failure. May cause cardiopulmonary system effects.**Inhalation:** Harmful if inhaled. May cause adverse central nervous system effects including

headache, convulsions, and possible death. May cause visual impairment and possible permanent blindness. Causes irritation of the mucous membrane.

Chronic: Prolonged or repeated skin contact may cause dermatitis. Chronic inhalation and ingestion may cause effects similar to those of acute inhalation and ingestion. Chronic exposure may cause reproductive disorders and teratogenic effects. Laboratory experiments have resulted in mutagenic effects. Prolonged exposure may cause liver, kidney, and heart damage.

Section 4 - First Aid Measures

Eyes: Immediately flush eyes with plenty of water for at least 15 minutes, occasionally lifting the upper and lower eyelids. Get medical aid immediately.

Skin: Immediately flush skin with plenty of soap and water for at least 15 minutes while removing contaminated clothing and shoes. Get medical aid if irritation develops or persists. Wash clothing before reuse.

Ingestion: If victim is conscious and alert, give 2-4 cupfuls of milk or water. Never give anything by mouth to an unconscious person. Get medical aid immediately. Induce vomiting by giving one teaspoon of Syrup of Ipecac.

Inhalation: Get medical aid immediately. Remove from exposure to fresh air immediately. If breathing is difficult, give oxygen. Do NOT use mouth-to-mouth resuscitation. If breathing has ceased apply artificial respiration using oxygen and a suitable mechanical device such as a bag and a mask.

Notes to Physician: Effects may be delayed. Ethanol may inhibit methanol metabolism.

Section 5 - Fire Fighting Measures

General Information: Containers can build up pressure if exposed to heat and/or fire. As in any fire, wear a self-contained breathing apparatus in pressure-demand, MSHA/NIOSH (approved or equivalent), and full protective gear. Water runoff can cause environmental damage. Dike and collect water used to fight fire. Vapors can travel to a source of ignition and flash back. During a fire, irritating and highly toxic gases may be generated by thermal decomposition or combustion.

Flammable Liquid. Can release vapors that form explosive mixtures at temperatures above the flashpoint. Use water spray to keep fire-exposed containers cool. Water may be ineffective. Material is lighter than water and a fire may be spread by the use of water. Vapors may be heavier than air. They can spread along the ground and collect in low or confined areas. May be ignited by heat, sparks, and flame.

Extinguishing Media: For small fires, use dry chemical, carbon dioxide, water spray or alcohol-resistant foam. Use water spray to cool fire-exposed containers. Water may be ineffective. For large fires, use water spray, fog or alcohol-resistant foam. Do NOT use straight streams of water.

Section 6 - Accidental Release Measures

General Information: Use proper personal protective equipment as indicated in Section 8.

Spills/Leaks: Scoop up with a nonsparking tool, then place into a suitable container for disposal. Use water spray to disperse the gas/vapor. Remove all sources of ignition. Absorb spill using an absorbent, non-combustible material such as earth, sand, or vermiculite. Do not use combustible materials such as saw dust. Provide ventilation. A vapor suppressing foam may be used to reduce vapors. Water spray may reduce vapor but may not prevent ignition in closed spaces.

Section 7 - Handling and Storage

Handling: Wash thoroughly after handling. Remove contaminated clothing and wash before reuse. Ground and bond containers when transferring material. Do not breathe dust, vapor, mist, or gas. Do not get in eyes, on skin, or on clothing. Empty containers retain product residue, (liquid and/or vapor), and can be dangerous. Keep container tightly closed. Avoid contact with heat, sparks and flame. Do not ingest or inhale. Use only in a chemical fume hood. Do not pressurize, cut, weld, braze, solder, drill, grind, or expose empty containers to heat, sparks or open flames.

Storage: Keep away from heat, sparks, and flame. Keep away from sources of ignition. Store in a cool, dry, well-ventilated area away from incompatible substances. Flammables-area. Keep containers tightly closed. Do not store in aluminum or lead containers.

Section 8 - Exposure Controls, Personal Protection

Engineering Controls: Use explosion-proof ventilation equipment. Facilities storing or utilizing this material should be equipped with an eyewash facility and a safety shower. Use adequate general or local exhaust ventilation to keep airborne concentrations below the permissible exposure limits. Use only under a chemical fume hood.

Exposure Limits

| Chemical Name | ACGIH | NIOSH | OSHA - Final PELs |
|----------------|--|--|--|
| Methyl alcohol | 200 ppm TWA; 250 ppm STEL; skin - potential for cutaneous absorption | 200 ppm TWA; 260 mg/m ³ TWA 6000 ppm IDLH | 200 ppm TWA; 260 mg/m ³ TWA |

OSHA Vacated PELs: Methyl alcohol: 200 ppm TWA; 260 mg/m³ TWA; 250 ppm STEL; 325 mg/m³ STEL

Personal Protective Equipment

Eyes: Wear chemical goggles.

Skin: Wear appropriate protective gloves to prevent skin exposure.

Clothing: Wear appropriate protective clothing to prevent skin exposure.

Respirators: A respiratory protection program that meets OSHA's 29 CFR §1910.134 and ANSI Z88.2 requirements or European Standard EN 149 must be followed whenever workplace conditions warrant a respirator's use.

Section 9 - Physical and Chemical Properties

Physical State: Liquid

Appearance: clear, colorless

Odor: alcohol-like - weak odor

pH: Not available.

Vapor Pressure: 128 mm Hg @ 20 deg C

Vapor Density: 1.11 (Air=1)

Evaporation Rate: 5.2 (Ether=1)

Viscosity: 0.55 cP 20 deg C

Boiling Point: 64.7 deg C @ 760.00mm Hg

Freezing/Melting Point: -98 deg C

Autoignition Temperature: 464 deg C (867.20 deg F)

Flash Point: 11 deg C (51.80 deg F)

Decomposition Temperature: Not available.

NFPA Rating: (estimated) Health: 1; Flammability: 3; Reactivity: 0

Explosion Limits, Lower: 6.0 vol %

Upper: 36.00 vol %

Solubility: miscible

Specific Gravity/Density: .7910g/cm³

Molecular Formula:CH₄O**Molecular Weight:**32.04

Section 10 - Stability and Reactivity

Chemical Stability: Stable under normal temperatures and pressures.**Conditions to Avoid:** High temperatures, incompatible materials, ignition sources, oxidizers.**Incompatibilities with Other Materials:** Acids (mineral, non-oxidizing, e.g. hydrochloric acid, hydrofluoric acid, muriatic acid, phosphoric acid), acids (mineral, oxidizing, e.g. chromic acid, hypochlorous acid, nitric acid, sulfuric acid), acids (organic, e.g. acetic acid, benzoic acid, formic acid, methanoic acid, oxalic acid), azo, diazo, and hydrazines (e.g. dimethyl hydrazine, hydrazine, methyl hydrazine), isocyanates (e.g. methyl isocyanate), nitrides (e.g. potassium nitride, sodium nitride), peroxides and hydroperoxides (organic, e.g. acetyl peroxide, benzoyl peroxide, butyl peroxide, methyl ethyl ketone peroxide), epoxides (e.g. butyl glycidyl ether), Oxidants (such as barium perchlorate, bromine, chlorine, hydrogen peroxide, lead perchlorate, perchloric acid, sodium hypochlorite)., Active metals (such as potassium and magnesium)., acetyl bromide, alkyl aluminum salts, beryllium dihydride, carbontetrachloride, carbon tetrachloride + metals, chloroform + heat, chloroform + sodium hydroxide, cyanuric chloride, diethyl zinc, nitric acid, potassium-tert-butoxide, chloroform + hydroxide, water reactive substances (e.g. acetic anhydride, alkyl aluminum chloride, calcium carbide, ethyl dichlorosilane).**Hazardous Decomposition Products:** Carbon monoxide, irritating and toxic fumes and gases, carbon dioxide, formaldehyde.**Hazardous Polymerization:** Will not occur.

Section 11 - Toxicological Information

RTECS#:**CAS#** 67-56-1: PC1400000**LD50/LC50:**

CAS# 67-56-1:

Draize test, rabbit, eye: 40 mg Moderate;

Draize test, rabbit, eye: 100 mg/24H Moderate;

Draize test, rabbit, skin: 20 mg/24H Moderate;

Inhalation, rat: LC50 = 64000 ppm/4H;

Oral, mouse: LD50 = 7300 mg/kg;

Oral, rabbit: LD50 = 14200 mg/kg;

Oral, rat: LD50 = 5628 mg/kg;

Skin, rabbit: LD50 = 15800 mg/kg;

Carcinogenicity:

CAS# 67-56-1: Not listed by ACGIH, IARC, NIOSH, NTP, or OSHA.

Epidemiology: Methanol has been shown to produce fetotoxicity in the embryo or fetus of laboratory animals. Specific developmental abnormalities include cardiovascular, musculoskeletal, and urogenital systems.**Teratogenicity:** Effects on Newborn: Behavioral, Oral, rat: TDLo=7500 mg/kg (female 17-19 days after conception). Effects on Embryo or Fetus: Fetotoxicity, Inhalation, rat: TCLo=10000 ppm/7H (female 7-15 days after conception). Specific Developmental Abnormalities: Cardiovascular, Musculoskeletal, Urogenital, Inhalation, rat: TCLo=20000 ppm/7H (7-14 days after conception).**Reproductive Effects:** Paternal Effects: Spermatogenesis: Intraperitoneal, mouse TDLo=5 g/kg (male 5 days pre-mating). Fertility: Oral, rat: TDLo = 35295 mg/kg (female 1-15 days after conception). Paternal Effects: Testes, Epididymis, Sperm duct: Oral, rat: TDLo = 200 ppm/20H (male 78 weeks pre-mating).**Neurotoxicity:** No information available.

Mutagenicity: DNA inhibition: Human Lymphocyte = 300 mmol/L. DNA damage: Oral, rat = 10 umol/kg. Mutation in microorganisms: Mouse Lymphocyte = 7900 mg/L. Cytogenetic analysis: Oral, mouse = 1 gm/kg.

Other Studies: Standard Draize Test (Skin, rabbit) = 20 mg/24H (Moderate) Standard Draize Test: Administration into the eye (rabbit) = 40 mg (Moderate). Standard Draize test: Administration into the eye (rabbit) = 100 mg/24H (Moderate).

Section 12 - Ecological Information

Ecotoxicity: Fish: Fathead Minnow: 29.4 g/L; 96 Hr; LC50 (unspecified) Goldfish: 250 ppm; 11 Hr; resulted in death Rainbow trout: 8000 mg/L; 48 Hr; LC50 (unspecified) Rainbow trout: LC50 = 13-68 mg/L; 96 Hr.; 12 degrees C Fathead Minnow: LC50 = 29400 mg/L; 96 Hr.; 25 degrees C, pH 7.63 Rainbow trout: LC50 = 8000 mg/L; 48 Hr.; Unspecified ria: Phytobacterium phosphoreum: EC50 = 51,000-320,000 mg/L; 30 minutes; Microtox test No data available.

Environmental: Dangerous to aquatic life in high concentrations. Aquatic toxicity rating: TLM 96 > 1000 ppm. May be dangerous if it enters water intakes. Methyl alcohol is expected to biodegrade in soil and water very rapidly. This product will show high soil mobility and will be degraded from the ambient atmosphere by the reaction with photochemically produced hydroxyl radicals with an estimated half-life of 17.8 days. Bioconcentration factor for fish (golden ide) < 10. Based on a log Kow of -0.77, the BCF value for methanol can be estimated to be 0.2.

Physical: No information available.

Other: None.

Section 13 - Disposal Considerations

Chemical waste generators must determine whether a discarded chemical is classified as a hazardous waste. US EPA guidelines for the classification determination are listed in 40 CFR Parts 261.3. Additionally, waste generators must consult state and local hazardous waste regulations to ensure complete and accurate classification.

RCRA P-Series: None listed.

RCRA U-Series: CAS# 67-56-1: waste number U154; (Ignitable waste).

Section 14 - Transport Information

| | US DOT | IATA | RID/ADR | IMO | Canada TDG |
|-------------------------|----------|------|---------|-----|--------------------|
| Shipping Name: | METHANOL | | | | METHANOL |
| Hazard Class: | 3 | | | | 3(6.1) |
| UN Number: | UN1230 | | | | UN1230 |
| Packing Group: | II | | | | II |
| Additional Info: | | | | | FLASHPOINT 11 C |

Section 15 - Regulatory Information

US FEDERAL

TSCA

CAS# 67-56-1 is listed on the TSCA inventory.

Health & Safety Reporting List

None of the chemicals are on the Health & Safety Reporting List.

Chemical Test Rules

None of the chemicals in this product are under a Chemical Test Rule.

Section 12b

None of the chemicals are listed under TSCA Section 12b.

TSCA Significant New Use Rule

None of the chemicals in this material have a SNUR under TSCA.

SARA**Section 302 (RQ)**

CAS# 67-56-1: final RQ = 5000 pounds (2270 kg)

Section 302 (TPQ)

None of the chemicals in this product have a TPQ.

SARA Codes

CAS # 67-56-1: acute, flammable.

Section 313

This material contains Methyl alcohol (CAS# 67-56-1, 99%), which is subject to the reporting requirements of Section 313 of SARA Title III and 40 CFR Part 373.

Clean Air Act:

CAS# 67-56-1 is listed as a hazardous air pollutant (HAP). This material does not contain any Class 1 Ozone depleters. This material does not contain any Class 2 Ozone depleters.

Clean Water Act:

None of the chemicals in this product are listed as Hazardous Substances under the CWA. None of the chemicals in this product are listed as Priority Pollutants under the CWA. None of the chemicals in this product are listed as Toxic Pollutants under the CWA.

OSHA:

None of the chemicals in this product are considered highly hazardous by OSHA.

STATE

CAS# 67-56-1 can be found on the following state right to know lists: California, New Jersey, Florida, Pennsylvania, Minnesota, Massachusetts.

California No Significant Risk Level: None of the chemicals in this product are listed.

European/International Regulations**European Labeling in Accordance with EC Directives****Hazard Symbols:**

T F

Risk Phrases:

R 11 Highly flammable.

R 23/24/25 Toxic by inhalation, in contact with skin and if swallowed.

R 39/23/24/25 Toxic : danger of very serious irreversible effects through inhalation, in contact with skin and if swallowed.

Safety Phrases:

S 16 Keep away from sources of ignition - No smoking.

S 36/37 Wear suitable protective clothing and gloves.

S 45 In case of accident or if you feel unwell, seek medical advice immediately (show the label where possible).

S 7 Keep container tightly closed.

WGK (Water Danger/Protection)

CAS# 67-56-1: 1

Canada

CAS# 67-56-1 is listed on Canada's DSL List. CAS# 67-56-1 is listed on Canada's DSL List.

This product has a WHMIS classification of B2, D1A, D2B.

CAS# 67-56-1 is listed on Canada's Ingredient Disclosure List.

Exposure Limits

CAS# 67-56-1: OEL-ARAB Republic of Egypt: TWA 200 ppm (260 mg/m³); Skin OEL-AUSTRALIA: TWA 200 ppm (260 mg/m³); STEL 250 ppm; Skin OEL-BELGIUM: TWA 200 ppm (262 mg/m³); STEL 250 ppm; Skin OEL-CZECHOSLOVAKIA: TWA 100 mg/m³; STEL 500 mg/m³ OEL-DENMARK: TWA 200 ppm (260 mg/m³); Skin OEL-FINLAND: TWA 200 ppm (260 mg/m³); STEL 250 ppm; Skin OEL-FRANCE: TWA 200 ppm (260 mg/m³); STEL 1000 ppm (1300 mg/m³) OEL-GERMANY: TWA 200 ppm (260 mg/m³); Skin OEL-HUNGARY: TWA 50 mg/m³; STEL 100 mg/m³; Skin OEL-JAPAN: TWA 200 ppm (260 mg/m³); Skin OEL-THE NETHERLANDS: TWA 200 ppm (260 mg/m³); Skin OEL-THE PHILIPPINES: TWA 200 ppm (260 mg/m³) OEL-POLAND: TWA 100 mg/m³ OEL-RUSSIA: TWA 200 ppm; STEL 5 mg/m³; Skin OEL-SWEDEN: TWA 200 ppm (250 mg/m³); STEL 250 ppm (350 mg/m³); Skin OEL-SWITZERLAND: TWA 200 ppm (260 mg/m³); STEL 400 ppm; Skin OEL-THAILAND: TWA 200 ppm (260 mg/m³) OEL-TURKEY: TWA 200 ppm (260 mg/m³) OEL-UNITED KINGDOM: TWA 200 ppm (260 mg/m³); STEL 250 ppm; Skin OEL IN BULGARIA, COLOMBIA, JORDAN, KOREA check ACGIH TLV OEL IN NEW ZEALAND, SINGAPORE, VIETNAM check ACGI TLV

Section 16 - Additional Information

MSDS Creation Date: 7/21/1999

Revision #4 Date: 3/14/2001

The information above is believed to be accurate and represents the best information currently available to us. However, we make no warranty of merchantability or any other warranty, express or implied, with respect to such information, and we assume no liability resulting from its use. Users should make their own investigations to determine the suitability of the information for their particular purposes. In no event shall Fisher be liable for any claims, losses, or damages of any third party or for lost profits or any special, indirect, incidental, consequential or exemplary damages, howsoever arising, even if Fisher has been advised of the possibility of such damages.

Messiah College Biodiesel Fuel Generation Project

DE-FG36-08GO88068

Appendix D. – Manuals and Reports

Appendix D.3.

2010 Biodiesel Final Report

Messiah College Biodiesel Fuel Generation Project
DE-FG36-08GO88068
Appendix D. – Manuals and Reports

Final Project Report

Biodiesel Project

Messiah College

5/10/2010

Abstract

The Biodiesel project is a long complicated project that consists of multiple facets. This paper will document the progress and advancements of two of these facets: biodiesel testing and methanol recovery. As well as documenting the progress of the project, it will serve as a guide for future work as outlined in the CDP SOPO, and CDP SOW. Included in the report is the most updated Gantt chart for the CDP.

Introduction

Biodiesel is an environmentally friendly alternative to petroleum-based diesel fuel for use in unmodified diesel engines or as a fuel oil substitute. Biodiesel is produced by a reaction that occurs when vegetable oils are combined with an alcohol and a catalyst. Its primary advantage is that it is an agri-based, non-toxic, biodegradable renewable fuel. Biodiesel combustion produces less carbon monoxide, unburned hydrocarbons and particulate emissions than petrodiesel.

Although several large-scale biofuel production plants are in various stages of development in central Pennsylvania, processor and process research and development has not yet been accomplished to support ASTM D 6751 certifiable biodiesel production by entrepreneurs and institutions seeking to leverage the opportunities afforded by biodiesel for recycling and other small-scale applications. Small scale production could become a viable commercial option, given the low cost of production equipment. However, equipment for process research and quality testing is expensive, preventing many small-scale producers from improving or certifying their processes. Moreover, there is no local laboratory readily available to small-scale producers in central Pennsylvania that provides access to test equipment and expertise needed for process optimization and quality assurance.

This project will meet these needs by establishing a laboratory facility and a research and development program that will advance biodiesel processor and process design in support of small scale producers. Our work will include researching the ability to produce ASTM certified fuel using various feedstocks, possibly including recycled or unconventional materials, and production ingredients. The project will also develop scalable processor and process designs suitable for ASTM D 6751 certifiable small-scale biodiesel production.

Messiah College personnel have already gained significant working knowledge about biodiesel production by designing and testing their own small scale system that produces biodiesel from waste cooking oil collected from the College's main campus dining facilities. The College is currently making small quantities of biodiesel, testing it in unmodified diesel engines and using it as a supplement to heating fuel oil. This project will enable College personnel to extend their work into applied research and development that will support future commercial applications of small-scale biodiesel production. The academic work of this project will support entrepreneurs and institutions in our region seeking to plan small business entrepreneurial ventures in biodiesel production.

The objectives for the Messiah College Congressionally Directed Project (CDP) include the following:

Prepare a laboratory facility for the development and optimization of processors and processes, ASTM quality assurance, and performance testing of biodiesel fuels.

Developing scalable processor and process designs suitable for ASTM certifiable small-scale biodiesel production, with the goals of cost reduction and increased quality.

Conduct research into biodiesel process improvement and cost optimization using various biodiesel feedstocks and production ingredients.

The project has succeeded in completing the first of these three tasks and is currently working on a way to test the successfulness of the second through the research done in the third task. The project has also been working on several side projects that deal with the by-product disposal of biodiesel, including methanol recovery.

Currently the methanol recovery team has two working prototypes. One is capable of processing 50 gallons of glycerine while the other can process 8 gallons. The team is currently undertaking process optimization research that will increase the productivity of the system while making it more cost effective. They have successfully gathered much data that will allow them to identify the key process variables of methanol recovery and are working to make the appropriate conclusions and correlations from the data.

Background

Context

See the **Introduction** section for information on context.

Technical

Biodiesel Testing

All relevant technical background information can be found in the following.

<\\collab-main\collabenergy\Biodiesel\Resources>

Methanol Recovery

All relevant technical information can be found here:

<\\collab-main\collabenergy\Biodiesel\Methanol Recovery System>

Narrative

Biodiesel Testing

Messiah College Biodiesel Fuel Generation Project

DE-FG36-08GO88068

Appendix D. – Manuals and Reports

The testing aspect of the biodiesel project set out to run the first wave of tests within the newly equipped biodiesel testing lab. Previous groups researched and acquired equipment capable of 5 ASTM D6751 tests. The first phase of this team was to lay out the proposed tests and testing procedures to be completed throughout the course of the school year. With all of the tests laid out, and all of the equipment calibrated or working properly, the team moved on to creating biodiesel samples.

A total of 18 unique biodiesel samples were reacted through the course of the school year. The samples were all created by varying different aspects of the production process. The testing team set out to test the effect of varying catalyst type and quantity, as well as the effect of water washing on biodiesel quality. The samples were created according to Table 1.1.

| KOH | Unwashed | Single Wash | Fully Washed |
|------------------|----------|-------------|--------------|
| Minimal Catalyst | K1 | K2 | K3 |
| Precise Catalyst | K4 | K5 | K6 |
| Excess Catalyst | K7 | K8 | K9 |

| NaOH | Unwashed | Single Wash | Fully Washed |
|------------------|----------|-------------|--------------|
| Minimal Catalyst | N1 | N2 | N3 |
| Precise Catalyst | N4 | N5 | N6 |
| Excess Catalyst | N7 | N8 | N9 |

Table 1.1

A total of seven tests were laid out in order to test the biodiesel samples for different aspects of quality. The seven tests were:

Water & Sediment (Centrifuge)

Acid Number (Auto-titrator)

Flash point (Pensky-Martin closed cup flash point tester)

Total and Free Glycerine (GC)

Cloud Point (Cloud Point Bath)

Sulfated Ash Test (Furnace)

27/3 test

The first 5 tests required equipment within the biodiesel testing lab. The 6th test, the sulfated ash utilized the Thermolyne Furnace owned by the engineering department. The final test, the 27/3 test, is a common test for small scale or “backyard” producers. The 27/3 test was executed to test the validity of the test more so than gaining technical data.

The sample production took significantly more time than expected, due to initial lack of equipment, shipping delays and other unforeseen circumstances. Eventually the samples were all created, and testing could begin.

Five of the seven tests went according to the procedure laid out. The Gas chromatograph fell out of calibration, and the team was unable to use the GC for quality testing. The sulfated ash test required some equipment that was unavailable, but a suitable substitute was found and used for the testing.

To be inserted: results of testing

Methanol Recovery

In the 2009-2010 academic year, the methanol recovery project team worked on digitizing the methanol recovery system to work towards integrating methanol recovery into the biodiesel processor.

The year began working off past successes. The two important successes from prior years were the ability to produce 95%+ purity methanol and reducing the methanol content of the waste glycerin to the point it was no longer considered a hazardous material. The first success enabled the team to reuse the recovered waste methanol in the biodiesel processor without the need to add fresh methanol to increase the purity. The latter success allowed the biodiesel team to enter a partnership with another Messiah College student group, the Earthkeepers, to use the waste glycerin in the school's compost pile to speed up the decomposition. The high purity methanol is produced using an eight gallon methanol recovery system. This system was designed by the biodiesel intern over the summer. This system used a few major components from the old 55 gallon system including the column head, pump and electrical box. The old system used an open-loop system to cool the column head. This system required an exorbitant amount of fresh water. It was also discovered that the highest purity methanol was produced using very cold water, which is hard to achieve with five gallon buckets of ground water. For this reason, a closed loop system which utilized an intercooler was created for the methanol recovery system. This allowed the system to operate continuously on less than 20 gallons of water. Furthermore, the water supplied to the system is near the freezing point. The coolness of the column condensation column is credited with helping improve the purity of the methanol. Once the system once operating with desirable resulting purity, a wireless data acquisition system (DAQ) was combined with thermocouples to allow the team to digitally record the temperature as a function of time for various sections of the system. The team then purchased a laptop for use with the DAQ to record and store data on the thermal behavior of the methanol recovery system. Since purity of recovered methanol had been dealt with, the team decided to move onto reviving the 55 gallon methanol recovery system to produce the volume capabilities of the team. This meant designing and implementing new parts as well as integrating parts available to the biodiesel team from past project work. The heater used on the revived system came from past project work. The pump was purchased and is the same pump used on the 8 gallon system. Biodiesel team members built a new column head similar to the one used on the smaller system under the supervision of the biodiesel intern. This portion of the project has just been implemented and will allow future biodiesel project members to compare and contrast between the smaller and larger systems.

Project Plan

Methanol Recovery

The methanol recovery team sought to follow the following Gantt Chart to guide its work for the '09-'10 academic year.

| September-October | November-January | January-March | March | April | | May |
|---------------------------------|--|--|--|--|----------|-----|
| Finish making changes to M.R.U. | Begin testing with the DAQ to find critical temperatures for methanol recovery | Test the M.R.U and begin to draw conclusions | Make changes to M.R.U. to address critical temps | Test critical temps again to measure the results of the changes made to the M.R.U. | SSE prep | SSE |

The methanol recovery team began the year working on finishing modifications made to the methanol recovery unit at the end of the summer. This required acquiring of several important parts some of which took longer than initially expected. Once the Methanol Recovery Unit (MRU) was made operational, we began using the Data Acquisition Unit (DAQ) to monitor system temperatures. Since the MRU took a while to operate, it took us a while to gather a sufficient range of samples. From this data, we were able to draw some conclusions on several process elements. This data also confirmed multiple process variables. Because the last two objectives took longer to complete than initially expected, we did not get to make as many changes to the MRU as we would have liked. We also did not get to test these changes to determine their effect on the process variables previously determined. Overall this year's team accomplished much and has left the project in a position for next year's team to succeed.

Phase Analysis

The Biodiesel Project and its sub-projects are currently in the following phases: research, design, testing and prototyping. Due to the nature of the project, it will always be in the research phase as it is an ongoing tenant of the project. The project is also in the testing phase. Using the lab, the team is working on testing the quality of our biodiesel produced against the ASTM standard. Using this information, the team will be able to design, redesign the testbed processor in order to produce quality fuel. The methanol recovery project is currently in the prototyping and testing phase. The project sub-team is working on testing the quality of their prototype and are working on improving it based on the results of their testing.

Schedule

Our project is currently proceeding as laid out in the project plan that was updated in Fall 2009. Currently both sub-teams are proceeding close to being on schedule with only minor setbacks thus far. The testing team was delayed in their work because of equipment issues. The methanol recovery team has had similar problems. Their work was delayed in that they did not receive the equipment they were hoping for in time to implement it into their process. Because of this, they have not made the changes to the prototype as laid out in the project plan. Attached in the appendices is the most up to date Gantt chart for the Biodiesel project.

Resource Analysis

The needs of the Biodiesel project are currently being sufficiently met by the DOE CDP grant. No other considerations for resources are needed at this time.

Budget

The overall budget for the three year grants period includes three main classifications of funds. The first is the Direct Funds which entail the total monies available for all activities associated with the CDP SOPO and SOW. The total monies available in direct funds for the three year period of the grant is \$328,000 as see in Table 1.2 below. The second classification is the Indirect Funds which are monies that are intended to offset the indirect costs associated with the work being done to accomplish the grant objectives. The total of these funds is directly correlated to the Direct Funds at a 50% rate therefore the total, as seen below, for the Indirect Funds is \$164,000. The final classification of funding include with this work is the Cost Share requirement. The Cost Share is the amount of funds required to be contributed toward the project by Messiah College or other outside partners to meet the expectations set out by the grant agreement. According to this agreement the total Cost Share funding must be 20% of the overall funds for the grant. As seen below, this total is \$123,000 for the three year grant period. The combination of the Direct, Indirect and Cost Share funds establish the overall grant budget of \$615,000.

| | Overall | Current | % |
|----------------|---------------------|---------------------|--------------|
| Direct Funds | \$328,000.00 | \$182,176.00 | 55.5% |
| Indirect Funds | \$164,000.00 | \$91,089.00 | 55.5% |
| Cost Share | \$123,000.00 | \$35,055.00 | 28.5% |
| Total: | \$615,000.00 | \$308,320.00 | 50.1% |

Table 1.2: Grant Funding Breakdown

The current total for the three classifications of grant funding can be seen in the column labeled “Current” in Table 1.2 above. The percent of the total funds expended to date can also be seen in the column labeled “%”. Currently about half of the Direct and Indirect Funds for the grant have been spent which correlates well with the remaining 1.5 years of the 3 year grant period. The Cost Share requirements in slightly low for the overall grant period, however this was expected in the planning for the grant budget and any unmet Cost Share requirements will be accommodated with remaining Indirect Funds at the end of the grant period.

Future work

Biodiesel Testing

Being the first team to use the biodiesel testing lab, the team laid the groundwork for many other tests to be performed in the testing lab. This year's team focused on catalyst quantity and type, as well as water washing, future testing could include such things as reaction temperature, reaction time, alcohol type and amount, settling time, various washing methods (drywash vs. water wash) or any variation. The tests will greatly facilitate the future production of a biodiesel processor by specifically laying out the best production techniques.

Methanol Recovery

Future work for the methanol recovery system should be as follows: continue gathering temperature vs. time curves, compare quality of recovered methanol between short and long column head, begin integration of the PLC, integrate system into the larger biodiesel process, and restructure PLC program based on time-temp curves. These things will continue to further refine the methanol recovery process and will contribute to the already extensive knowledge we as a project have gained on the finer details of methanol recovery.

Conclusion

Over the course of the last year, the biodiesel project made significant progress. The biodiesel testing team made important strides that will continue the core of the grant work. The methanol recovery team also made improvements in the realm of methanol recovery. Though much work was accomplished this semester by both projects, there is much more work to be done. Testing will continue over the next year and a half under the canopy of the grant. Work will continue for both projects as laid out in the future work section of this report and also as directed by the project manager.

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Appendix

Methanol Recovery results can be found here: <\\collab-main\collabenergy\Biodiesel\Methanol Recovery System\Methanol Recovery2009-2010>

The most current Gantt chart for the biodiesel project can be found here:

http://www.thecollaboratoryonline.org/wiki/File:Biodiesel_Gantt_Chart.pdf

Links to the CDP SOPO and CDP SOW can be found here: <\\collab-main\collabenergy\Biodiesel\CDP>

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Appendix D.4.

2010 Biodiesel Production Manual

1. Waste Vegetable Oil Collection:

Use vacuum pump on ambulance to collect oil

Switch the (Mod. Disc) button on the dash board to activate power for vacuum pump

If air is blowing out of tubes then pump is ready to vacuum

If air is being sucked into tubes pump is pressurized

Turn all 4 valves if you want to switch from one to the other

Turn valve on hose on vacuum pump to vacuum or blow out oil

Collect oil from tote behind Lottie nelson

Collect oil from tote behind Bakers dinner (15 South about 10 miles on right)

Use vacuum pump to transfer oil from ambulance into holding tank in the garage

Titration

Take sample of Waste Vegetable Oil as it is drawn into the H/M tank

Completely dissolve 1g of KOH in one liter of distilled water (This solution can be used for many titrations). Store it in hazard-labeled bottle in shelving.

Mix 10mL of isopropyl alcohol in small container with 1mL of a sample of Waste Vegetable Oil. (Be exact with these measurements)

Add 3 drops of phenolphthalein

Using a measuring cylinder drop 0.1mL at a time of the KOH/water solution into the WVO/isopropyl solution.

Mix the solution vigorously as each drop is added. If the solution remains pink for 30 seconds, and endpoint has been reached. (Equivalent to a pH of 8.5)

Calculate the required KOH input for the biodiesel reaction using the following equation:

$$(7+\#\text{mL solution added}) = (\# \text{ grams KOH added per Liter WVO})$$

$$1 \text{ Liter} = 0.264 \text{ Gallons}$$

$$1 \text{ Gallon} = 3.785 \text{ Liters}$$

Methoxide Mixing

*Use goggles, gloves, and mask for this procedure

Measure out necessary amount of KOH

Acquire the proper amount of Methanol (Ration: 4 parts WVO to 1 part methanol)

For Messiahs processor 41-42 gal WVO and 8-9 gal of methanol

Place 95 micron filter at the bottom of the Methoxide tank

Pour in half the required methanol

Slowly pour in half the required KOH

Pour in final half of required methanol

Slowly pour in final half of required KOH

Allow ample amount of time for solution to mix (10 min)

Heating and Mixing

Valves 3 and 6 should be open.

All other valves should be closed

Turn on switches for pump and heater

Heat WVO until it reaches a temperature of 140 degrees F or 60 degrees C

Open valve 4. Do not open all the way. Methoxide mixture should be released slowly enough so that the oil and methoxide mixture are mixing just above the main loop a little bit up the tube of where the methoxide mixture is coming from.

Allow all the methoxide mixture to be mixed in and then close valve 4.

Allow a minimum of 5 minutes for mixture to circulate in the main stream

Turn off the pump and heater

Let the solution settle for 12-18 hours

Glycerin Removal

Make sure batch has had proper amount of settling time; minimum 12-18 hours

Get 3 five gallon pails (15 gallons) ready to collect glycerin

Open valve 2 and empty all glycerin (dark colored liquid) from the Heating &

Mixing tank into the five-gallon buckets

Transport recovered glycerin to where it can be used by methanol recovery system

Washing

Close valves 1,2,4,5, and 6.

Open valves 3 and 7

Turn on pump and pump biodiesel from heating & mixing tank into the washing tank

Attach green hose to water pump outside garage

Wash 1: Use hose to add 10 gallons of water to the wash tank. Add water very slowly otherwise emulsification will occur. Run the bubbler for 30 minutes and then allow the water to settle for 1 hour. After 1 hour drain the wash water out (valve 5) into 5 gallon buckets and dump into 55 gallon drum marked wash water

Wash 2: Use hose to add 10 gallons of water to the wash tank. Add water very slowly otherwise emulsification will occur. Run the bubbler for 2 hours and then allow the water to settle for 2 hours. After 2 hours drain the water out (valve 5) into 5 gallon buckets and dump into 55 gallon drum marked wash water

Wash 3: Use hose to add 10 gallons of water to the wash tank. Add water very slowly otherwise emulsification will occur. Run bubbler for 3 hours and then allow the water to settle for 3 hours. After settling, drain the water out (valve 5) into 5 gallon buckets and dump into 55 gallon drum marked wash water

Using vacuum pump transport wash water to the union and dump it down the grease trap.

After washing is complete and wash water has been drained off turn on bubbler and let it run for 12-18 hours. Any water left in the biodiesel will then evaporate

Storage and Distribution of Biodiesel

Turn on switch for transfer pump and pump all of the biodiesel from the washing tank into the 55 gallon drum.

Biodiesel can then be used in a variety of ways

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Appendix D.5.

2011 Centrifuge Testing Report

Table of Contents:

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Abstract

The Biodiesel Centrifuge Project attempts to determine how waste vegetable oil can be best purified before it goes through the chemical process to be turned into biodiesel. This centrifuging process involves using a Raw Power Centrifuge to separate by density the pure oil from the impurities in the oil. The project's focus is to determine the best RPM speed the centrifuge should be run at and the best flow rate the oil should be put through the centrifuge to efficiently create the most pure oil. The way in which these specifications are determined is by holding one of the variables constant and then varying the other and comparing the results.

Introduction

The biodiesel centrifuge project seeks to obtain the most pure quality of waste vegetable oil as possible prior to the biodiesel reaction to ensure the highest quality of fuel to be produced. To accomplish this purpose the project is using a Raw Power Centrifuge to centrifuge its collected wasted vegetable oil. Research is being done on what the best flow rate and RPM speed for the centrifuge is so that the most pure oil can be produced and the most efficient way the process can be run. A testing procedure has been written up and implementation of the equipment necessary to run the procedure has been completed. Testing procedures have been determined, samples have taken and tested, and results quantified and analyzed. After analyzing our data we were able to conclude that the most effective flow rate into the centrifuge should be 10 gals/hr and the most effective RPM speed 3600. In the future the centrifuge will be set to these specifications to produce as pure an oil to be reacted as possible.

Background

Context

The United States Department of Energy provided Messiah College with a grant to research small scale biodiesel production. Since the time of the issuing of this grant the biodiesel group within the larger energy group has sought to provide feedback to the United States Department of Energy as to how the most effective and high quality small scale biodiesel can be produced. The information gathered will then be disseminated for the purpose of informing small, non industrial producers about how to properly make biodiesel so they can produce a high quality fuel, ensure healthy procedure as well as environmentally safe procedures, and produce their fuel in the most cost effective way as possible. By providing this information the biodiesel group will be able help produce environmentally friendly energy that will reduce fuel costs and support the re-use of materials such as used cooking oil.

The biodiesel group has delved into many aspects of the biodiesel process including pre-reaction oil purification, water washing techniques, and processor design, each of these having many sub research areas included in them. The biodiesel centrifuge project assessed the area of pre-reaction waste vegetable oil purification, reasoning that a higher quality fuel could be produced if the oil to be reacted was more pure to begin with.

Technical

It takes no lengthy explanation to reason that by starting with more pure waste vegetable oil a more pure biodiesel will be able to be produced. The method determined to best satisfy the need

of purifying the oil was by centrifuging the oil to separate out pure waste vegetable oil from impurities by density.

The biodiesel centrifuge project decided to use a Raw Power Centrifuge to accomplish the task of oil purification. A more detailed overview of the specific centrifuge we used can be found at the link below. <http://www.wvodesigns.com/store/wvo-centrifuges/basic-raw-power-centrifuge.html> The reason for the group choosing this centrifuge was many. This centrifuge provided a way to heat the incoming oil allowing better separation to occur. Also the centrifuge was variable speed, meaning the RPM speed at which the centrifuge spins can be controlled by turning a knob and looking at the corresponding digital readout in the control box. The biodiesel centrifuge project concluded that the raw power centrifuge would be able serve the purpose of meeting the requested goals.

The biggest difficulty with the centrifuging process is that the waste vegetable oil being input into the centrifuge has a large variability in its purity depending on how pure the oil is at pickup. This is a problem because it is then difficult to determine to extent to which the centrifuging process is effective because there can be no set level of purity. Also in colder weather the centrifuge may experience difficulties because oil is more viscous at lower temperatures. To overcome this problem we did all our testing so that it could be analyzed on a percentage basis. Our sample collecting procedure called for us to take a control sample of the oil before it had be processed through the centrifuge and then compare that sample with a sample that had been put through the centrifuge. In this way we were able to determine a percentage difference in our oil and thus know how well the centrifuge was purifying our oil.

At the present moment we have a very efficient and streamline centrifuging system that can be seen in **(Figure 1.1)** in the appendix of this report. The problems we have dealt with have been solved and proper centrifuging parameters have been established.

Narrative

Over the past year much progress has been made in the pre-reaction waste vegetable oil purification process. During the summer, in the projects infant stage, the project was able to acquire the actual centrifuge. Upon acquisition of the centrifuge a system was developed to get oil to run into the centrifuge and then out into the heating and mixing tank ready to be processed. The impure waste vegetable oil would be pumped from a large holding tank up into a smaller holding tank that was mounted on the top of racking. This oil, being at a high elevation, would then be gravity fed, with allowance for flow rate control by a valve, into the centrifuge. After being separated the pure waste vegetable oil would run by gravity into another holding tank and the impurities would flow into a waste vessel. Once in the clean holding tank the waste vegetable oil could easily be pumped into the heating and mixing tank ready to be reacted. A diagram of the above explained process can be seen in the appendix of this report **(Fig. 1.1)**.

Once the logistics of running the centrifuge had been determined, a testing procedure was required to ensure that testing was the same every time. This procedure was written up and followed for the entirety of testing. This procedure can be seen in the appendix.

Once samples were collected they were brought down to the biodiesel lab to be analyzed. Two tests were performed on our samples; a Water and Sediment test as well as a titration test, to determine how pure the oil was after being centrifuged as opposed to before being centrifuged. These two tests are outlined in the appendix.

The group found that at slower flow rates a more pure oil could be produced. It was determined that since the centrifuging of oil was not a rate limiting step in the process of making biodiesel, even at very slow flow rates, a slow flow rate of 10 gallons per hour would be implemented to ensure the highest quality of waste vegetable oil. Therefore to centrifuge enough oil to make a batch of biodiesel the time taken would be 4 hours. After analyzing test results the group was also able to determine the proper RPM speed to be 3600 RPM's.

The goals of this project were met since oil was able to be purified to a substantial extent. The group observed that titration numbers of the oil decreased, not only creating a better final biodiesel product but lower costs as well, due to the decreased amount of KOH that had to be used during the reaction.

The most difficult obstacle to overcome was the fact that the collected oil (the oil in the unpurified holding tank) varied in purity. Therefore the purity of the oil after being centrifuged would vary somewhat to the purity of oil that went into the centrifuge. To solve this problem a control sample of waste vegetable oil was taken prior to the oil passing through the centrifuge. This oil was then compared to oil after it had been centrifuged. The two samples were compared on a percentage basis to determine how well the centrifuge was actually purifying the oil.

This project was very helpful in showing how important professional data collecting and testing procedures are to the project as well as the importance of staying on schedule in the completion of tasks.

Project Plan

Below is my project planning article for the year.

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Goals/Deliverables:

To research and discover the most effective flow rate and RPM speed of the Centrifuge

To research and discover the most effective RPM speed of the Centrifuge

Test Centrifuge speeds in increments of 1000 RMP from 2200-4200 RPM

Test titration numbers of samples before and after different RPM speeds

Test water and sediment concentration before and after different RPM speeds

To research and discover the most effective flow rate through Centrifuge

Figure out way to test variance in flow rate of oil in and out of Centrifuge

Propose flow rate increments to test

Test titration numbers of samples before and after different flow rates

Test water and sediment concentration before and after different flow rates

Write and report results

Procedure

The procedure for testing will be done as follows: A sample of Waste Vegetable Oil (WVO) will be taken before the WVO has passed through the centrifuge. This will be the control of the testing procedure. 10 gallons of WVO will be allowed to flow through the centrifuge to ensure steady state conditions and then a sample will be taken. Samples will be taken for each of the

different RPM speed and flow rates. When performing a test we will hold one variable constant and vary the other. i.e. When testing RPM speed the flow rate will remain constant. When the two samples of the test have been collected they will be brought to the lab where they will be tested for titration number and their contents of water and sediment. These results will be compared and recorded. Once all data has been collected a report will be written.

Work Breakdown Structure

Necessary supplies collected and installed

Waste Vegetable Oil

Sample jars

Flow rate meter

Samples Collected

Samples taken while RPM speed is varied

Samples taken while flow rate is varied

Samples labeled and brought to lab

Testing done on control samples and interest samples

Titration number tested

Water and Sediment test performed

Report Written

Data collected and recorded

Data analyzed and conclusions formed

Phase Analysis

The centrifuge phase of the biodiesel project is nearing completion. After the acquisition of more data and testing on that data the suggested RMP speed and flow rate will be able to be verified. Once the correct values for these variables are known the centrifuge will be able to be set to the proper speed and flow rate and run to produce very pure waste vegetable oil.

The phase of centrifuging is nearing its completion but this phase fits into a larger framework of the overall project. A final report is due in the next year from the biodiesel group to the United States Department of energy concerning the research done on small scale biodiesel processing. The aspect of waste vegetable oil purification by means of a centrifuge will be included in this report.

Further work on this project would include finding a way to automate the process so that centrifuging would shut down after a certain amount of oil had been centrifuged.

Schedule

The schedule for the project can be seen in the gphant chart in the appendix of this report. Through December the project was right on schedule to be completed. Equipment had been installed and procedure and tests had been determined. The actual acquisition of samples and testing fell slightly behind schedule during the spring semester due to less class periods and difficulties with oil flowing because of cold weather. The group was still able to complete what it had hoped by the writing of this report.

Resource Analysis

In the future it would be helpful to find some human resources who have experimented with waste vegetable oil centrifuging. A more accurate flow meter would be useful in being able to more adequately specify the flow rate. Since the project has for the most part been completed most of our resources have been acquired and therefore a limited number of resources are needed. It is the biodiesel groups hope that the documentation of this project will provide a base of knowledge for any who want to continue the project in the future.

Budget

After the initial cost of starting up the project the biodiesel centrifuge has very few weekly cost. The power to run the centrifuge is the only daily cost that can be attributed to the project.

Start up costs included: the purchasing of the Raw Power Centrifuge, a racking system, a gravity fed holding tank, and minor plumbing to plumb the system. A cost breakdown of how

the expenses associated with making one gallon of biodiesel can be seen in the appendix and how purifying our waste vegetable oil will decrease these costs.

Future Work

Future work for this project is to design a more automated process for centrifuging so that the centrifuge can be programmed to shut off once the required amount of oil has been centrifuge. Along with automation it is desired that a method of cleaning the centrifuge be developed to ensure impurities do not build up in the centrifuge and lead to less pure oil. Also, testing over a long period of time should be completed to analyze the durability of the centrifuge. The most important future work that can be done on the project is to analyze how much biodiesel quality is enhanced by starting with a more pure oil. It has been determined that a higher quality of fuel will be produced, but the extent of how much the biodiesel will be improved is an area into which more testing could be done.

Conclusion

The purification of waste vegetable oil before the biodiesel reaction is a key aspect of being able to produce high quality biodiesel fuel. The use of a Raw Power centrifuge to purify oil has been found to be a cost effective and reasonably straightforward approach to being able to satisfy this need. The biodiesel group hopes that by providing small scale producers with the proper RPM speeds and flow rates with which to run the Raw Power centrifuge, small scale producers will be able to produce a higher quality fuel with less hassle. The specifications of these two

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variables are; 10 gals/hr for the flow rate into the centrifuge and 3600 RPM for the speed.

Further work can be done on the automation of the centrifuging process and the long term effects of how the Raw Power centrifuge is able to perform, but the project has nearly been completed.

This newly acquired information about how to purify waste vegetable oil will aid the biodiesel group in being able to produce a high grade biodiesel fuel and explain how to replicate it to any who might be interested, including the United States Department of Energy, the Energy Group at Messiah College, and small scale producers all over the world.

Appendix

Figure 1.1

Centrifuge Process

Photo: Jordan Beckler



Figure 1.3 Raw Power Centrifuge



Biodiesel Centrifuge Testing Procedure

Sample Collection

Set RPM speed

Set flow rate

Collect sample before oil has passed through centrifuge

Ensure steady state conditions have been reached by letting 10 gallons of oil pass through centrifuge

Collect sample of oil after it has passed through centrifuge

After every 40 gallons clean centrifuge to ensure similar conditions

Titration Test

Pour KOH distilled water solution into burette

Fill beaker with 10 mL of isopropyl alcohol solution and 1 mL of biodiesel sample solution

Put three drops of phenolphthalein (indicator) into alcohol, biodiesel solution

Slowly drop KOH distilled water solution with burette into beaker until solution turns pink color (all free fatty acids have been neutralized by strong base KOH)

Record # of mL needed to neutralize (This is titration number)

Repeat for other sample of biodiesel

Compare percentage difference between control sample and sample that has passed through centrifuge

Centrifuge Test

Fill 100 mL test tubes with control and centrifuge biodiesel samples

Place in centrifuge and let process run

Compare percent water and sediment between control biodiesel samples and sample that has passed through the centrifuge

Figure 1.3



Titration Test

Figure 1.4



Figure 1.5 Lab Centrifuge



Figure 1.6

Test Specimens



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Cost Analysis

| | | | | |
|------------------------|-------------------|-----------------------|--------------------------|-----------------------|
| Methanol: | \$3.10/gal | \$24.75 | | \$0.503/gallon |
| KOH: | \$90/50 lb | 453.6 grams/lb | \$5.51/42 gallons | \$0.138/gallon |
| Ambulance fuel: | | \$4 | \$4.00 | \$0.10/gallon |
| Electric to run pumps: | | \$10 | \$10 | \$0.25/gallon |
| Labor: | 2 hr | (\$8/hr) | \$16 | \$16.00 |

Bold = Areas of saving

Table 1.1 **Results**

| <u>RPM Speed</u> | <u>Flow Rate</u> <u>Gal/min</u> | <u>% Decrease in</u> <u>Sediment</u> | <u>% Decrease in Titration number</u> |
|-------------------------|--|---|--|
| <u>3000</u> | <u>1</u> | <u>40 %</u> | <u>4 %</u> |
| <u>3300</u> | <u>1</u> | <u>20 %</u> | <u>1 %</u> |
| <u>3600</u> | <u>0.5</u> | <u>50 %</u> | <u>15 %</u> |
| <u>3900</u> | <u>0.5</u> | <u>50 %</u> | <u>10 %</u> |
| <u>3600</u> | <u>0.15</u> | <u>90 %</u> | <u>15 %</u> |
| <u>3600</u> | <u>0.5</u> | <u>70 %</u> | <u>15 %</u> |

Messiah College Biodiesel Fuel Generation Project

DE-FG36-08GO88068

Appendix D. – Manuals and Reports

Appendix D.6.

2011 Methanol Recovery Report

Messiah College Biodiesel Fuel Generation Project

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Biodiesel: Methanol Recovery

Final Project Report

Messiah College

5/09/11

Abstract

The methanol recovery project is a part of the biodiesel project, which processes the byproduct of the biodiesel reaction. The byproduct is considered hazardous material due to the methanol content in the glycerin. This makes the choices for uses of the byproduct very limited; either pay for disposal or process it to make it no longer hazardous. Previous students had made methanol recovery systems and started to explore different systems and methods for recovering the methanol alcohol from the glycerin. During my time working on the project I completed research and created a new methanol recovery system under a grant from the United States Department of Energy.

Introduction

The typical biodiesel reaction at Messiah College is approximately 40 gallons of waste vegetable oil is reacted with 10 gallons of meth-oxide which is a combination of methanol alcohol potassium hydroxide. The result of this mixture is 40 gallons of biodiesel and 10 gallons of byproduct. The byproduct is composed from approximately 25% methanol alcohol and the rest being glycerin. The goal of the project is to extract the methanol alcohol from the glycerin so that the methanol alcohol can be used to make more biodiesel and the glycerin can be used to make soap or be used as compost. This extraction is completed by a distillation process. During my time on the project we I researched different systems that are already in use and different variables that affect the time that is required to complete the process and the purity of the methanol alcohol that is recovered from the system. From my research into methanol recovery systems I developed a design and constructed a system from the grant that is very consistent produces high quantity methanol alcohol.

Background

Context

This project is a very important aspect of biodiesel production. Due to the hazardous nature of the byproduct it is very difficult to dispose of it in an environmentally safe way. This is the single largest problem that small scale biodiesel produces run into. Most of them end up diluting it with tons of water and pour it outside. This causes a couple different problems. First, the methanol alcohol that is diluted and could have been used to produce more biodiesel one it had been extracted from the glycerin. Second, any amount of methanol alcohol that comes in contact with a plant or animal is very hazardous and has the potential to make the organism sick or possibly kill it. Another option of disposal is to pay for hazardous chemical removal, this is very expensive and you do not get to use the methanol or glycerin after it has been removed. The final option which is what my research and work has been on is a method of separating the methanol from the glycerin so the glycerin is no longer hazardous and the methanol can then be used again, and the glycerin can then be used or disposed as a non hazardous material. The major drawbacks of this option are that it is a very energy intensive process and can be dangerous if certain precautions are not taken. This means that the most environmentally friendly options are to either pay for removal or to make a methanol recovery system which is as efficient as possible. The recovery system than allow us to reuse every product of the reaction so nothing is wasted. This system is also is the only option for those in areas where hazardous material removal is not available. Due to possible future projects in Africa we must consider the material which they have available and the resources they have at their disposal. In Africa it is difficult to obtain methanol so being able to recovery what they can from their byproduct is very helpful,

they would also be able to use the glycerin to fertilize their crops. There are also no chemical disposal companies which would be able to handle the byproduct.

Technical

My research into this project included internet and article research but most importantly was the information that I gathered from analyzing the past methanol recovery systems. By analyzing them I was able to gather ideas for heating, tank size, and column design. The first part of the system that I researched was the size of the tank, which is where the bulk of the glycerin is held and heated. This part was difficult to research because not many producers of biodiesel do this process. This was hard to decide on, on one side the larger the pot size the higher the purity of the methanol that is recovered. This was gathered from the original system which had a 55 gallon pot and produced very high quality methanol on a very consistent basis. There was also a very small 10 gallon system that followed the larger system. This system heated up to recovery temperature much quicker and produced less pure methanol. From experience of using both of these systems we found that the smaller 10 gallon system did not process the byproduct fast enough to keep up with the rate at which we were producing biodiesel, yet at the same time we did not have the time to wait for the bigger processor to heat up. From this we were able to weigh the option and come to a conclusion on the size of the tank. During my summer internship with the project I met with biochemistry major by the name of Scott Hoeckele and discussed the distillation which we were doing with the project. Scott explained the different types of columns which he has used in his chemistry experience. We looked at the past systems columns and he showed me the types of columns that he uses in the lab for distillations. From this experience we came to the conclusion that the column that we were using is what is known as a reflux column

which is not ideal for distillation, we then went over the design of simple distillation columns which helped to increase the purity of the recovered methanol and decrease the heating time of the system. The next major aspect that I researched was the circulation which would also fill and drain the system. In the past systems they required a circulation system so that the glycerin did not sit on the heater and burn due to the thickness of the glycerin; this also ensured that the heating was uniform. In past systems to fill it with byproduct you had to remove a plug or some kind of stopper to pour in the glycerin, with this new system I decided that it would be nice if there was a way to pump it in so I looked into different plumbing configurations to accomplish this, and arrived at one that would be able to accomplish this, while at the same time being able to easily empty the system. The vapor condenser was the next aspect that I looked at; energy consumption is very high with the old setup. This setup consisted of an air conditioner used to cool water, and then the water was then pumped through part of the column to condense the vapor back down into liquid. To eliminate this system I researched different condensers and decided that a simple radiator with a fan should be able to do the job of condensing the vapor to liquid.

Narrative

The first step that was accomplished in the construction of a new methanol recovery system was to find a suitable tank as a base. The research helped me to decide on a stainless steel tank that could hold and process approximately 28 gallons of by product at a time. This container was purchased from a golf course, this tank was previously used to grow a bacteria used on the golf courses. Some of the key features of this tank were that it had 1 inch of foam insulation built into the sides of the tank; this allowed for more efficient heating by reducing the amount of heat

lost into the atmosphere. Along with more efficient insulation this tank also has a 1500 watt heater built into the tank. In tank heaters like this we found to be much more efficient than inline heaters which were used in previous systems. After finishing picking out a tank to hold the bulk of the glycerin a circulation system had to be developed to make sure that the glycerin did not sit around the heater and burn, this system was taken from past systems and incorporated some new components. First, we reused a "Clear Water 1inch" pump from a past system which was able to pump the very viscous glycerin at all temperatures. This circulation system also incorporated a fill and drain system which would allow for the user to fill the system without having to pour the byproduct, this will reduce the amount of spill in the future. The second part of this, being able to easily empty the system while the system is still warm was very important, because after the methanol has been recovered from the glycerin it becomes very thick at room temperature, it flows much easier at higher temperatures. The next step of the research was to decide on a column design. From researching this with Scott over the summer I was able to make a simple distillation column for the new methanol recovery system. Due to the very high energy consumption with any distillation system I decided to make the condenser as simple and low energy as possible. To accomplish the low energy condenser I attached a radiator with a fan to the end of the distillation system; this would ensure that no methanol vapor would exit the system. This replaced a setup that consisted of an air conditioner cooling water, then pumping the water through part of the column to condense the vapor back down into liquid using large amounts of energy. This new system used a slightly modified control panel from the past 10 gallon system. This allowed us to control safety checks and automatic shut offs for the fan, heater, and pump.

The main goal for this system was to be able to process the by product as fast as possible while still maintaining a methanol purity that could be used. This system reduced the previous heating time from 8 hours in the small system to 6 hours. This was due to the built in insulation and large built in heater. The purity of the methanol is very consistent the system produces 90% or greater pure methanol except for the last 4 liters which stay around 85% pure. This tells us when the process is almost finished, and all of the methanol can be used directly or blended with new methanol to produce more biodiesel. The system is drastically different from previous systems from the tank, to the condenser, and to the processing time. This system is the next stepping stone in the timeline of methanol recovery. Although the system is quite a bit faster we are not able to completely run a batch during the allotted project time which requires outside time to finish.

Project Plan

Phase Analysis

When I began to work on the project it was in a research stage. A small scale processor had been developed using a 10 gallon tank and a reflux column. This system provided research into the difference between the reflux and distillation columns. With having this small system it also created problems keeping up with the amount of byproduct that we were producing from the biodiesel processor. This system was very difficult to use due to its size and the uneven heating which was caused by the location of the heaters. The small system showed a need of a bigger more consistent methanol recovery system.

From this need I began to do research into methanol recovery systems. I was able to use the smaller methanol recovery system as a guide and pick which aspect worked well and which

ones needed to be changed. This research included looking into column design, circulation systems, heaters, and condensers. The bulk of this research was done by looking at the past system, with some research being completed online.

Following the research I began the design and construction phase. This included selecting components for the system, figuring out plumbing configurations, and calculating dimensions.

Once the design had been approved by Mike Zummo the grant advisor we ordered the parts.

Once the parts had arrived we plumbed the system, which was completed over the course of a month. After it was assembled we began to use the system.

The project is currently in a maintenance phase with the option for more research. The current prototype is able to consistently recovery the methanol alcohol out of the glycerin byproduct of the biodiesel. It is able to process the byproduct in a rate which keeps up with biodiesel production so that there is no back up of byproduct.

Schedule

Attached is a Gantt chart which was the guideline that governed the last few months of the project. Through the process of transitioning from stage to stage I found that I had allotted more time than necessary for each part of the prototype research, design, and construction. The only part where more time would have been nice to have would have been being able to process more of the byproduct than I was able to. With the timing of the last semester I was able to run the system 8 complete times which provided me with some data to analyze purities and temperatures; but having more data would have been nice.

Resource Analysis

This project is currently funded by a grant from the department of Energy which runs out this coming December which will make funding and purchasing different than it is now. With possible future research into this project concerning a vacuum system it will require additional funds. The total cost of adding this vacuum system is unknown due to the uncertainty of the design of the vacuum system and the other modifications which may be necessary to the system. My estimates for adding this vacuum system to the methanol recovery prototype would be in the \$250 range. All other cost would be related to the maintenance of the system which is currently unknown to me at this point in time due to the amount of completed testing.

Budget

The budget is managed by Michael Zummo, attached in the appendix is a cost break down of the new methanol recovery system and a cost analysis for running the system.

Future Work

Future work for this project includes research into the contaminate of the recovered methanol. Using information about the kind of contamination in the system we will be able to find the source of the contaminate and modify the system or clean part of the system to reduce the impurities in the recovered methanol.

Research into the benefits of a vacuum system on the prototype methanol recovery system is also needed. In limited lab scale testing with vacuum on this system we were able to reduce the amount of time that was required to heat the glycerin to recovery temperature. When the glycerin did reach recovery temperature the methanol vapor was also coming off of the glycerin at a higher rate compared to not having a vacuum. This would be very ideal process to reduce the amount of time that would be required to run the system.

Research needs to be completed into the correlation between the temperature of the glycerin in the processor and the flash point of the glycerin. This research can only be done by running the system more and collecting many samples at different points along the system run time. This research will require many flash point tests and many different sets of data. In the end of the research, when a correlation is determined the uncertainty of whether or not enough methanol has been removed from the glycerin will be easier to answer just by looking at the current temperature rather than having to draw off a sample and then traveling back to the lab to do a flash point test.

Conclusion

System is a good stepping stone from where we were to possible future work. I believe that I have made a large stride in the right direction with the work which I have completed over the past two school years and last summer. The current prototype is reliable and consistent with its results, but still takes a while to completely run which is sometimes inconvenient due to the amount of time that is available to work on it during class. Hopefully with the implementation of a vacuum system we are able to reduce the amount of time required for the system to process the byproduct.

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Appendix D.7.

2012 Biodiesel Production Manual

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| <i>Schematic of Biodiesel Processor</i> | 7 |

General System Information and Overview

The Biodiesel Processor is designed to heat waste vegetable oil, react the oil with methoxide creating biodiesel and glycerin waste, and clean the fuel through a washing and drying process. The system circulates the waste vegetable oil in the mixing tank while heating it in a tube heater. Upon heating to the recommended reaction temperature, the heater is turned off and specific amounts of reactants (which are calculated based upon titration of the waste vegetable oil being reacted) are added to the oil. After the reaction occurs, the products are separated into waste glycerin and biodiesel. The Biodiesel is transferred into the washing tank for cleaning. After cleaning and drying for approximately a day, the finished biofuel is removed from the processor.

System Operating Procedures

Precautions

1. *Gloves and safety glasses should always be worn during all processes of biofuel production.*
2. *The Waste vegetable oil is heated to 60⁰C creating a risk of burns if liquid is touched, use caution around the heated liquids.*
3. *KOH is highly reactive and corrosive. Wear safety goggles and gloves when handling. Add a warning about methanol flammability and also include the MSDS sheets for both methanol and KOH.*

Biodiesel Production

Prior to starting the biodiesel production process, always ensure that all valves are closed.

Biodiesel Reaction

Add filtered waste vegetable oil into the mixing tank.

Total volume should not exceed 42 gallons so as to allow room for the reactants to be added.

Open valves Three and Six, and close valve seven. Include a sketch of picture, or reference one that is somewhere else in this document.

Switch on the heating and mixing (H/M) pump and the heater on the control panel to the left of the processor.

Allow the oil to heat to 60⁰C (140⁰ F). This should take approximately one hour with slight variations due to ambient temperature.

The heater has a safety shutoff and will not heat over 60⁰C.

Turn off heater after 60⁰C is achieved.

Titrate the Waste Vegetable Oil (see Appendix A for titration instructions)

Add approximately 8 gallons of methanol to the methoxide tank. Explain that this step can be performed during the heating cycle.

Slowly add the calculated amount of KOH to the methoxide tank.

DO NOT INHALE VAPORS AS THE KOH IS ADDED

Slowly open valve four, and close when the methoxide tank is empty. Note that the Methoxide should not be mixed until most or all of the KOH has dissolved.

Allow approximately one hour for the oil to circulate.

Shut valve 3 and turn off the H/M pump.

Drain the lines into a gallon container from valve one and valve ten. (close valves after oil slows to a drip) dumping collected oil into the mixing tank.

Allow the biodiesel and glycerin to settle (all valves should be closed and the pump and heater should be off) for a minimum of 4 hours.

Drain the glycerin out of valve two, shutting off when biofuel begins to come out.

Glycerin can be stored in closed containers for later processing in Methanol Recovery system.

Washing

Close Valve six and open valve seven and valve three. I think it would be very valuable to have a picture of the system with arrows showing each step/valve.

Turn on the pump allowing for biofuel to transfer into the washing tank.

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After all the biodiesel is transferred into the washing tank, turn off the pump, close valves three and seven, and open valve six.

Drain the lines into a gallon container from valve one and valve ten. (Close valves after oil slows to a drip) dumping the collected fuel into the washing tank.

Add approximately ten gallons of water to the biofuel in the washing tank.

Bubble the mixture for approximately thirty minutes. Note that it is important on the first wash cycle to monitor the biodiesel to ensure that emulsification does not occur.

Allow the oil to settle for approximately one hour.

Drain the water from valve five until biodiesel begins to come out.

Repeat steps 5-8 times increasing the bubbling time by one or two hours each time until the water comes out clear. (typically three or four washes is sufficient)

Drying

Bubble the mixture for at least 4-8 hours (this step cannot be overdone, so it is safe to continue this as long as possible).

Drain the biofuel out of valve five.

Maintenance and Troubleshooting Procedures

Troubleshooting

Before performing any maintenance be sure all devices are turned off and unplugged.

Warning

This Step may be dangerous.

Always unplug the control panel before performing any electrical maintenance work.

Always wear gloves and safety goggles when cleaning the tanks.

Maintenance Procedures

After every use drain all pipes and lines and close all valves.

Visual inspection for leaks in the pipes should be done during every batch of diesel production.

Figures, Illustrations, Drawings, and Tables

Include anything that is used and/or may be wanted or desired by operators or maintainers. Examples include circuit and piping diagrams, exploded parts diagrams, etc. All drawings should be labeled on their own page with a drawing title.



Figure 1: Picture of Biodiesel Processor

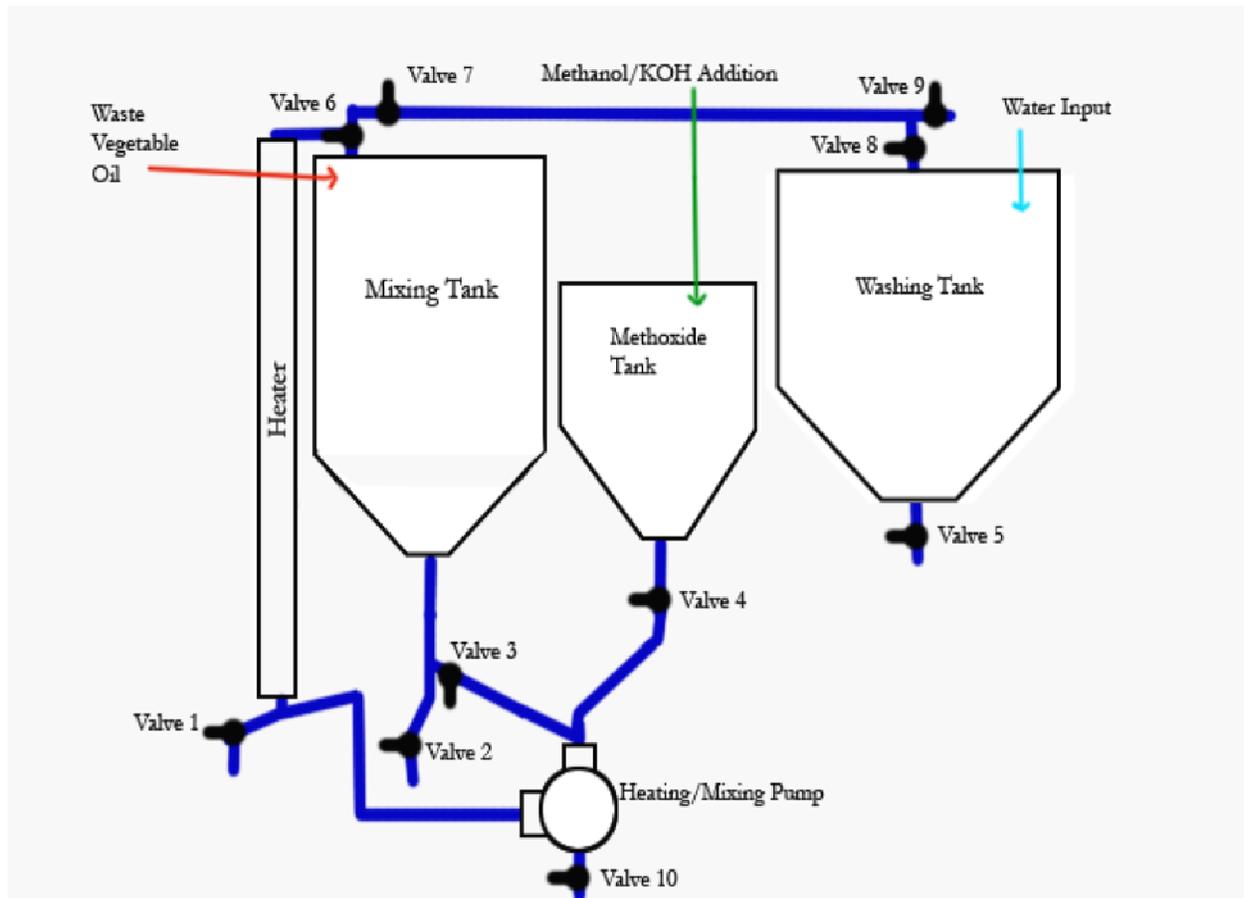


Figure 2: Schematic of Biodiesel Processor

Specifications

Place any specifications that people may want to know. Examples may include size, weight, pressures, temperatures, etc. Include Units.

| <u>Item</u> | <u>Value</u> |
|--|---------------|
| Maximum Waste Vegetable Oil | 42 Gallons |
| Waste Vegetable Reacting Temperature | 60°C (140°F) |
| Recommended Methanol Amount | 8 Gallons |
| Minimum Amount of KOH per Liter of WVO | 8 Grams/Liter |
| Recommended Wash Volume | 10 Gallons |

Appendix A

Titration

Take a sample of waste vegetable oil from the heating and mixing tank.

Completely dissolve 1 g of KOH in one liter of distilled water (This can be used for many titrations). Store it in a hazard labeled bottle in shelving.

Mix 10 mL of isopropyl alcohol in small container with 1 mL of a sample of waste vegetable oil.

Add 3 drops of phenolphthalein.

Using a measuring cylinder, drop 1 mL at a time of the KOH/water solution into the waste vegetable oil and isopropyl alcohol solution

Mix vigorously as each drop is added. If the solution remains pink for 30 seconds after a drop was added, the endpoint has been reached (pH level of 8.5)

Calculate the required KOH input for the biodiesel reaction using the following equation:

$(8 + \# \text{ of mL of solution added}) = \# \text{ of grams of KOH per liter of Waste Vegetable Oil}$

1 Liter = .264 Gallons

1 Gallon = 3.785 Liters

Note: All supplies should be in the yellow flammable cabinet

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Appendix D.8.

2012 Centrifuge Manual

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General System Information and Overview

The Centrifuge

The purpose of the centrifuge is to reduce the impurities in the vegetable oil before the oil goes through the biodiesel processing stage. Reducing the impurities, such as food particles, dirt and water, has many positive impacts that, in the end, provides for better quality biodiesel.

How It Works- It would be helpful to have a drawing or photo here to help explain how it works.

Used vegetable oil enters the inner drum of the centrifuge

The inner drum will be spinning up to 6000 RPMs. The centripetal forces cause the more dense particles (the impurities) to be pushed to the outer edge of the inner drum

There is a lip around the inner drum that causes the outer particles to stay in, but allows the central fluids (the vegetable oil) to spill out of the inner drum (see figure 1.4

The oil that spills out is then drained into a holding tank or fed directly to the biodiesel processor

The Heater

The heater is used to increase the temperature of the vegetable oil before it enters the centrifuge. This decreases the viscosity of the oil, thus allowing the centrifuge to separate the impurities more effectively.

How It Works- A drawing or photo would be helpful here.

Oil flows through the tubes of the heater

As the oil passes through, the high temperature coil heats the oil and lowers the viscosity

Important!

Oil must be flowing through the heater before turning on the heater. This is necessary because if there is no flow, the heater may overheat and damage the device

System Operation Procedure

You need to add the general operations steps. How to add oil to the tank, how to begin flow, how to start the centrifuge, how to stop it, etc.

Cleaning and Maintenance

After every 40 gallons cycle of production, the centrifuge should be cleaned.

Turn off the heater, close all valves, and turn off the centrifuge.

Undo three clasps on the side of the centrifuge lid. Remove lid.

Scrape the inside of the inner bowl walls, cleaning out dirt developed on the sides.

Poke three bottom holes in the bowl to ensure they are not clogged.

Replace lid and attach the three clasps.

There is a tube of waste that leads from the centrifuge to a bucket that needs to be emptied occasionally.

Troubleshooting

If there is no oil coming out of the centrifuge...

Wait a minute, as it takes some time for the centrifuge to fill with oil and then flow out of it.

Ensure that both the handle and the red valve are open.

Check for clogs in the input and output tubes. Use compressed air to shoot through the tubes to try and clear any blockages.

If the oil coming out of the centrifuge is of poor quality...

Decrease the flow rate

Ensure that the centrifuge is running properly.

Clean the centrifuge again.

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Appendix D.9.

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Messiah College Biodiesel Production Group

Methanol Recovery Unit

Operational and Maintenance Procedures

Rev (Original)

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General System Information and Overview

The methanol recovery system is a single stage batch distillation system that includes a single stage centrifugal pump, two heating elements and an forced-air cooled condenser. The pump is used for filling, draining and recirculation of the system. The condenser is for condensing the distilled methanol vapor. The pipe heater and tank heaters are to heat up the glycerin mixture that contains the methanol to be recovered. Various piping components are connected via piping unions to facilitate removal for maintenance.

Methanol Recovery System Electrical Controller

General Information and Overview

The methanol recovery system has four electrical loads that need controlling. These are a tank heater, pipe heater, fan motor, and pump motor. The electrical controller provides a single point of control for these electrical loads. It also provides two temperature indications.

The controller receives 120VAC 60Hz power via a NEMA 5-15P plug connector. This connector supplies power for the internal control circuitry along with the supply power for the fan motor, pump motor, and pipe heater. The tank heater supply power is provided via another NEMA 5-15P connector. The reason for the second power supply is that the sum of power requirements of all the loads is greater than what can be provided via a single 120VAC 15 or 20A circuit. The power outputs to the electrical loads are via four NEMA 5-15R power receptacles.

The in-tank heater connects to the black connector. The pump connects to the yellow connector. The cooling fan connects to the gray connector. The pipe heater connects to the orange connector. The connection of loads to the wrong connection will defeat interlocks and possibly cause improper system operation and damage.

The controller also requires two thermocouple inputs to provide temperature sensing from controlling the cycling of the heating elements on and off on the methanol recovery system which measures the heater on contact temperature. The actual embedded temperature controllers are Direct Automation SOLO-4848RR temperature controllers. Each controller is powered from the same power supply that powers the heating element that it controls. This ensures that if power is available to the heater then temperature control and monitoring is available. The controller can work with type J, K, T, U, and S thermocouples.

The control circuit has two control modes. The transfer mode energizes the pump motor supply to turn on the pump for filling and draining of the methanol recovery system. The distillation mode energizes the pump motor control relay, fan motor control relay, tank heater control circuit, and pipe heater control circuit. The system is designed to be failsafe for shutting down the heating elements to minimize the risk of the heaters remaining energized if the pump is not running or if the appropriate heater on contact temperature is above the desired temperature.

The system is started by verifying the emergency stop is reset, selecting the desired mode of operation, and then pressing the start button. Switching between modes is accomplished by switching the mode selector switch. The system does not need to be stopped and restarted when switching between modes of operation.

Stopping the system is accomplished by pressing the stop pushbutton or in the event of an emergency by pressing the emergency stop button. In either cause it requires the operator to press the start button to restart the system. The loss of supply power will also require the operator to press the start button to restart the system.

System Operating Procedures

Precautions

1. Hot glycerin will cause severe burns.
2. Ensure level is visible within the sight glass whenever recovering glycerin.
3. Opening of the control panel will expose live energized parts.

Initial System Conditions

1. Ensure two separate 15 amp circuits are available before starting.

System Startup

1. Plug the two power supply cord into separate 120V supply circuits.
2. Verify the Emergency Stop Button is reset by turning it clockwise.

System Filling

1. Shut/Check shut valves V-1, V-4.
2. Open/Check open valves V-3, V-2
3. Place system mode switch into TRANSFER.
4. Check that the Emergency Stop Button is released by rotating in the clockwise direction.
5. Momentarily depress the green Start button.
6. If pump is not primed, use a funnel and pour approximately 1 gallon of glycerin into the suction line plastic tubing.
7. Place suction tubing in container into container containing unrecovered glycerin.
8. Continue filling until sightglass level is $\frac{1}{2}$ full in the middle segment.
9. If the need arises to switch containers during filling, shut V-2 and open V-1. Move the suction hose and shut V-1 and reopen V-2.
10. When complete and desiring to immediately start recovering methanol continue on in the next section.
11. Otherwise shut V-2 and V-3 and Push the Stop Button.

Normal System Operation

1. Verify that the pipe heater controller is set for 240F with an hysteresis of 75F.
2. Verify that the tank heater controller is set for 320F.
3. Verify level is visible within the sightglass.
4. Place/Verify that a container is present to collect methanol condensate.
5. Shut/Check shut V-2,V-4.
6. Open/Check open V-1,V-3.
7. Switch the System Mode Switch to DISTILLATION.
8. If the pump is not already running, verify the Emergency Stop button is released by turning it clockwise and momentarily pushing the green Start button.
9. Periodically monitor level in the tank to ensure level remains visible in the sight glass.
10. Periodically monitor level in the methanol condensate storage container.
11. When complete if desired to drain the system, go to the system drain procedure. Otherwise, go to the system shutdown procedure.

System Drain

Warning

Hot glycerin will cause severe burns.

1. Switch the System Mode Switch to TRANSFER.

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2. Place a suitable collection container under the outlet for V-4.
3. Shut/Check shut V-2
4. Open/Check open V-1
5. Open/Check open V-4.
6. Shut/Check shut V-3
7. If the pump is not already running, momentarily opress the green Start Button.
8. If the need arises to switch containers, shut V-4 and open V-3, switch containers and the open V-4 and shut V-3.
9. Done draining, shutdown the unit per the shutdown procedure.

System Shutdown

1. Push the stop button.
2. Shut/Check Shut V-1,2,3,4.
3. If desired, unplug the unit.

Abnormal System Operations

1. Emergency Stop
 - 1.1 Perform the following steps is quickly as safely possible.
 - 1.2 Press the Large Red Emergency Stop button.
 - 1.3 Shut/Check Shut V-2
 - 1.4 Shut/Check Shut V-4
 - 1.5 Remove power from the unit by either unplugging or opening the supply breaker.
2. Draining the system without using the pump.
 - 2.1 Ensure the system is off by pressing the stop button.
 - 2.2 Place a suitable container at the end of the inlet supply hose.
 - 2.3 Open/Check open V-1.
 - 2.4 Open/Check open V-2. Continue draining until unit is drained.
 - 2.5 Shut V-2,V-1.

Maintenance and Troubleshooting Procedures

Troubleshooting

Warning

*Lethal voltages are present inside the controller. Use extreme caution when troubleshooting the control circuit.

NOTE: This is a basic troubleshooting guide. Advanced troubleshooting should be attempted by knowledgeable personnel only.

NOTE: Verify following before troubleshooting.

*Tank heater is plugged into the BLACK connector.

*Pipe Heater is plugged into the ORANGE connector.

*Pump is plugged into the YELLOW connector.

*Fan is plugged into the GRAY connector.

*The emergency stop button is reset by turning it clockwise and it spring returns out.

A) Nothing is turning on including temperature controllers.

- 1) Is the controller plugged in?
- 2) Is power available to the outlets?

B) Only one temperature controller is energized.

- 1) Are both power supply cords plugged in?
- 2) Is power available to both supply cords?
- 3) If the above solutions don't work, trouble shooting of the control circuit is required.

C) The tank heater does not turn on.

- 1) If the TOP temperature controller is not energized follow the guide for "B".
- 2) Is the mode switch in DISTILLATION?
- 3) Check the setpoint of the tank thermocouple.
- 4) Check the settings of the TOP temperature controller.
- 5) If the above solutions do not work trouble shoot the control circuit.

D) The pipe heater does not turn on.

- 1) If the BOTTOM temperature controller is not energized follow the guide for "B".
- 2) Is the mode switch in DISTILLATION?
- 3) Check the setpoint of the pipe thermocouple.
- 4) Check the settings of the BOTTOM temperature controller.
- 5) If the above solutions do not work trouble shoot the control circuit.

E) The pump does not turn on.

- 1) If the BOTTOM temp controller is not energized troubleshoot per troubleshooting guide "B".
- 2) Does the pump turn on when the start switch is depressed?
 - a) If the pump only runs with the start switch continuously depressed the internal control circuit needs troubleshooting.
- 3) Is the pump running light on?
 - a) If yes, troubleshoot the pump.
- 4) If the above solutions do not work trouble shoot the control circuit.

F) The fan does not turn on.

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1) If the BOTTOM temp controller is not energized troubleshoot per troubleshooting guide “B”.

2) Does the fan turn on when the mode switch is in DISTILLATION?

3) Is the fan running light on?

a) if yes, troubleshoot the fan.

4) If the above solutions do not work trouble shoot the control circuit.

Maintenance Procedures

1. Pump Replacement.

1.1 Verify system is shutdown and power removed from the unit.

1.2 Unplug the pump from the controller.

1.3 Remove the bolts that hold the pump base to the pump support frame. Be careful because the pump support from may spring

1.4 Shut/Check shut V-1,V-3.

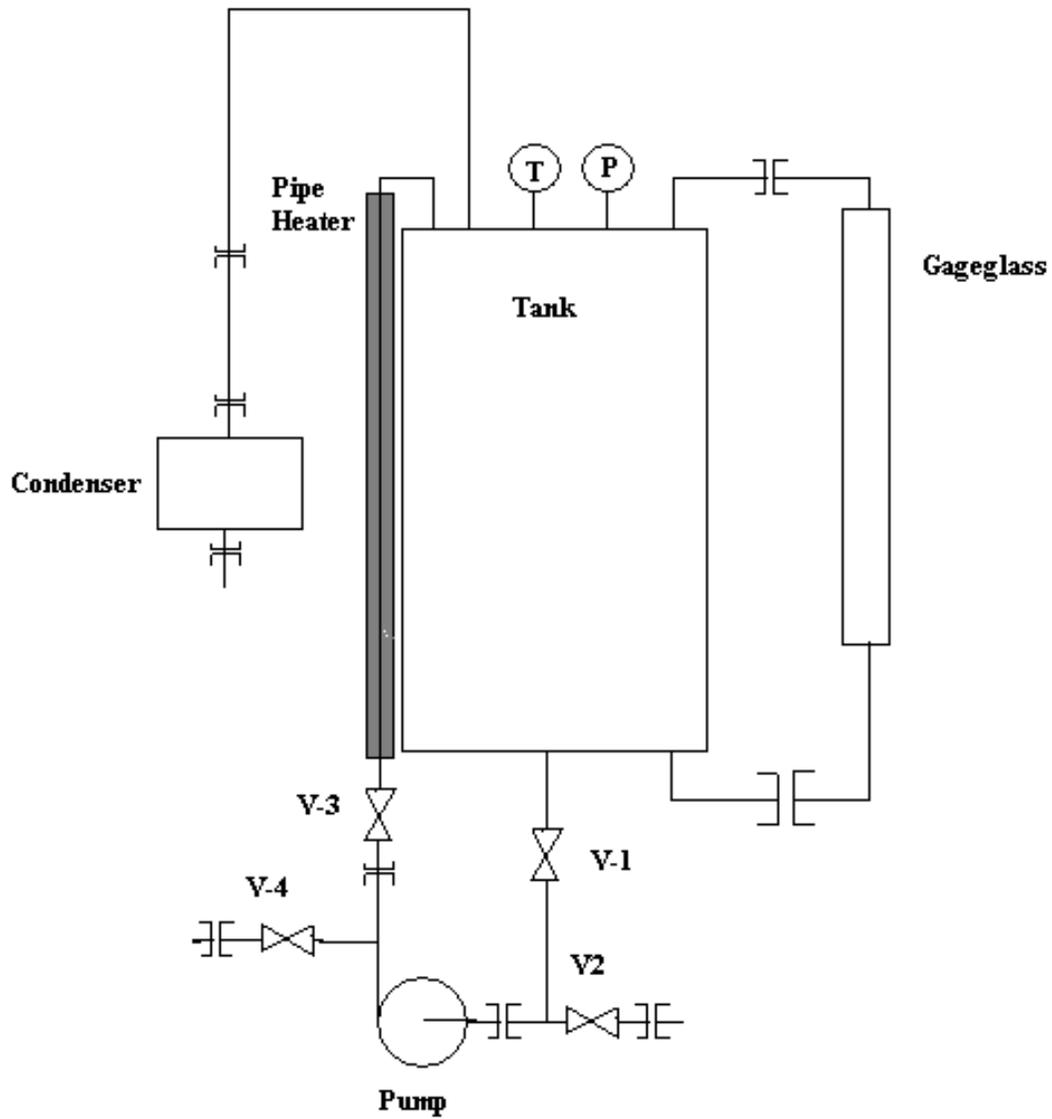
1.5 Open the union at the pump discharge line just before V-3. The pump discharge line will spring from residual bending stresses.

1.6 Open the union at the pump suction.

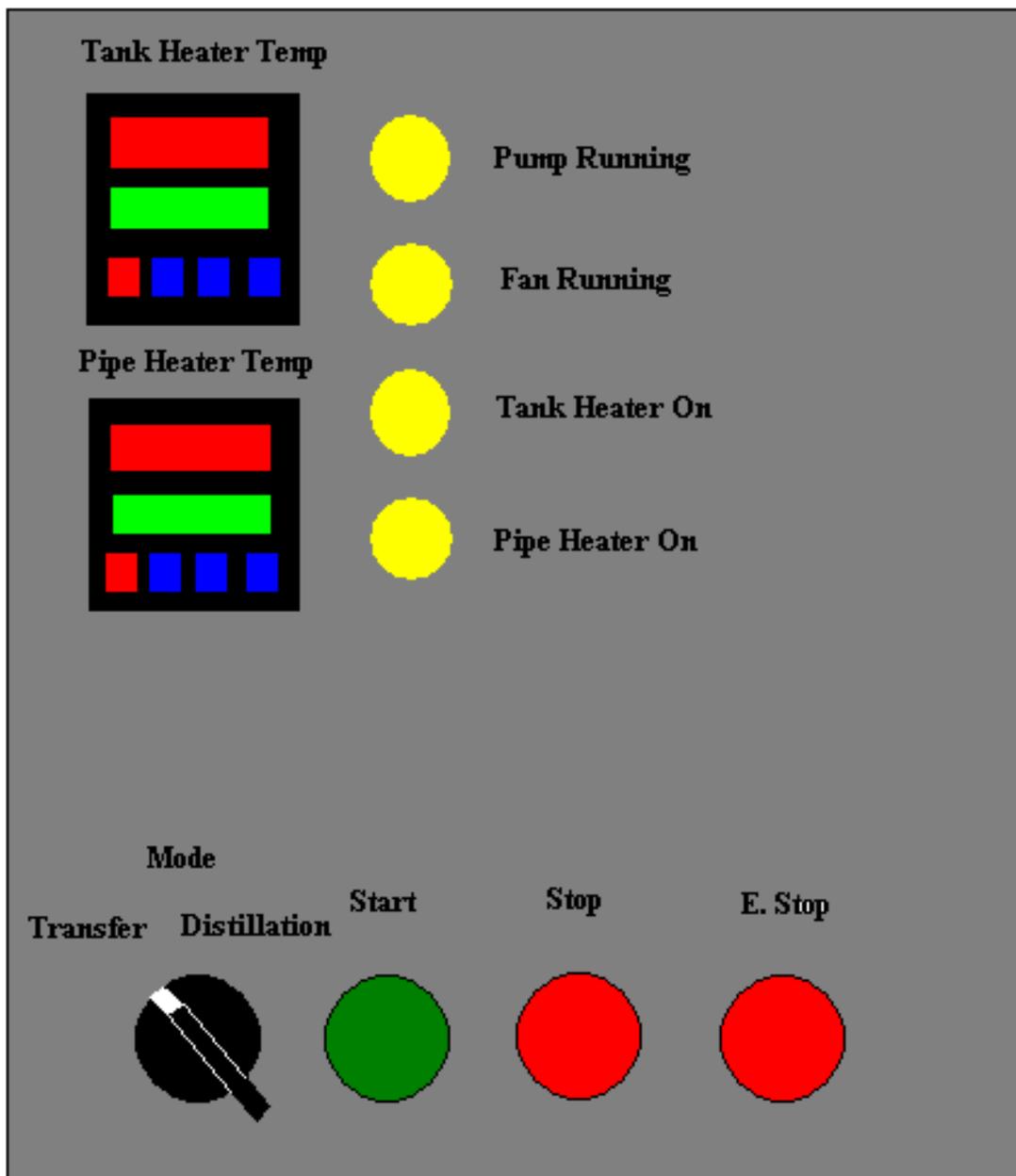
1.7 Installation of the new pump is the reverse of the removal.

Figures, Illustrations, Drawings, and Tables

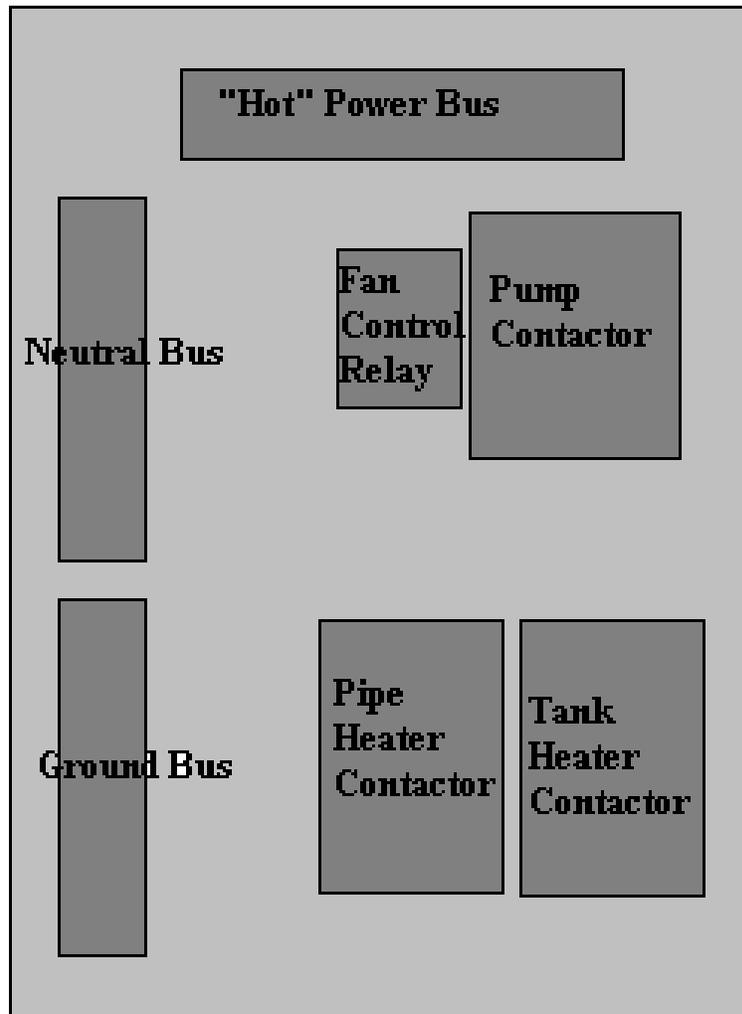
Methanol Recovery System Piping Diagram



Methanol Recovery Control Panel



Internal Controller Component Layout



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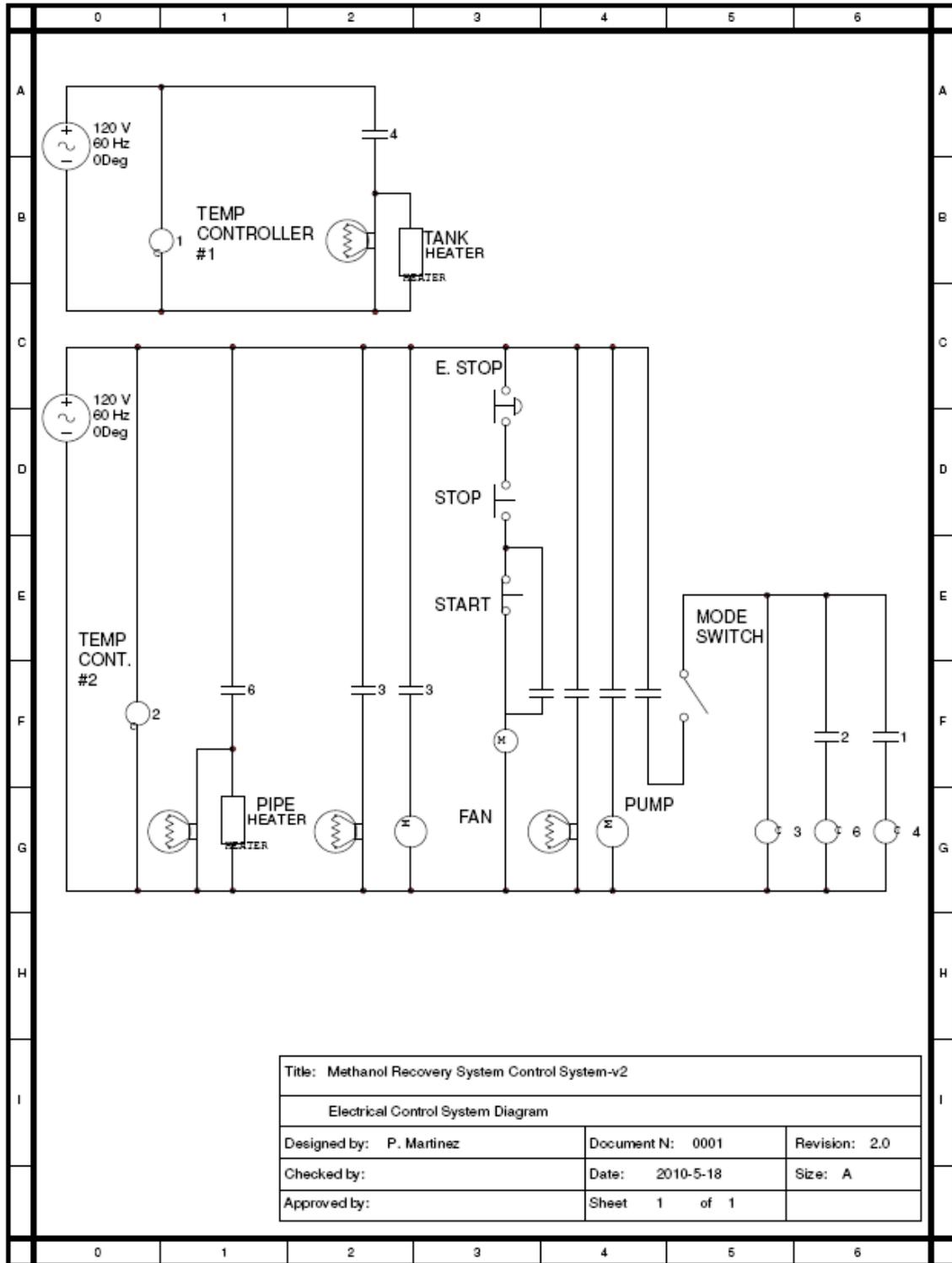
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Specifications

| <u>Item</u> | <u>Value</u> |
|----------------------------------|--------------|
| Pipe Heater Temperature Setpoint | 240 F |
| Pipe Heater Temperature Reset | 165 F |
| Tank Heater Temperature Setpoint | 300 F |
| Tank Heater Temperature Reset | 165 F |
| Pipe Heater Power | 720 W |
| Tank Heater Power | 1500 W |
| Tank Capacity (Total) | 35 Gal. |
| Tank Capacity (Normal Operation) | 27 Gal. |
| Pump Power | 0.5 HP |
| Pump Capacity | 12 GPM |
| Pump Shutoff Head | 50 PSID |

Control Circuit Schematic



Operation Manual: Diesel Engine and Press



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Well done so far Michael! Keep up the good work.

General System Information and Overview

The diesel engine and press are used for mechanical expelling of oil from oil-bearing seeds. The diesel engine provides power to the press via a set of three belts running from the diesel engine to the drive pulley located on the press. Seeds are fed in through a hopper located at the one end of the press and into the screw chamber. The seeds are then augured down the screw chamber with the pressure applied to the seeds increasing as they travel down the chamber. Oil is expelled through tiny slits machined on the face of each of the rings which make up the screw chamber. The press cake is expelled at the end of the press at the end of the screw chamber.

System Operating Procedures

Precautions- It may be helpful to include a photo or drawing here to point out specific areas that are dangerous.

1. Moving parts on the press and engine pose a hazard to personal safety. Keep appendages clear of belts, the flywheel, and pullies.
2. The diesel engine exhaust pipe will get hot during engine operation. Do not touch the exhaust pipe while the engine is running or has recently run. Allow 30 minutes after the engine is shut down before handling the exhaust pipe.

Initial System Conditions

Check Water: Check the water float indicator on the water hopper. Jiggling the stick will help to determine true level water. Fill water hopper with clean water until float indicator is at the top, or until water is visible at the top of the hopper.

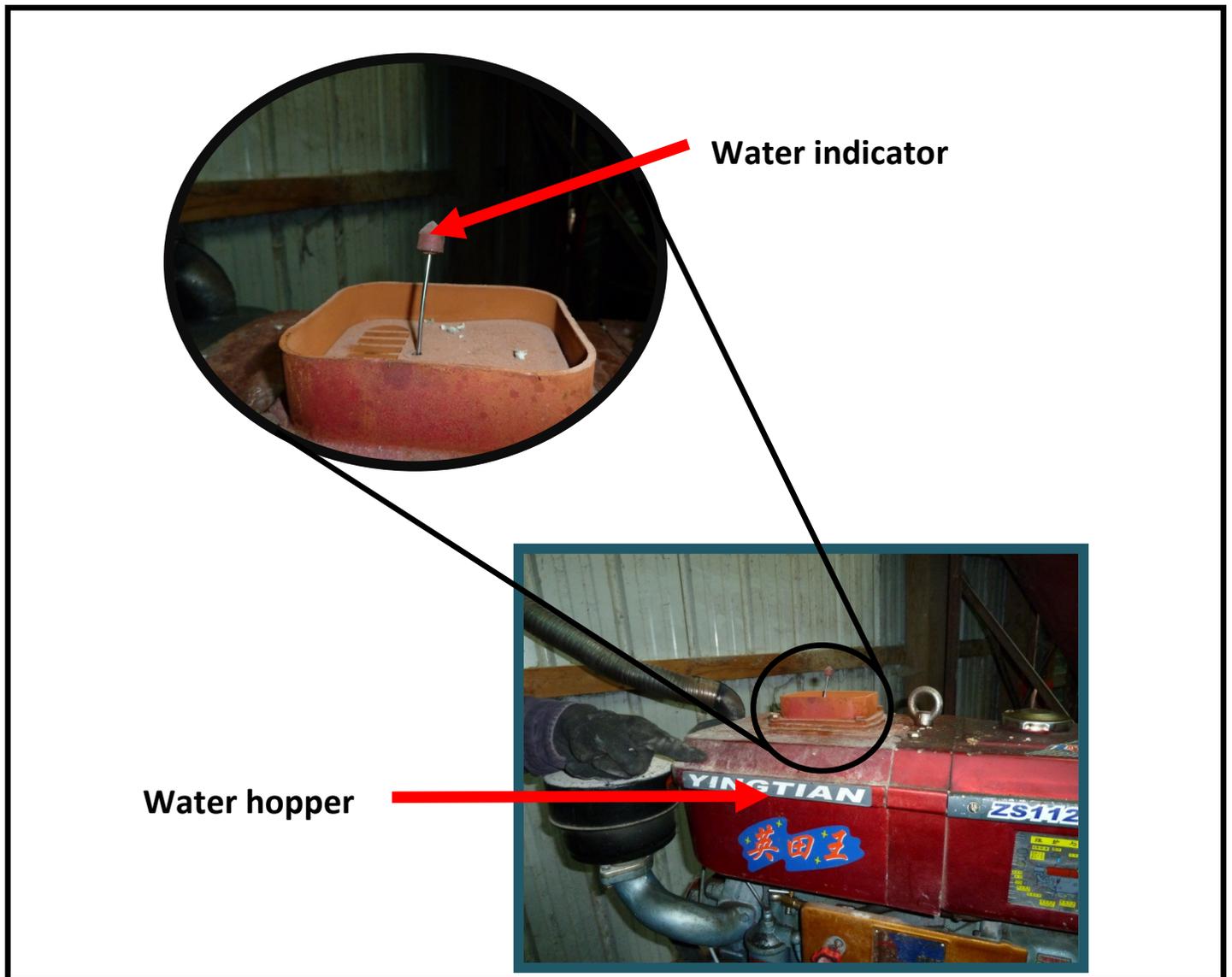


Diagram 1

Check Fuel: Using only clean diesel fuel, fill the fuel tank until it is full. If the ambient temperature is above 45 °F, straight biodiesel or a petro diesel and oil blend can be used as a substitute to petro diesel fuel.



Fuel Tank

Diagram 2

Check Engine Oil: Remove dip stick from engine and check that the oil level is within the operating range. Oil should be up to the full line on the dip stick.



Diagram 3

Check Press Lubricant Oil: With engine NOT RUNNING, remove the lube oil fill cap. The fill cap is located on the top of the gear box next to the drive pulley. The gear box requires 5 liters of 90 weight gear oil. Visually check that the gears are wet with lubricating oil. Replace the cap after filling or checking the gear oil.



Lube Oil Fill Cap



Check Drive Belts: Make sure to thoroughly inspect all drive belts for wear. If any of the drive belt are showing signs of significant wear, replace the worn out belts prior to starting the engine.

Check Mounting Bolts: Check the four bolts below the engine to ensure tightness. If any of the bolts are loose, tighten them with a 3/8" wrench prior to starting the engine.

Check Press Drive Pulley: Check the press drive pulley to ensure that it is tightly adhered to the shaft. If this is not the case, replace the shaft key.

Check Belt Tensioner: Check the belt tensioner to ensure that it is lowered and there is no tension on the belts.

Check Press Screw: Rotate the press drive pulley to ensure that the press screw rotates freely. If the screw does not rotate freely, remove it from the press and clear the debris. (See screw removal instructions under the maintenance and troubleshooting section)

System Startup

Set the red throttle knob to 2/3 open to assure good flow of fuel to the engine.

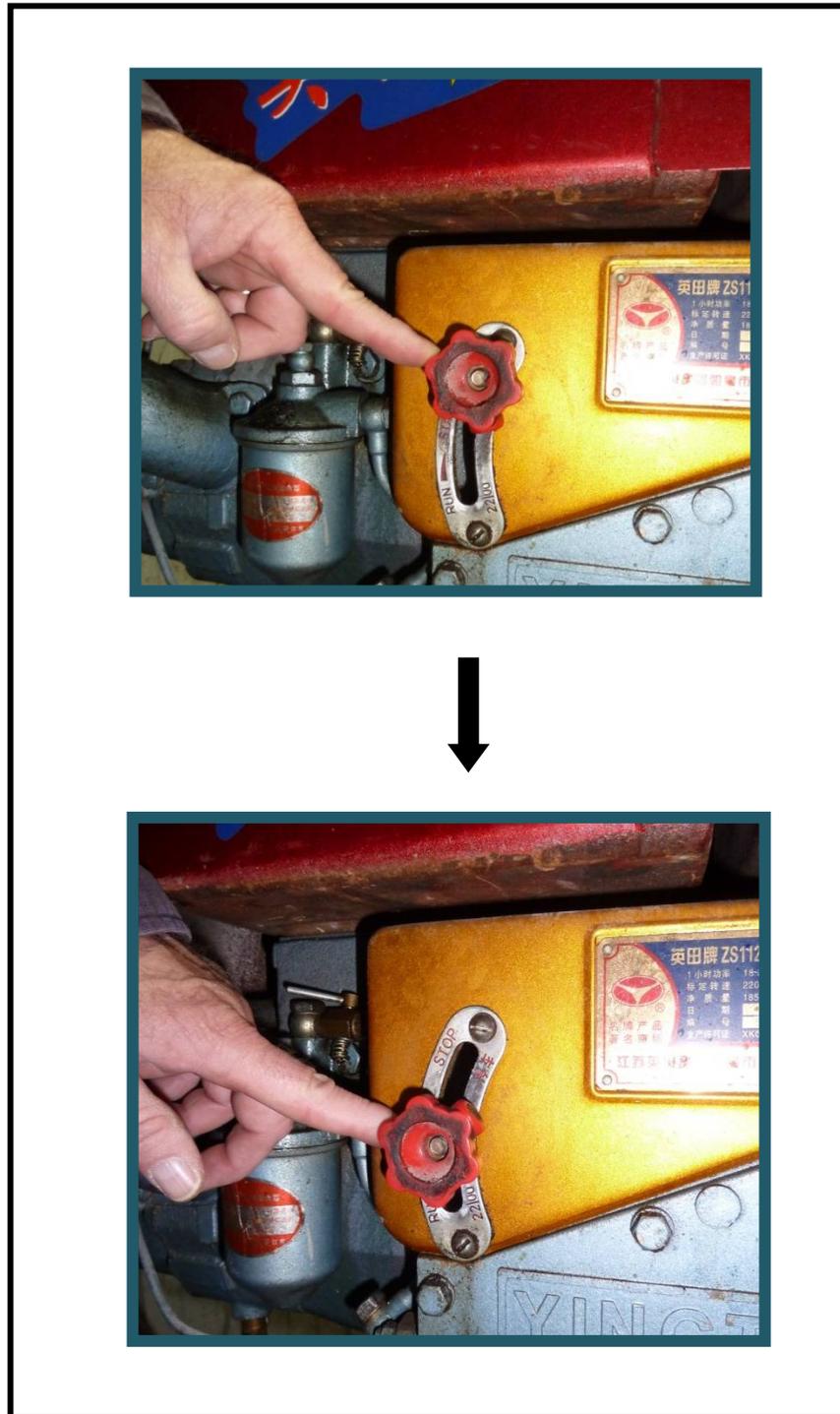
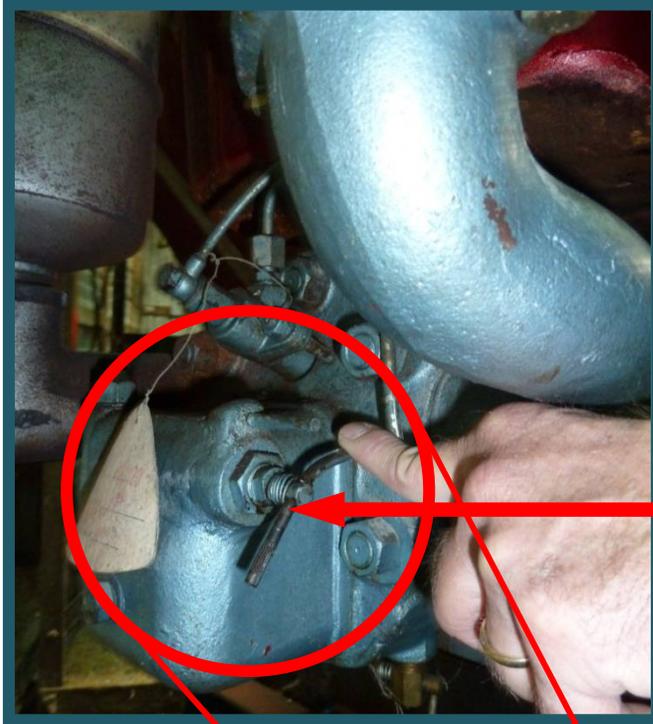


Diagram 5

Move engine decompression lever (close to cylinder) forward to ensure engine spins at lower compression.



Decompression Lever

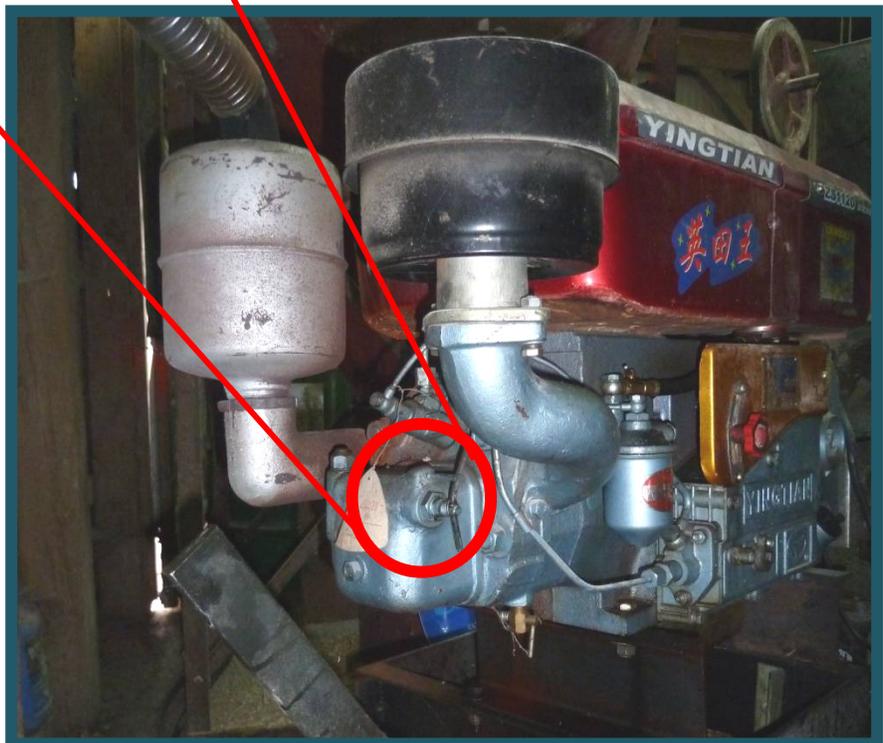


Diagram 6

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Engage starter by using either the engine switch or touching cable to proper battery cable. (If the engine does not contain a starter, see step 5)

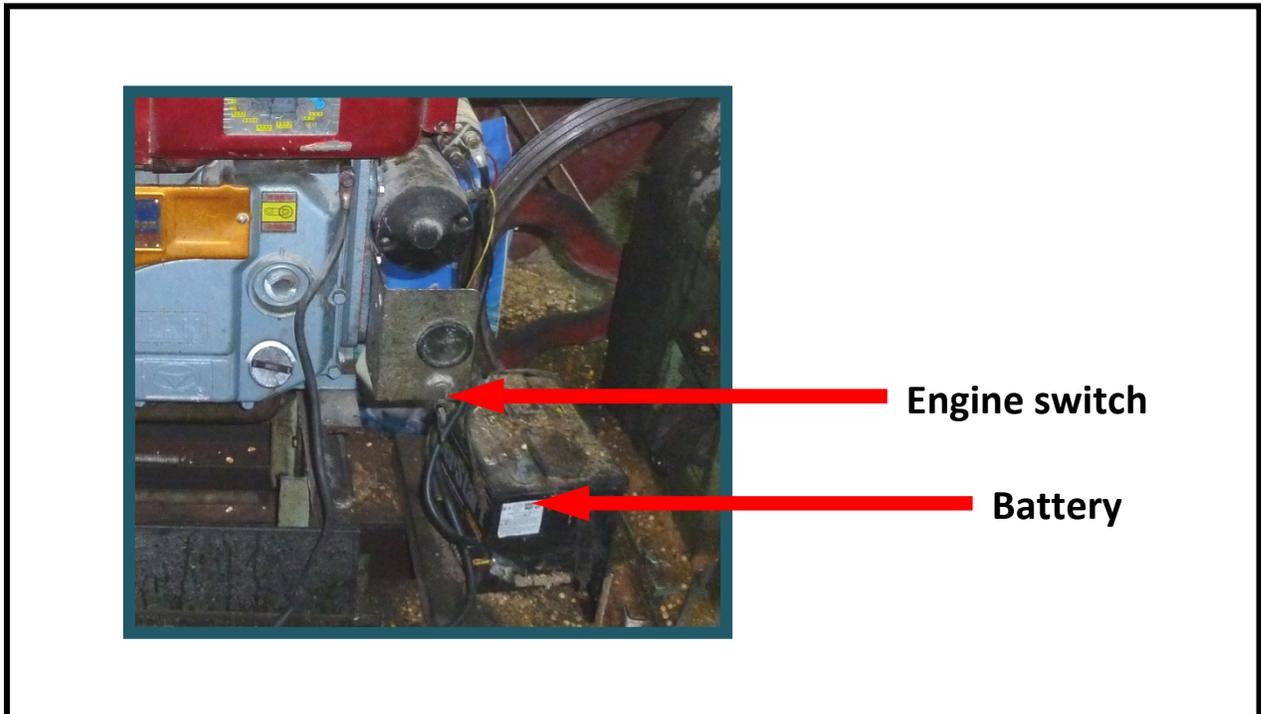


Diagram 7

With the engine spinning, release the decompression lever.

If the engine does not have a starter follow steps 6 – 8, otherwise, proceed to step 9.

Take the hand crank and insert it into the hand crank slot

Push down the decompression lever and spin the hand crank in the clockwise direction.

With the engine spinning, release the decompression lever and remove your hand from the hand crank at the same time

Once the engine is running, slowly raise the belt tensioner until the press is spinning freely.

With the press spinning freely, add the seeds to the feed hopper.

If the belts begin to slip after adding the seeds, raise the tensioner until the slipping stops.

Continue to add seeds in order to keep the hopper full

Normal system operation

System Shutdown

1. Allow the feed hopper to empty and the remaining press cake to exit the press
2. Lower the tensioner pulley in order to completely disengage the engine driving the press.
3. Slowly lower the engine rpm's and shut down the engine by moving the throttle lever up (see diagram 5 for throttle lever location)

Abnormal System Operations

Maintenance and Troubleshooting Procedures

Troubleshooting

If engine does not turn over:

Check battery charge (see diagram 7)

Make sure throttle is open 2/3 (see diagram 5)

Check to make sure drive belts are very loose.

Retry start procedure

If engine still does not start:

Loosen fuel line nut at the cylinder

Depress the decompression lever (see diagram 6)

Engage Starter and spin engine until fuel flows from fuel line. Retighten fuel line and start again.

If no fuel flow

Remove fuel filter and clean

If there is still no fuel flow, check fuel lines for clogs

If screw does not spin

Warning

1. If the engine does not have a starter, hand cranking will be necessary in order to start the engine. Use extreme caution while starting the engine in this manner. There is the slight possibility that the hand crank does not release when the engine begins to run posing a large hazard to the operator. If this occurs, remove hands and fingers from the location of the moving hand crank as quickly as possible and shut down the engine.

Maintenance Procedures

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1. Change engine oil every 250 hours.
2. Change fuel filters every 250 hours.

Figures, Illustrations, Drawings, and Tables

Include anything that is used and/or may be wanted or desired by operators or maintainers. Examples include circuit and piping diagrams, exploded parts diagrams, etc. All drawings should be labeled on their own page with a drawing title.

Specifications

Place any specifications that people may want to know. Examples may include size, weight, pressures, temperatures, etc. Include Units.

Item

Value

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Appendix D.11.

2012 Automated Processor Manual

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MESSIAH COLLEGE BIODIESEL PROJECT

PLC Controlled Processor

Operations and Maintenance Manual

11/30/2011

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1.0 System Overview

1.1 General System Description

1.1.1 The mechanical system is a biodiesel production system that consists of a mixing/reaction/wash tank, a methoxide storage tank, ten normally closed electrical solenoid valves, a transfer/recirculation pump, and an in-tank heater. There are four manually operated ball valves. One is a reaction tank sample/drain valve. The second is a methoxide tank drain/sample valve. The third is a methoxide tank isolation valve that isolates the methoxide tank from the rest of the system. The fourth is manual vent for the reaction tank. The whole unit is mounted on a movable rolling cart for system mobility as required. There are also two manually controlled throttle valves to allow flow rate control for the air and water supplies.

1.1.2 The electrical system includes the controller cabinet that contains the circuitry that allows automatic and manual control of the electrical control components. It also contains a means of shutting off all the outputs regardless of whether they are in manual or automatic control via an emergency stop control. The control panel also includes various status indications lights, pushbuttons, and switches to indicate the status and control the system as required.

The panel contains a mimic schematic to allow a visual representation of the status of the physical system. The output indications are located at the appropriate locations with respect to the mimic schematic with the associated manual override switches located adjacent to the associated indication light so that an operator can visualize what the system is doing during automatic or manual operation.

The automatic control of the system is accomplished by using a programmable logic controller (PLC). It uses various digital and analog 24VDC inputs and outputs to various digitally controlled relay controlled 120VAC outputs. The use of a PLC allows for future logic changes without physically rewiring the control cabinet. Certain exterior inputs that involve enabling the outputs during automatic control uses negative logic so that if the input circuit is opened the outputs are automatically turned off.

1.2 User Controls Overviews (Refer to Figure 4.7)

1.2.1 **Emergency Stop Button** – This trips the Master Control Relay (MCR) which turns off the AC control power that powers all the outputs. Additionally, this also opens an enable switch input to the PLC to place the system in a fault state so that the PLC will automatically shut down the outputs. This ensures that even if the MCR is reset the automatically controlled outputs will not automatically turn on.

1.2.2 **MCR Reset Button** – This resets the MCR to restore AC control power to enable the outputs to be energized.

1.2.3 **PLC Reset Button** – This resets the PLC state to allow automatic operation from when the PLC enters a fault state. The PLC will only reset when the conditions that caused the PLC to enter the fault state are cleared.

1.2.4 **Start Button** – This starts the automatic biodiesel production process from the initial system ready state

1.2.5 **Pause Switch** – This temporarily suspends the process. This differs from the emergency stop in that outputs are still enabled in manual control as well as the current system state is preserved.

1.2.6 **Manual Override Switches** – There are twelve manual override switches on the mimic schematic that control their respective components. They have three positions AUTO, OFF/SHUT, ON/OPEN.

1.3 System Status Light Descriptions (Refer to Figure 4.7)

- 1.3.1 **MCR Tripped** – This indicated that the MCR is tripped.
- 1.3.2 **AC Cont Pwr Avail** – This indicates that AC control power is available to energize the system outputs.
- 1.3.3 **Sys Trouble** – This indicates that the PLC is in a fault state.
- 1.3.4 **Sys Ready** – This indicates that the System is ready to start processing a new batch of biodiesel.
- 1.3.5 **Man Override** – This indicates that either one of the manual override switches are not in AUTO position or that a PLC input/output is being forced to a particular state so that the normal PLC program is not controlling the PLC output.
- 1.3.5 **Man Pause** – This indicates the manual pause switch is pausing the system process.
- 1.3.6 **Sys Fill** – The System is performing an initial system fill.
- 1.3.7 **Heat/Mix/React** – This system is the states where the chemical reactions are happening.
- 1.3.8 **Gly Drain** – The system is draining the glycerin.
- 1.3.9 **Washing** – The System is washing the biodiesel to remove excess reactants.
- 1.3.10 **Drying** – The system is air drying the biodiesel.
- 1.3.11 **Sys Drain** – The system is draining the system.
- 1.3.12 **Output Energized Lights** - There are 12 green lights on the mimic schematic that indicate whether an output is energized.
- 1.3.13 **Htr Pwr Avail** – This indicates that the power supply circuit for the tank heater is energized.

2.0 System Operations

2.1 System Precautions

- 2.1.1 Opening the control panel exposes many live energized parts with lethal voltages.
- 2.1.2 When adding methanol or potassium hydroxide, wear goggles and gloves. These chemicals are hazardous and can cause injuries if skin/eye contact happens.
- 2.1.3 Ensure there is adequate storage volume available in the various containers that receive the discharges from the system. Approximate required volumes are 25 gallons for biodiesel, 40 gallons for wash water, and 12 gallons for glycerin.
- 2.1.4 The glycerin waste has a relatively low flash point of approximately 40C.
- 2.1.5 Do not attempt to override or bypass the tank heater safety switches.
- 2.1.6 Ensure the methoxide tank drain is shut before adding methanol.
- 2.1.7 Verify the manual override switches are in either OFF/SHUT or AUTO before resetting the MCR.

2.2 Initial System Conditions

- 2.2.1 Two separate 15 Amp 120VAC circuits are available.
- 2.2.2 An air source is available between 50 and 100 PSIG.
- 2.2.3 A potable water source is available.
- 2.2.4 High purity methanol is available
- 2.2.5 Chemical grade potassium hydroxide is available.
- 2.2.6 Vegetable oil is available.
- 2.2.7 Storage containers for biodiesel, glycerin, and wash water discharges.

2.3 Initial System Energization

- 2.3.1 Verify/place all the manual override switches are in AUTO.
- 2.3.2 Open/check open the manual tank vent.
- 2.3.3 Verify the manual pause switch is off (10 o'clock position).
- 2.3.4 Plug in the power supply cords into two separate 120V circuits.
- 2.3.5 Verify the HTR PWR AVAIL light is on.
- 2.3.6 Verify the MCR TRIPPED light is on.
- 2.3.7 Verify the SYS TROUBLE light is on.
- 2.3.8 All other indication lights are off.
- 2.3.9 If these indications are not present, perform troubleshooting/repair as required.
- 2.3.10 Reset/verify reset the Emergency Stop pushbutton by rotating it slightly clockwise to release to locking mechanism.
- 2.3.11 Push the MCR RES pushbutton to reset the MCR.
- 2.3.12 Verify the MCR tripped light is off.
- 2.3.13 Verify the AC CONT PWR AVAIL light is on.
- 2.3.14 If the MCR doesn't reset, troubleshoot and repair as required.
- 2.3.15 Push the PLC RES pushbutton.
- 2.3.16 Verify the SYS TROUBLE light is off.
- 2.3.17 Verify the SYS READY light is on.
- 2.3.18 If the PLC doesn't reset, trouble and repair as required.
- 2.3.19 Depress the Emergency Stop Button.
- 2.3.20 Verify that MCR TRIPPED light is on.
- 2.3.21 Verify the AC CONT PWR light is off.
- 2.3.22 Verify the SYS READY light is off.

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2.3.23 Verify the SYS TROUBLE light is on.

2.3.24 If these indications are not present, troubleshoot/repair the MCR control circuit as required.

2.3.25 Reset the Emergency Stop pushbutton.

2.3.26 Reset the MCR.

2.3.27 Reset the PLC.

2.4 Normal System Operations

- 2.4.1 Verify the AC CONT PWR light is on. If not then reset the MCR.
- 2.4.2 Verify the SYS READY light is on. If not then reset the PLC.
- 2.4.3 Verify the MAN OVERRIDE light is off. If not then check the positions of all the manual override switches and ensure they are all in AUTO. If the light is still on, troubleshoot/repair as required.
- 2.4.4 Verify the MAN PAUSE light is off. If not then place the PAUSE switch in the 10 O'clock position.
- 2.4.5 Verify the HTR PWR AVAIL light is on. If not, troubleshoot/repair the circuit as required.
- 2.4.6 Verify there is adequate storage capacity for the system discharges. Approximate discharge volumes are 25 gallons of biodiesel, 12 gallons of glycerin, and 35 gallons of wash water.
- 2.4.7 Shut/Check shut the methoxide tank drain valve.
- 2.4.8 Fill the methoxide tank to the 5 gallon mark on the side of the tank.
- 2.4.9 Depress the START button.
- 2.4.10 Verify the SYS FILL light is on.
- 2.4.11 Verify the SYS READY light is off.
- 2.4.12 Verify the WVO IN valve is on.
- 2.4.13 Verify the RECIRC valve is on.
- 2.4.14 Verify the PUMP is on.
- 2.4.15 Take a small sample of vegetable oil from the reaction take through the drain valve and perform a free fatty acid titration to determine the amount of catalyst to add.
- 2.4.16 Slowly add the required amount of catalyst to the methoxide tank.
- 2.4.17 Continuous monitoring of the system is not required; however, monitor the system periodically to verify proper system operation is recommended.

2.5 Infrequent/abnormal system operation.

2.5.1 Emergency Stopping the system.

2.5.1.1 Depress the Emergency Stop button.

2.5.1.2 When the emergency condition permits recover/drain the system as required.

2.5.2 Temporarily suspending/pausing the process.

2.5.2.1 Place the PAUSE switch in the 2 o'clock position.

2.5.2.2 Verify that the MAN PAUSE light is on.

2.5.2.3 When ready to restore operation, return the MAN PAUSE switch to the 10 o'clock position.

2.5.3 Manually controlling an electrically controlled component.

2.5.3.1 Find the appropriate manual override switch on the mimic schematic.

2.5.3.2 Place the switch to either OFF/SHUT or OPEN/ON.

2.5.3.3 Verify that the MAN OVERRIDE light is on.

2.5.3.4 When manual control of the component is no longer required, place the control switch in AUTO.

2.5.4 Manually making a batch of biodiesel.

2.5.4.1 Place all twelve manual override switches in OFF or SHUT.

2.5.4.2 Verify the MAN OVERRIDE light is on.

2.5.4.3 Open the manual vent valve.

2.5.4.4 Verify the AC CONT PWR AVAIL light is on. If not, then reset the MCR.

2.5.4.5 Fill the system with waste vegetable oil as follows:

2.5.4.5.1 Shut/check shut the heating/mixing tank manual drain valve.

2.5.4.5.2 Place the override switches for RECIRC and WVO IN in OPEN.

2.5.4.5.3 Start the pump by overriding the manual override switch.

2.5.4.5.4 Fill the system until the sight glass is approximately two-thirds full.

2.5.4.5.5 Turn the pump off.

2.5.4.5.6 Shut the RECIRC and WVO IN valves.

2.5.4.6 Heat the system as follows:

2.5.4.6.1 Verify the HTR PWR AVAIL light is on.

2.5.4.6.2 Place the HTR override switch to ON.

2.5.4.6.3 When the temperature reaches 130F as read on the tank thermometer, turn off the heater.

2.5.4.7 Prepare the methoxide as follows (may be done concurrently with step 2.5.4.6):

2.5.4.7.1 Shut/check shut the methoxide drain valve.

2.5.4.7.2 Add approximately 5 gallons of methanol to the methoxide tank.

2.5.4.7.3 Take a sample of the vegetable oil from the heating/mixing tank drain.

2.5.4.7.4 Perform a titration to determine the free fatty acid content.

2.5.4.7.5 Slowly add the appropriate amount potassium hydroxide to the methoxide tank.

2.5.4.7.6 Open/check open the methoxide tank isolation valve.

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2.5.4.8 Add the methoxide to the vegetable oil as follows:

- 2.5.4.8.1 Open the METHOXIDE IN and RECIRC valves.
- 2.5.4.8.2 Turn on the pump.
- 2.5.4.8.3 When the methoxide tank is empty, place the mixing tank DRAIN valve override switch to the OPEN.
- 2.5.4.8.4 Place the METHOXIDE IN manual override switch to SHUT.
- 2.5.4.8.5 After five minutes, turn the pump off.
- 2.5.4.8.6 Shut the RECIRC and DRAIN valves.

2.5.4.9 After approximately two hours, drain the glycerin as follows:

- 2.5.4.9.1 Ensure the glycerin discharge container has approximately 12 gallons of storage space.
- 2.5.4.9.2 Place the mixing tank DRAIN manual override switch to OPEN
- 2.5.4.9.3 Place the GLY OUT manual override switch to OPEN
- 2.5.4.9.4 Start the pump.
- 2.5.4.9.5 When the glycerin discharge changes from dark to light turn off the pump.
- 2.5.4.9.6 Shut the DRAIN and GLY OUT valves.

2.5.4.10 Perform biodiesel washes as follows:

- 2.5.4.10.1 Open the WATER IN valve.
- 2.5.4.10.2 When the tank level is approximately three-fourths full, shut the WATER IN valve.
- 2.5.4.10.3 After approximately four hours open the DRAIN valve.
- 2.5.4.10.4 Open the WATER OUT valve.
- 2.5.4.10.5 Turn the pump on.
- 2.5.4.10.6 When the water is drained, turn the pump off.
- 2.5.4.10.7 Shut the WATER OUT and DRAIN valves.

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- 2.5.4.11 Repeat the washing process two times by performing step 2.5.4.10 again as required.

- 2.5.4.12 Air the biodiesel as follows:
 - 2.5.4.12.1 Open the AIR valve.
 - 2.5.4.12.2 After at least 48 hours, shut the AIR valve.

- 2.5.4.13 Drain the System as follows:
 - 2.5.4.13.1 Open the tank DRAIN valve.
 - 2.5.4.13.2 Open the WATER OUT valve.
 - 2.5.4.13.3 Turn the pump on.
 - 2.5.4.13.4 After approximately 1 gallon is drained or the discharge is a clear yellow, Open the BIODIESEL OUT valve.
 - 2.5.4.13.5 Shut the WATER OUT valve.
 - 2.5.4.13.6 When the system is drained, turn the pump off.

- 2.5.4.14 Shut all the system valves.

2.6 System Shutdown

2.6.1 Verify the system is drained by opening the manual mixing tank drain valve.

2.6.2 Shut/check shut all system valves.

2.6.3 Disconnect the water and air supplies.

2.6.4 Disconnect the electrical power supplies.

3.0 Troubleshooting and Maintenance

3.1 Flushing the Methoxide Supply Lines (Refer to Figures 4.4 and 4.7 as required)

- 3.1.1 Shut/Check Shut the methoxide supply line isolation valve
- 3.1.2 Check shut the methoxide flushing air isolation valve.
- 3.1.3 Connect air to the flushing air quick disconnect fitting.
- 3.1.4 Place a container under the methoxide tank drain valve.
- 3.1.5 Ensure the lid of the methoxide tank is on and is sealed.
- 3.1.6 Open the methoxide drain valve.
- 3.1.7 Open to flushing air isolation valve.
- 3.1.8 When air starts blowing out the drain and/or the methoxide tank vent shut the air flushing air isolation valve.
- 3.1.9 Ensure air is available to the biodiesel drying air supply valve.
- 3.1.10 Shut/check shut the heating/mixing tank manual drain and manual vent valves.

Warning

Automatic tank pressure is disabled during the following steps. Do not let tank pressure exceed 50 psig as read on the tank pressure gauge.

- 3.1.11 Place the manual override switch for the tank vent to SHUT.
- 3.1.12 Place the manual override switch for the tank drain to OPEN.
- 3.1.13 Open the methoxide supply line isolation valve.
- 3.1.14 Place the manual override for the methoxide in solenoid valve to OPEN.

Note

The following steps will cause the control system to enter a fault state due to higher than normal tank pressurization. This is expected.

- 3.1.15 Place the manual override switch for the air in solenoid valve to OPEN.

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- 3.1.16 When air starts to come out of the methoxide tank drain, shut the methoxide tank drain valve.
- 3.1.17 When air starts to enter the methoxide tank place the manual override switch for the methoxide supply valve in SHUT or AUTO.
- 3.1.18 Place the air in override switch in either SHUT or AUTO.

Caution

The following step vents the tank which produces high audible noise level. Hearing protection is recommended.

- 3.1.19 Place the vent override switch in OPEN.
- 3.1.20 When tank pressure is zero as read on the tank pressure gauge, place the tank vent override switch in AUTO.
- 3.1.21 Place the tank drain manual override switch in either SHUT or AUTO.
- 3.1.22 Reset the PLC by momentarily depressing the PLC RES pushbutton.

4.0

Tables and Figures

Figure 4.1

| PLC Controller Outputs | | | | | |
|------------------------|---------------|----------------|---------------|---------------|--------------|
| 1762-OW16 | Function | 1761-24BWA Out | Function | 1761-24BWA In | Function |
| 0 | Heater | 0 | Sys Drain Lt | 0 | Spare |
| 1 | Pump | 1 | Drying Lt | 1 | Spare |
| 2 | Water Out | 2 | Wash Lt | 2 | Spare |
| 3 | Glycerin | 3 | Sys Ready Lt | 3 | Spare |
| 4 | Biodiesel | 4 | Override Lt | 4 | E STOP Sense |
| 5 | Recirculation | 5 | Sys Pause Lt | 5 | MCR Sense |
| 6 | Tank Drain | 6 | Gly Drain Lt | 6 | PLC Reset |
| 7 | Water In | 7 | Sys trouble | 7 | Start |
| 8 | Vent | 8 | Sys Fill Lt | 8 | Pause |
| 9 | Air | 9 | Ht/Mix/Rxn Lt | 9 | Override |
| 10 | Methoxide | 1762-IF4 In | | 10 | Spare |
| 11 | Veg Oil In | 0 | Top Press | 11 | Spare |
| 12 | Spare | 1 | Bottom Press | 12 | Spare |
| 13 | Spare | 2 | Tank Temp | 13 | Spare |
| 14 | Spare | 3 | Spare | | |
| 15 | Spare | | | | |

Figure 4.2

| Internal Controller Wiring Color Code Chart | |
|---|---|
| Wire Color | Function |
| Green | Earth (Safety) Ground |
| White | 120VAC Neutral |
| Red | 120VAC Hot |
| Black | 120VAC Control Power (Isolated by Master Control Relay) |
| Blue | +24VDC |
| Yellow | DC Sensing Circuits Ground |
| Brown | Analog Sensor Line |

Figure
4.3

Internal Control Panel Layout

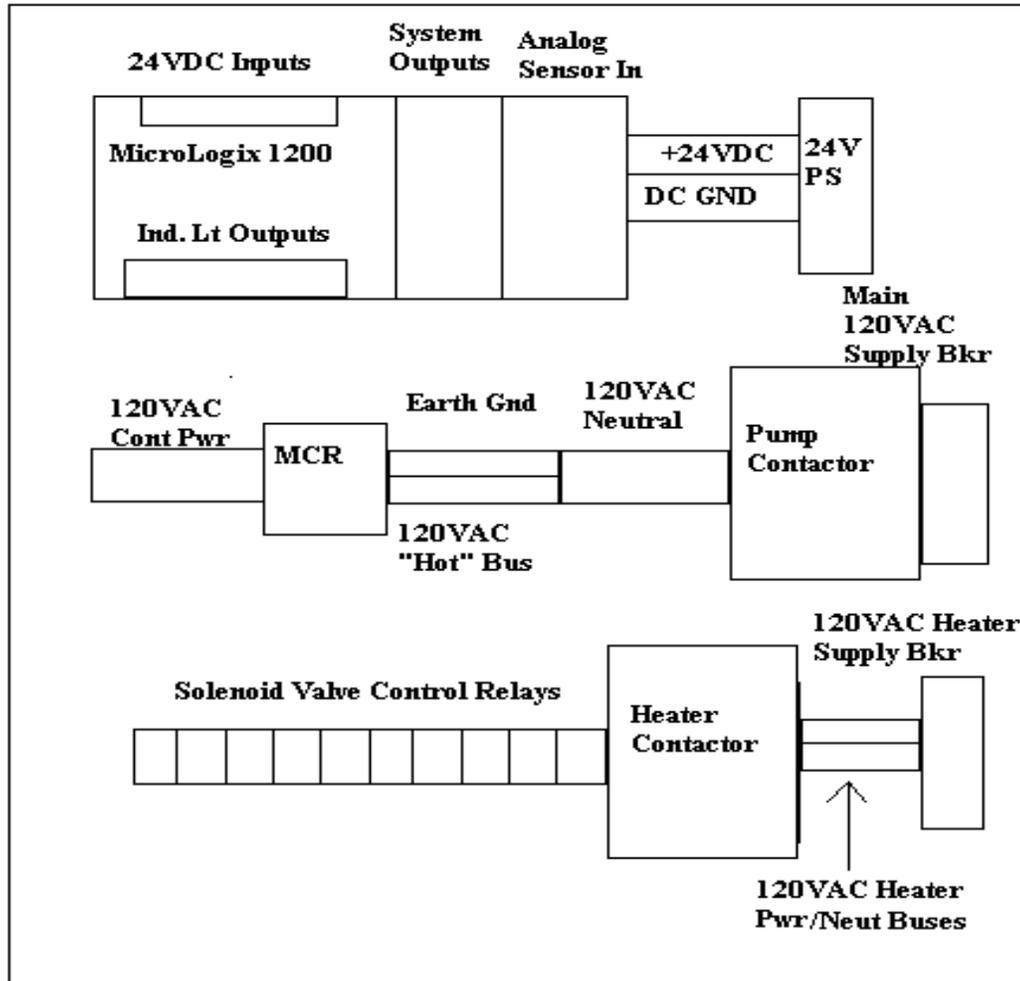


Figure 4.4

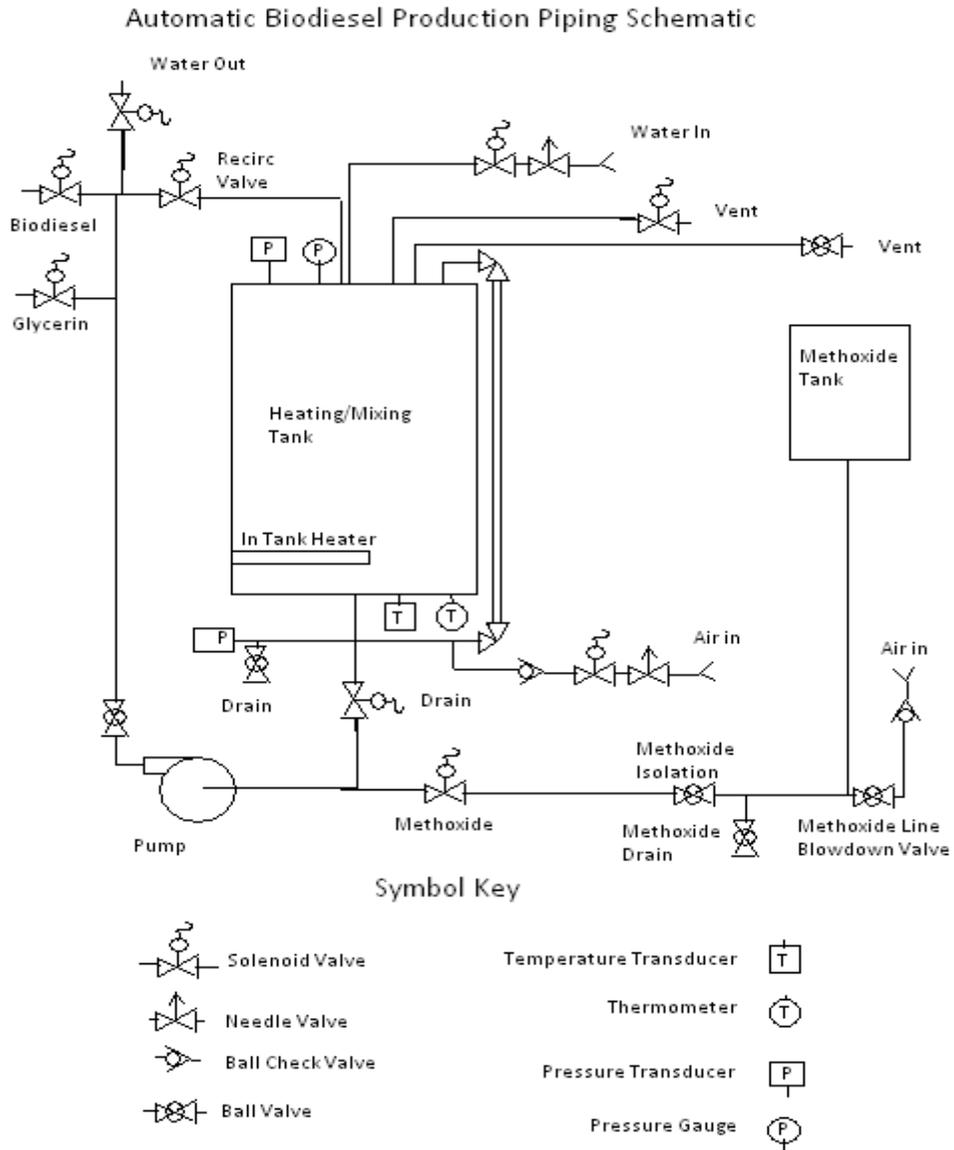


Figure 4.5

| <u>Item</u> | <u>Specifications</u> | <u>Value</u> |
|--|-----------------------|--------------|
| Tank Heater Temperature Setpoint | | 135F |
| Tank Heater Power | | 1500W |
| Tank Capacity (Total) | | 35 Gallons |
| Production Capacity (Normal Operation) | | 27 Gallons |
| Methoxide Tank Capacity(Total) | | 10 Gallons |

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Figure 4.6
Parts List

Distributor: Automation Direct

| Distributor Part # | Manufacturer Part # | Manufacturer | Description | Quantity |
|--------------------|---------------------|-------------------|----------------------------|----------|
| TTD25N-20-0100C-H | | Automation Direct | Temperature Transducer | 1 |
| PTD25-20-0015H | | Automation Direct | Pressure Transducer | 2 |
| GCX1100 | | Automation Direct | Black Pushbutton Switch | 3 |
| GCX1135 | | Automation Direct | E. Stop Push Button | 1 |
| GCX1300 | | Automation Direct | 22mm SPST Switch | 1 |
| SQM08D | | Automation Direct | DIN mount relay socket | 12 |
| QM2N1-A120 | | Automation Direct | DPDT 120V relay | 12 |
| N1C202006LP | | Automation Direct | 20" x 20" x 6" Case | 1 |
| CD12M-0B-0700-A1 | | Automation Direct | Transducer Cable 7m length | 3 |
| GH15DN-3-10A | | Automation Direct | Contactor | 2 |

Distributor: Digikey

| Distributor Part # | Manufacturer Part # | Manufacturer | Description | Quantity |
|--------------------|---------------------|---------------------|-------------------------------|----------|
| CW164-ND | GTS448E101AHR | E-Switch | DPDT Toggle Switch | 12 |
| ADR3575-U16 | TS3575SL | American Electrical | 35mm x 7.5mm x 16" DIN rail | 3 |
| 277-2005-ND | 2315256 | Phoenix Contact | 8 x 2 position DIN mount bus | 2 |
| 277-2007-ND | 277-2007-ND | Phoenix Contact | 16 x 2 position DIN mount bus | 3 |
| 285-1227-ND | DSP-10-24 | TDK-Americas | 24VDC 420mA power supply | 1 |

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Distributor: Mouser

| Distributor Part # | Manufacturer Part # | Manufacturer | Description | Quantity |
|--------------------|---------------------|-------------------|-------------------|----------|
| 607-2152A5 | 2152A5 | Chicago Miniature | 5/16" Green Light | 12 |
| 607-1050A3 | 1050A3 | Chicago Miniature | 1/2" Amber Light | 2 |
| 607-1050A4 | 1050A1 | Chicago Miniature | 1/2" Red Light | 2 |
| 607-1052A5 | 1052A5 | Chicago Miniature | 1/2" Green Light | 8 |

Distributor: Home Depot

| Distributor Part # | Manufacturer Part # | Manufacturer | Description | Quantity |
|--------------------|---------------------|------------------|----------------------------------|----------|
| | | Southwire | SJOOW 12-3 | |
| | | Southwire | Lamp wire 18-2 250' reel | 1 |
| | | Leviton | NEMA 5-15P Plug | 3 |
| | | Leviton | NEMA 5-15P Cord Receptacle | 2 |
| | | Tyco Electronics | Chrimp connectors- various sizes | |
| | | | Primary Wire-various colors | |

Distributor: Allen Bradley

| Distributor Part # | Manufacturer Part # | Manufacturer | Description | Quantity |
|--------------------|---------------------|---------------|------------------------------|----------|
| | 1762-OW16 | Allen-Bradley | 16 Relay output expansion | 1 |
| | 1762-IF4 | Allen-Bradley | (4) 4-20mA analog input mod. | 1 |
| | 1761-24BWA | Allen-Bradley | 14 input/10 Output PLC | 1 |

Figure 4.7

Automated Biodiesel Biodiesel Processor Control Panel Front Layout

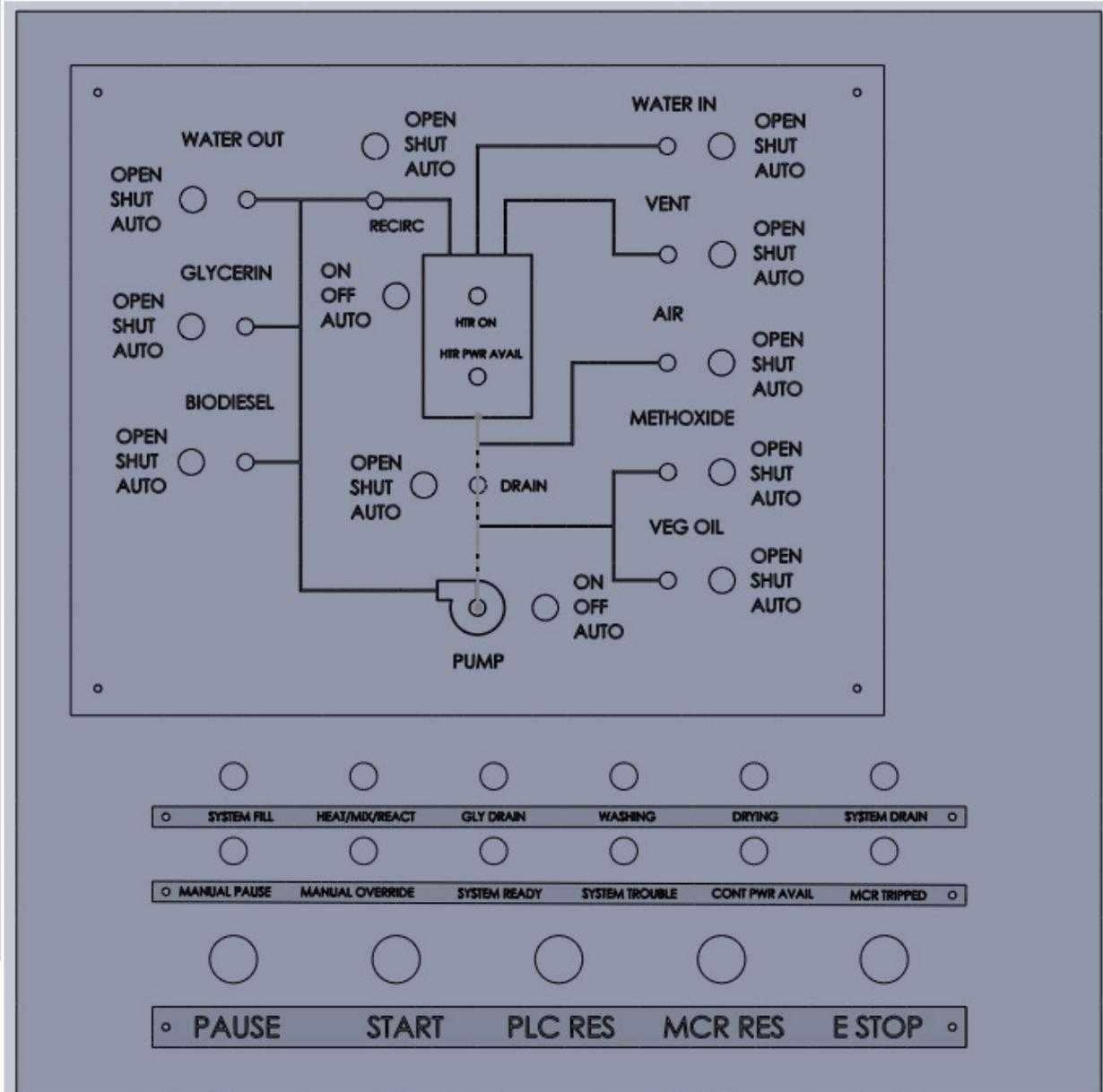


Figure 4.8

Heater Control Circuit

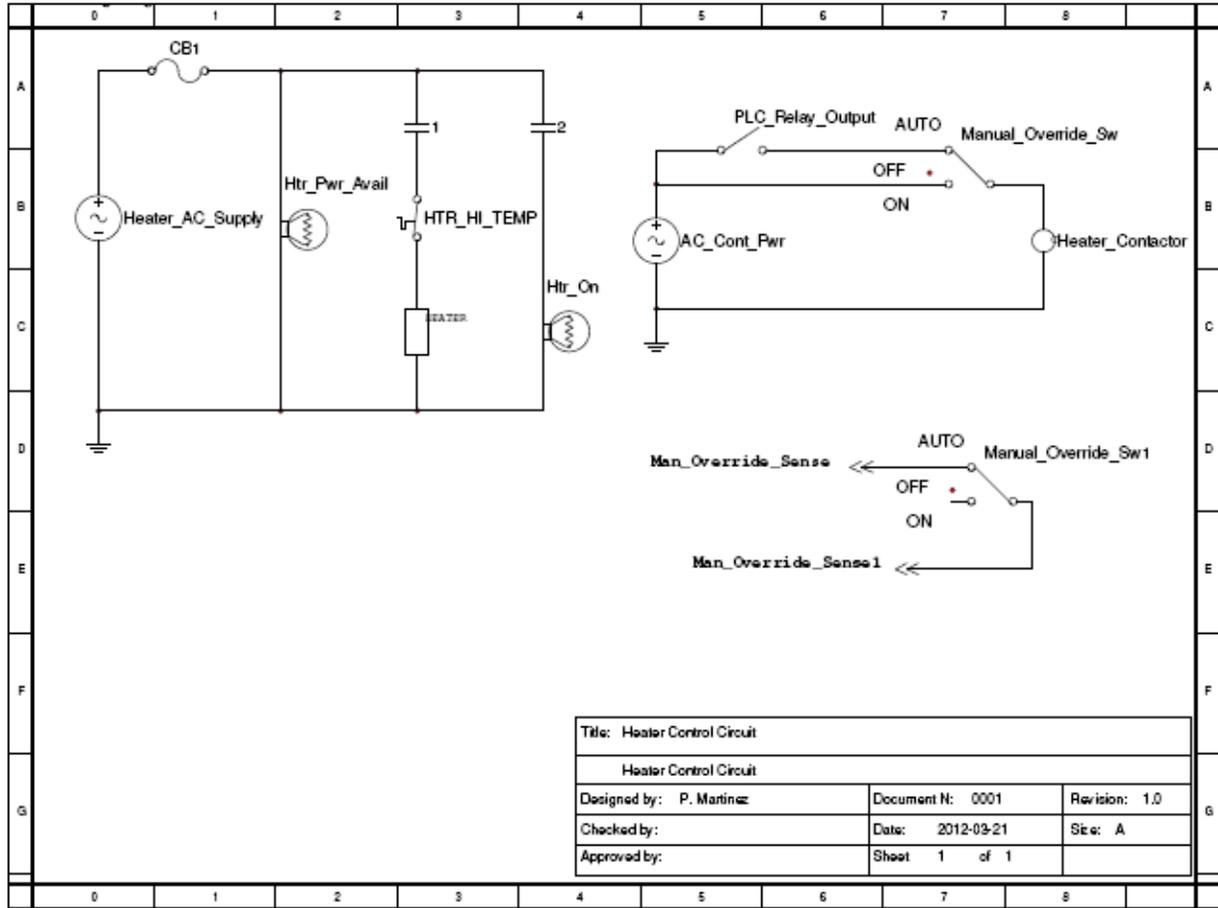
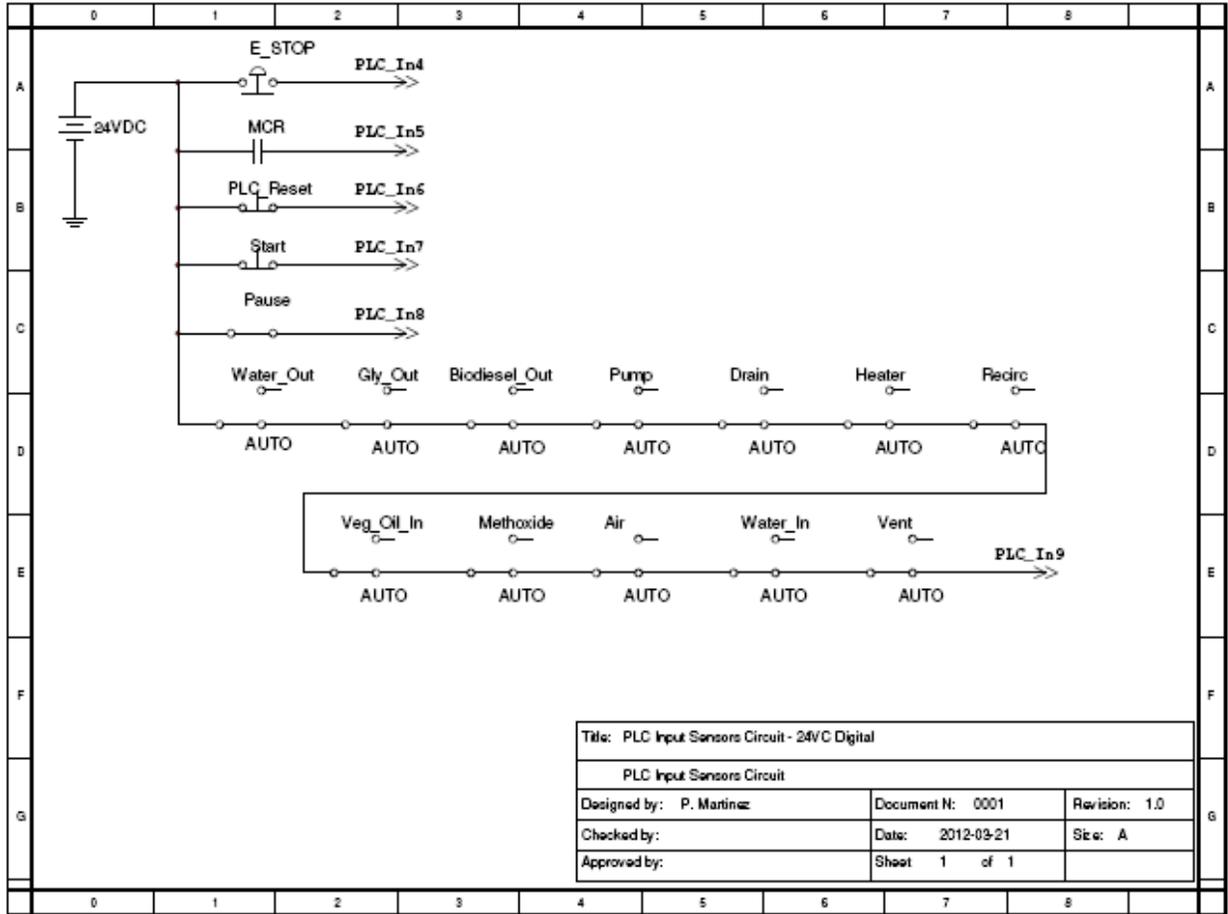


Figure 4.9

PLC Digital Inputs Schematic



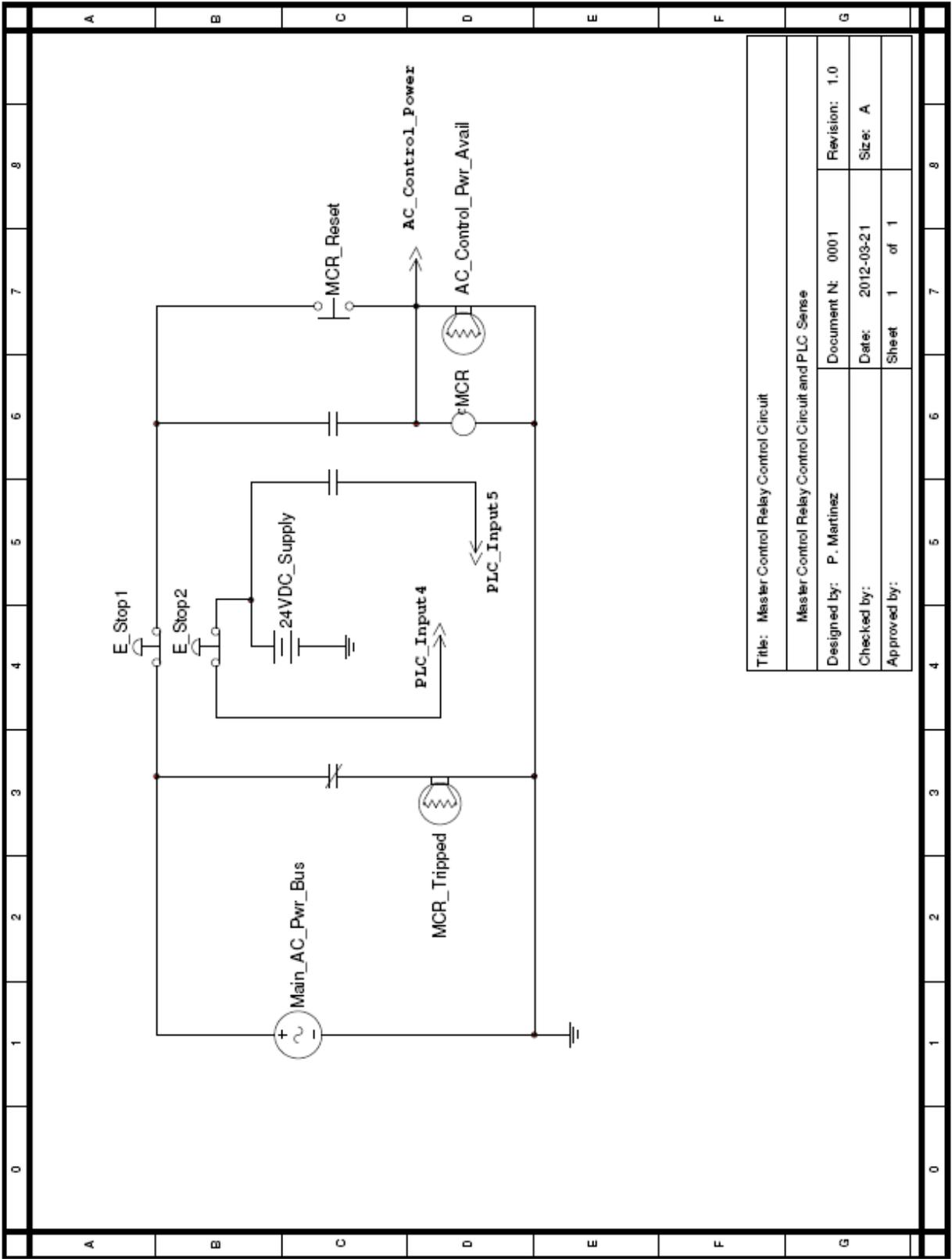
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Figure 4.10 Master Control Relay Circuit
Schematic



Title: Master Control Relay Control Circuit

| | |
|--|------------------|
| Master Control Relay Control Circuit and PLC Sense | |
| Designed by: P. Martinez | Document N: 0001 |
| Checked by: | Date: 2012-03-21 |
| Approved by: | Revision: 1.0 |
| | Size: A |
| | Sheet 1 of 1 |

Figure 4.11

Typical Solenoid Control Circuit

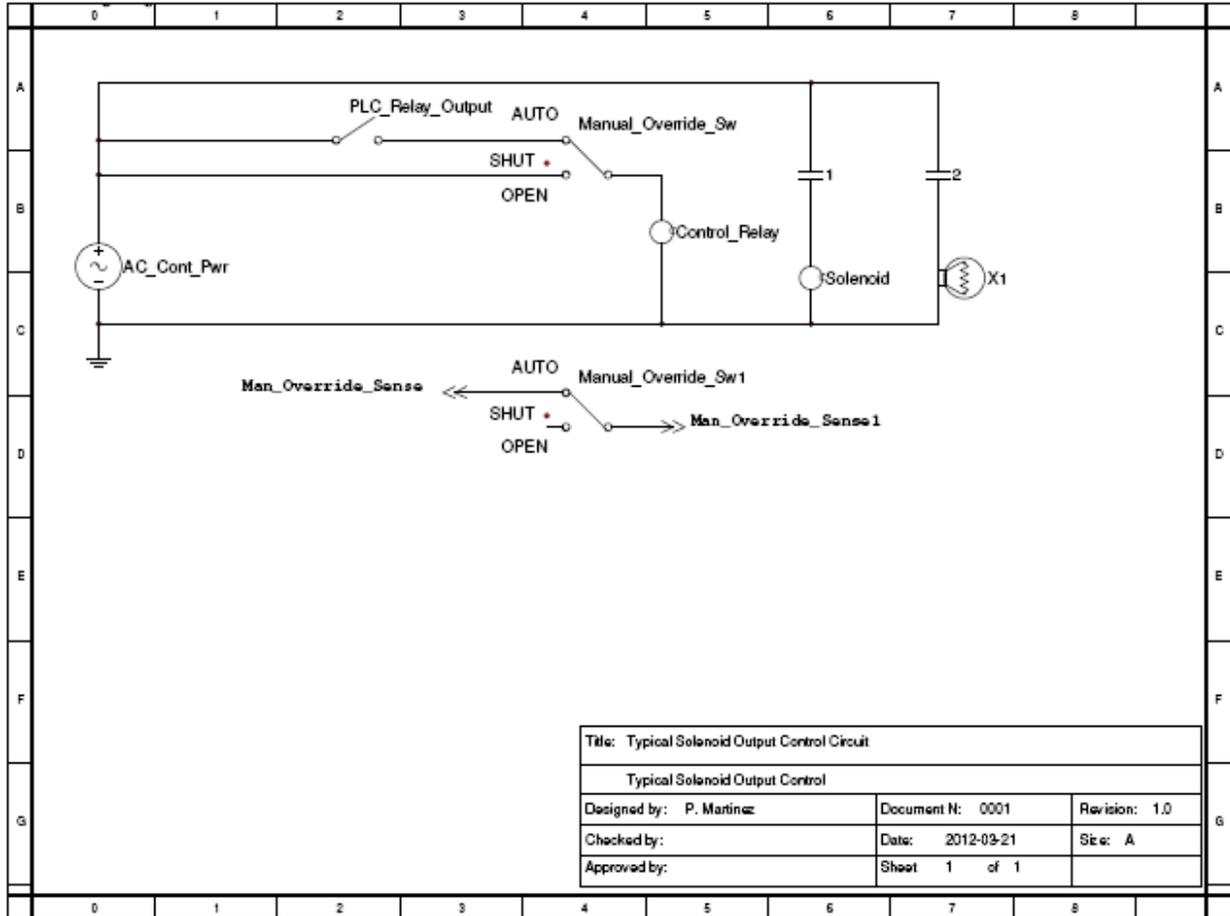
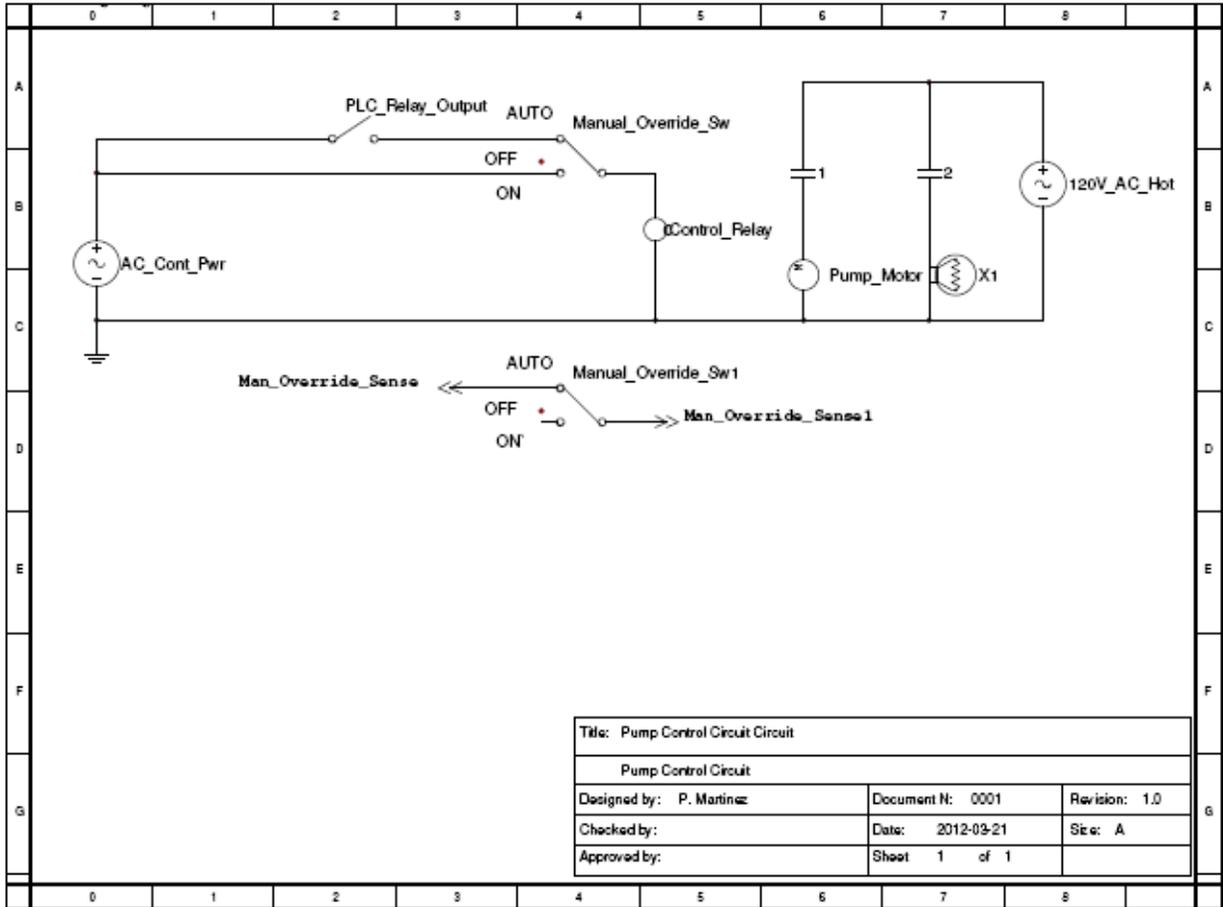


Figure 4.12

Pump Control Circuit



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Appendix D.12.

2010 Biodiesel Quality Testing Final Presentation

Biodiesel Quality Testing

Energy Group:
Trent Zempel
Andy Derr

Scope

GOAL:

Determine the effect of varied catalysts and water washing procedure on overall biodiesel quality

Tests:

1. Acid Number
2. Cloud Point
3. Flash Point
4. Water and Sediment
5. Total and Free Glycerin
6. Sulfated Ash
7. 27/3

Timeline

- August-December 2009 – Acquire necessary testing equipment and familiarize ourselves with biodiesel testing
- January 2010 – Intensive testing of all biodiesel samples
- February-March 2010 – Analysis of information and results
- April-May 2010 – Presentation/Dissemination of results



Samples

| KOH | Unwashed | Single Wash | Fully Washed |
|-------------------------|-----------------|--------------------|---------------------|
| Minimal Catalyst | | | |
| Precise Catalyst | | | |
| Excess Catalyst | | | |

| NaOH | Unwashed | Single Wash | Fully Washed |
|-------------------------|-----------------|--------------------|---------------------|
| Minimal Catalyst | | | |
| Precise Catalyst | | | |
| Excess Catalyst | | | |

Flash Point

Procedure:

ASTM D93-02a

Equipment:

Pensky-Martin Closed Cup Flashpoint Tester

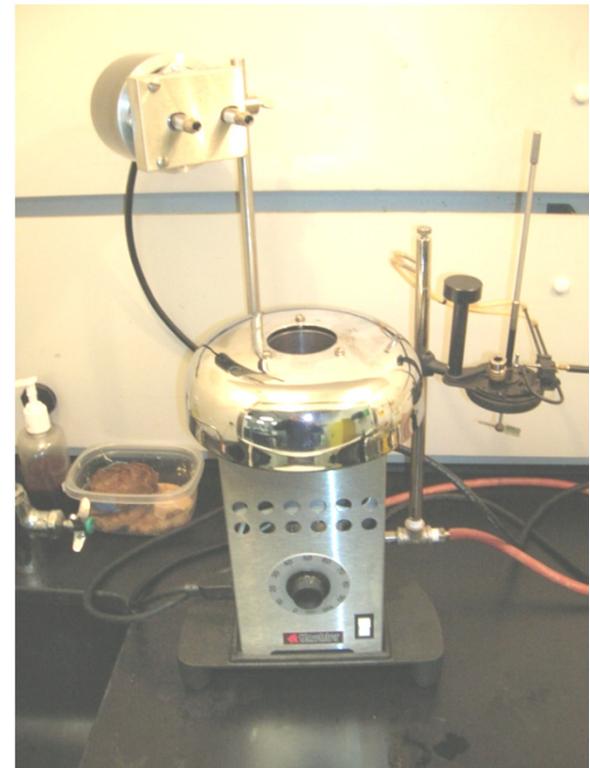
Result of test:

The flash point (temperature) of
the biodiesel sample

ASTM: 93°C Minimum

Effect on Biodiesel Quality:

Biodiesel with a high flash point will have different characteristics when used in an internal combustion engine.



Acid Number

Procedure:

ASTM D664-04

Equipment:

Denver Instruments Autotitrator Model 350

Result of test:

The Acid Number

ASTM: *0.80 mg KOH/g Max*

Effect On Biodiesel Quality

Highly acidic fuel can act as a solvent on rubber seals and hoses and can corrode fuel injection components



Cloud Point

Procedure:

ASTM D2500-02

Equipment:

Cloud Point Bath

Result of test:

The cloud point of the biodiesel sample

ASTM: "Report"

Effect on Biodiesel Quality:

The cloud point is an index of the lowest temperature in which the biodiesel can be used.



Water and Sediment

Procedure:

ASTM D2709-96

Equipment:

Koehler Heated Oil Centrifuge

Result of test:

This test indicates the presence of free water and sediment in Biodiesel.

ASTM: Less than 0.05% by volume

Effect on Biodiesel Quality:

Water causes problems in fuel systems and sediment can plug filters and leave deposits



Total & Free Glycerin

Procedure:

ASTM D6584-00

Equipment:

Gas Chromatograph

Result of Test:

Total and free glycerin

ASTM: Free: 0.02% mass

Total: 0.24% mass

Effect on Biodiesel Quality:

A high content of free glycerin can cause problems during storage while a high total glycerin content can lead to injectors becoming fouled and contribute to deposits on nozzles, pistons, and valves.



Sulfated Ash

Procedure:

ASTM D874-00

Equipment:

Thermolyne Furnace

Result of test:

Sulfated Ash is an indicator of how much soap or other residuals are in the fuel.

ASTM: Less than 0.02% mass

Effect on Biodiesel Quality:

It's usually an indication that there are contaminants in the fuel. High Sulfated Ash levels can clog Catalytic Converts on diesel vehicles.



27/3

Procedure:

27 mL of Biodiesel is combined with 3 mL of methanol and shaken vigorously. Then inspection of the sample to see if the biodiesel is completely reacted.

Result of Test:

This test is used to identify how complete the reaction of the vegetable oil was.

Effect on Biodiesel Quality:

If the vegetable oil didn't have enough methanol in the reaction then the transesterification process wasn't completed for the entire batch. Poorly reacted Biodiesel causes emulsions when water washing Biodiesel.



Sample Test

| | ASTM | Test Result |
|------------------|-------|-------------|
| Cloud Point | - | -1 °C |
| Flash Point | 93°C | 196°C |
| Water & Sediment | >.05% | 0% |
| 27/3 | - | Pass |

Questions

Messiah College Biodiesel Fuel Generation Project

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Appendix D.13.

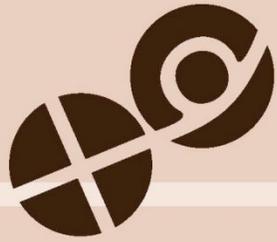
2010 Methanol Recovery Final Presentation

Biodiesel-Methanol Recovery

Steve Bray
Ben Studer

the Collaboratory
for strategic partnerships and applied research





Project Need

Methanol recovery system that can be used by the Biodiesel team to recover methanol from waste glycerin, a byproduct of biodiesel production.

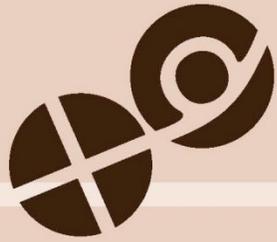
Why?

Environmental

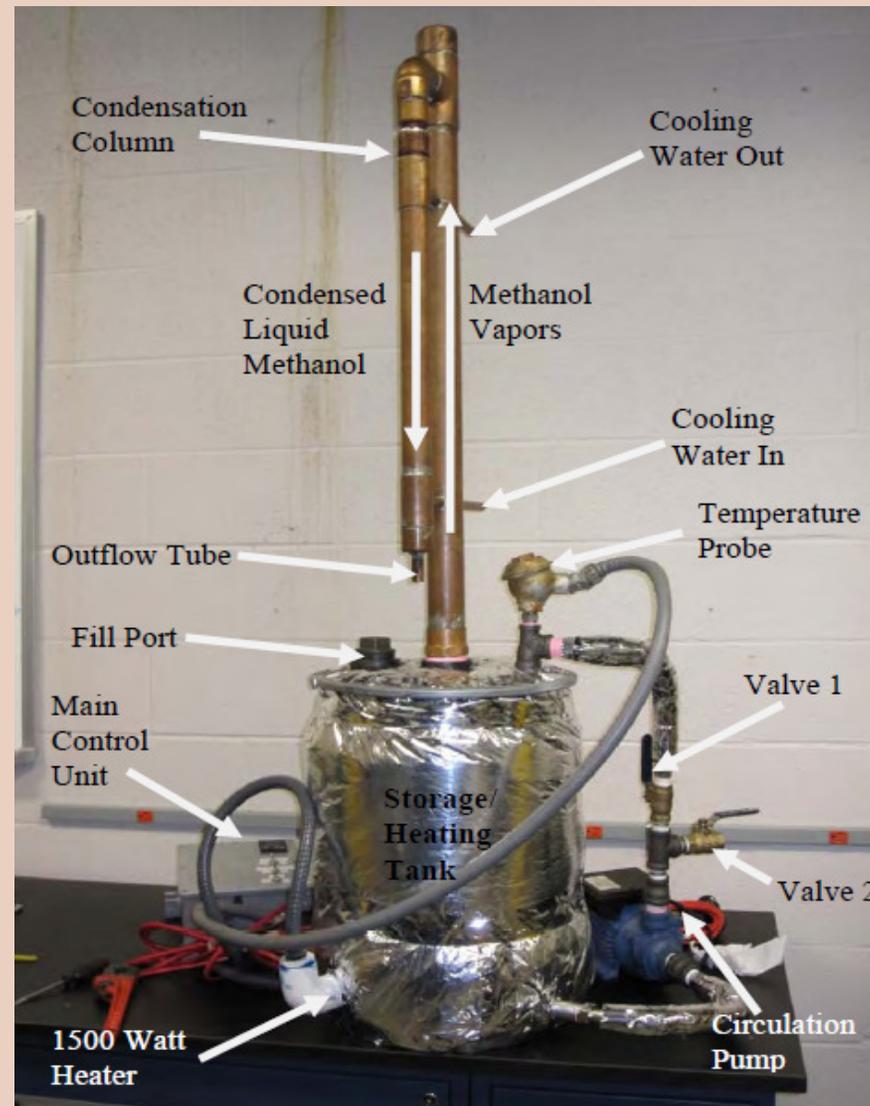
- Glycerin used for composting
- Glycerin with methanol environmentally hazardous

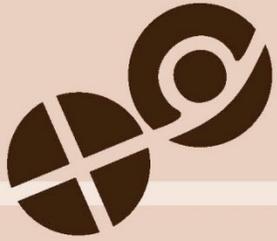
Economical

- Recycle methanol for reuse
- Disposable- glycerin with waste methanol has to be disposed of by chemically hazardous guidelines



How it works





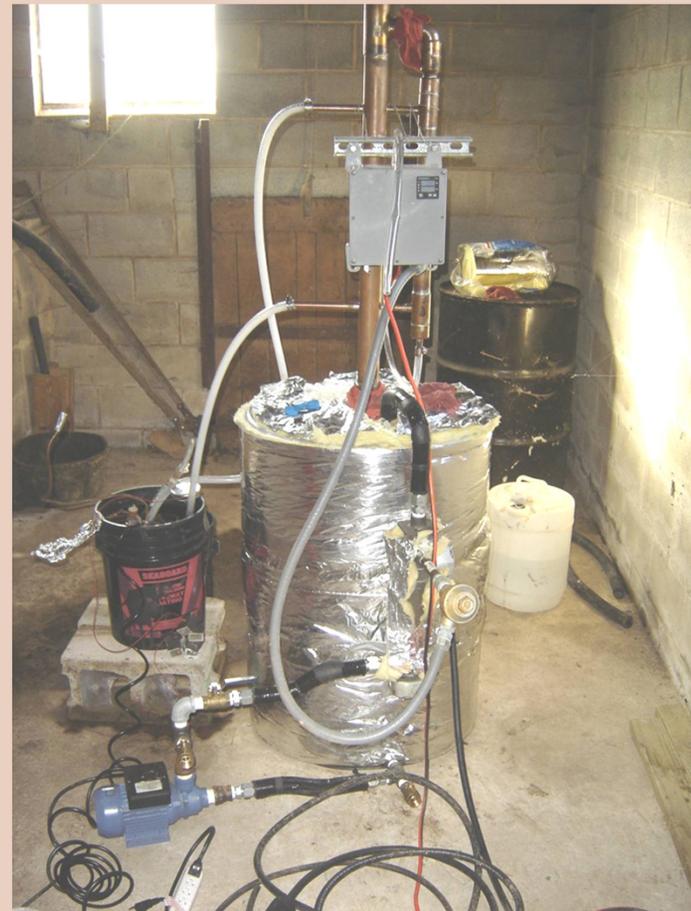
Project Objective

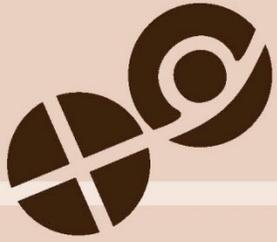
- **Build a system capable of recovering high quality methanol**
- **Test the recovered methanol to ensure quality**
- **Remove sufficient methanol to allow the proper disposal of the glycerine**



When last you saw...

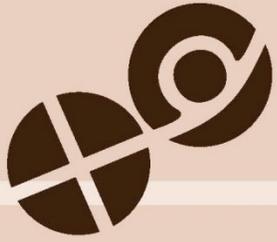
- The System was rather large
- The System was untested
- Relatively inefficient





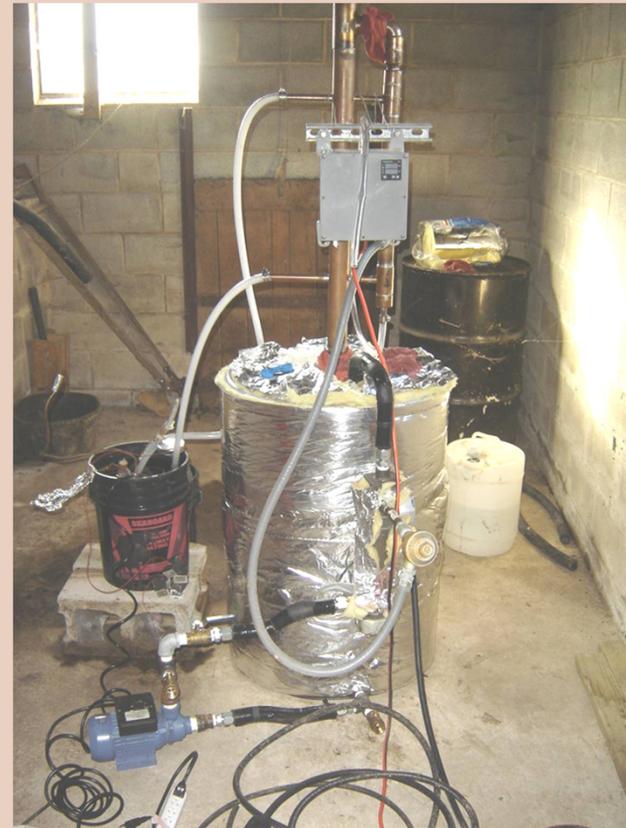
Since Last Year

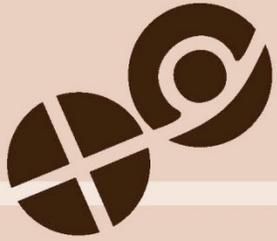
- **Project Received a Seed Grant from the Innovation Transfer Network**
- **Extensive testing on the old system**
- **Testing of the recovered Methanol**
- **Many improvements were made.**



Improvements Made

The size of the system was reduced

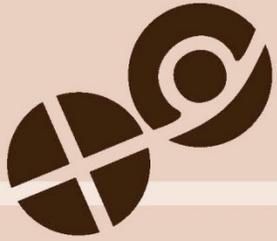




Improvements Cont'd

Temperature controllers were implemented to further refine the process





Improvements Cont'd

A water chiller was created to reduce the amount of water consumption





Testing

- Specific Gravity of the methanol correlates to methanol purity



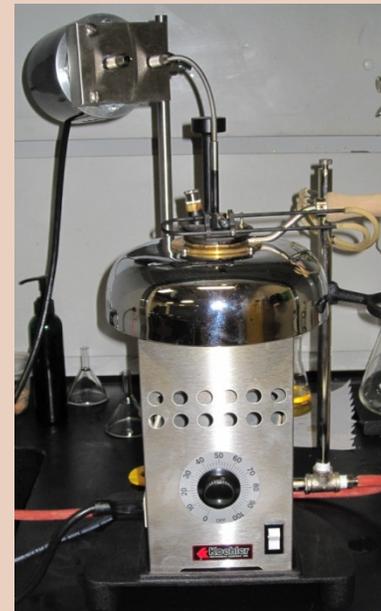
Hydrometer Temperature Adjustments
Reads in Percent Methanol Content
For Hydrometers Calibrated to 15C or 60F

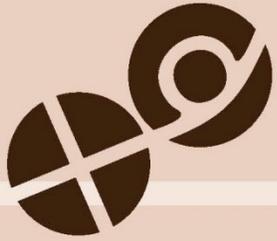
Actual measured specific gravity along left, actual measured temperature along top, Percent methanol content in middle

| | 52 F | 56 F | 60 F | 62 F | 64 F | 66 F | 72 F | 76 F | 80 F | 84 F | 88 F | 92 F | 96 F | 100 F |
|-------|------|------|------|------|------|------|------|------|------|------|------|------|------|-------|
| 0.871 | 73 | 72 | 71 | 71 | 70 | 69 | 69 | 68 | 67 | 66 | 65 | 64 | 63 | 62 |
| 0.868 | 74 | 73 | 72 | 72 | 72 | 71 | 70 | 69 | 68 | 67 | 66 | 65 | 64 | 64 |
| 0.865 | 75 | 75 | 74 | 73 | 73 | 72 | 71 | 70 | 69 | 68 | 67 | 66 | 65 | 65 |
| 0.862 | 77 | 76 | 75 | 74 | 74 | 73 | 72 | 71 | 71 | 70 | 69 | 68 | 67 | 66 |
| 0.859 | 78 | 77 | 76 | 75 | 75 | 74 | 74 | 73 | 72 | 71 | 70 | 69 | 68 | 67 |
| 0.856 | 79 | 78 | 77 | 77 | 76 | 75 | 74 | 73 | 72 | 71 | 71 | 70 | 69 | 69 |
| 0.853 | 80 | 79 | 79 | 78 | 78 | 77 | 76 | 75 | 74 | 73 | 73 | 72 | 71 | 70 |
| 0.850 | 81 | 81 | 80 | 79 | 79 | 78 | 77 | 76 | 75 | 74 | 73 | 72 | 71 | 71 |
| 0.847 | 82 | 82 | 81 | 80 | 80 | 79 | 78 | 78 | 77 | 76 | 75 | 74 | 73 | 73 |
| 0.844 | 84 | 83 | 82 | 82 | 81 | 80 | 79 | 78 | 77 | 76 | 75 | 74 | 73 | 74 |
| 0.841 | 85 | 84 | 83 | 83 | 82 | 82 | 81 | 80 | 79 | 78 | 77 | 77 | 76 | 75 |
| 0.838 | 86 | 85 | 84 | 84 | 83 | 83 | 82 | 81 | 80 | 79 | 79 | 78 | 77 | 76 |
| 0.835 | 87 | 86 | 85 | 85 | 85 | 84 | 83 | 82 | 81 | 81 | 80 | 79 | 78 | 77 |
| 0.832 | 88 | 87 | 87 | 86 | 86 | 85 | 84 | 83 | 83 | 82 | 81 | 80 | 79 | 79 |
| 0.829 | 89 | 88 | 88 | 87 | 87 | 86 | 85 | 85 | 84 | 83 | 82 | 81 | 81 | 80 |
| 0.826 | 90 | 90 | 89 | 88 | 88 | 88 | 87 | 86 | 85 | 85 | 84 | 83 | 83 | 82 |
| 0.823 | 91 | 91 | 90 | 89 | 89 | 88 | 88 | 87 | 86 | 85 | 84 | 84 | 83 | 82 |
| 0.820 | 92 | 92 | 91 | 91 | 90 | 89 | 89 | 88 | 87 | 86 | 85 | 84 | 84 | 83 |
| 0.817 | 94 | 93 | 92 | 92 | 91 | 91 | 90 | 89 | 88 | 87 | 86 | 85 | 84 | 83 |
| 0.814 | 95 | 94 | 93 | 93 | 92 | 92 | 91 | 90 | 89 | 88 | 87 | 86 | 85 | 84 |
| 0.811 | 96 | 95 | 94 | 94 | 93 | 93 | 92 | 91 | 90 | 89 | 88 | 87 | 86 | 85 |
| 0.808 | 97 | 96 | 95 | 95 | 94 | 94 | 93 | 92 | 91 | 91 | 90 | 89 | 88 | 87 |
| 0.805 | 98 | 97 | 96 | 96 | 95 | 95 | 94 | 93 | 93 | 92 | 91 | 90 | 89 | 88 |
| 0.802 | 99 | 98 | 97 | 97 | 96 | 95 | 94 | 94 | 93 | 92 | 91 | 91 | 90 | 89 |
| 0.799 | 100 | 99 | 98 | 98 | 97 | 96 | 95 | 95 | 94 | 93 | 92 | 91 | 91 | 90 |
| 0.796 | | 100 | 99 | 99 | 98 | 97 | 96 | 95 | 94 | 94 | 93 | 92 | 91 | 90 |
| 0.793 | | | 100 | 100 | 99 | 98 | 97 | 96 | 95 | 95 | 94 | 93 | 92 | 91 |
| 0.790 | | | | | 100 | 99 | 98 | 97 | 96 | 95 | 95 | 94 | 93 | 92 |
| 0.787 | | | | | | 100 | 99 | 98 | 97 | 96 | 95 | 94 | 93 | 92 |
| 0.784 | | | | | | | 100 | 99 | 98 | 97 | 96 | 95 | 94 | 93 |
| 0.781 | | | | | | | | 100 | 99 | 98 | 97 | 96 | 95 | 94 |
| 0.778 | | | | | | | | | 100 | 99 | 98 | 97 | 96 | 95 |
| 0.775 | | | | | | | | | | 100 | 99 | 98 | 97 | 96 |

Accuracy +/- 1%

- Flash point test glycerine to ensure safe disposal

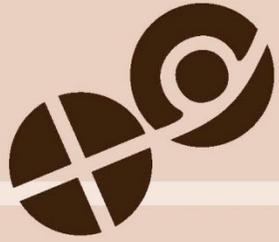




Current Work

- Using the temperature monitoring system to identify critical temperatures on the system throughout the methanol recovery process.
- Testing the purity of recovered methanol
- Testing the glycerine to ensure safe disposal





Composting

- Began working with the composting project on campus to dispose of glycerine
- Partnership has many benefits





Future Work

- Continue to monitor critical system temperatures in an effort to correlate them to recovered methanol purity
- Continue testing purity of recovered methanol
- Continue testing of glycerine to ensure safe disposal
- Begin work on a potential tie in to the biodiesel processor through the use of a PLC

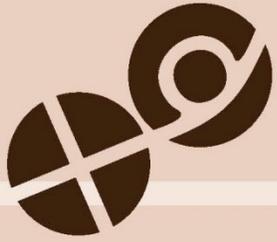


Acknowledgements

Professor Erikson

Mike Zummo

The Biodiesel Project Team



Questions?