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 produces 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 ordered 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 distilate is pure. The website mentioned above also listed several useful suggestion and resources for other suitable materials in building a methonal 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 specefically for methanol recovery from waste glycerol. Research done so far is a step in the right direction to starting the methanol recovery project.

References

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

Appendix C

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 is 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

Appendix C

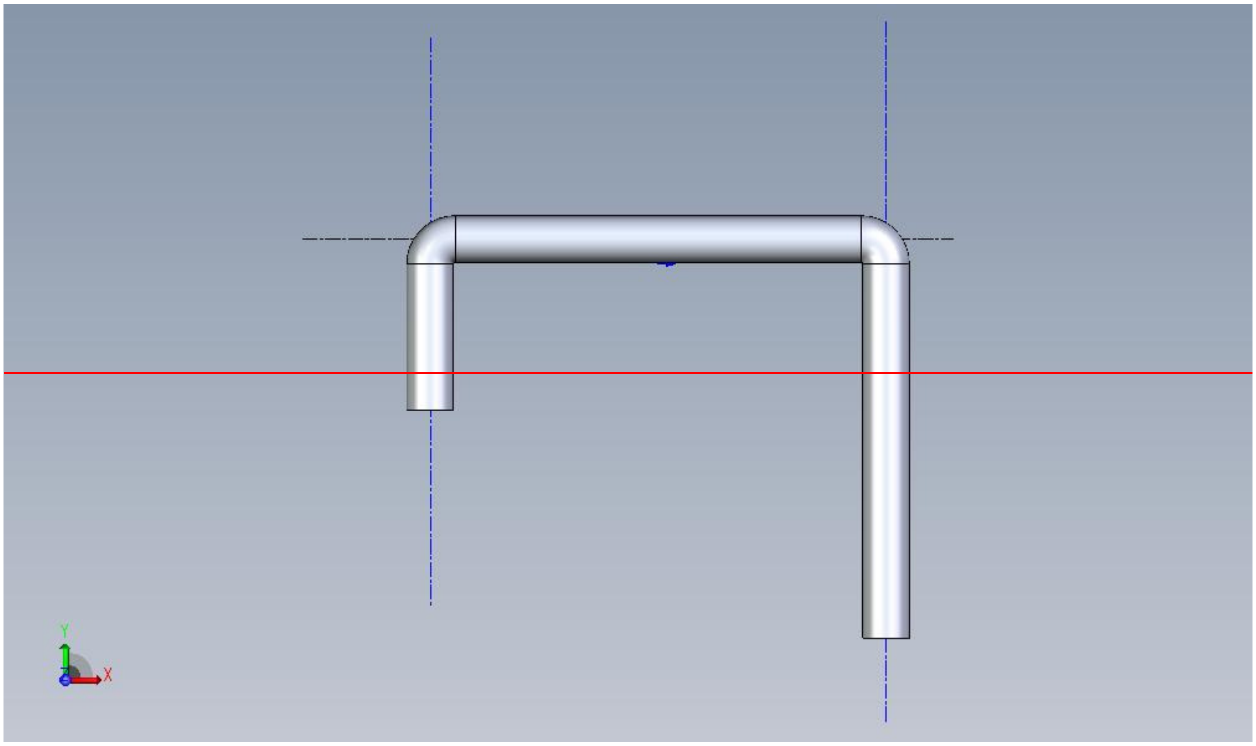
For testing and refinement phase of project the gas chromatograph will be used in testing the purity of refined methanol to ensure it can be reused in biodiesel production. It is the Biodiesel teams hope that such a gas chromatograph will be obtained with DOE Grant money before winter. If not the Chemistry 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

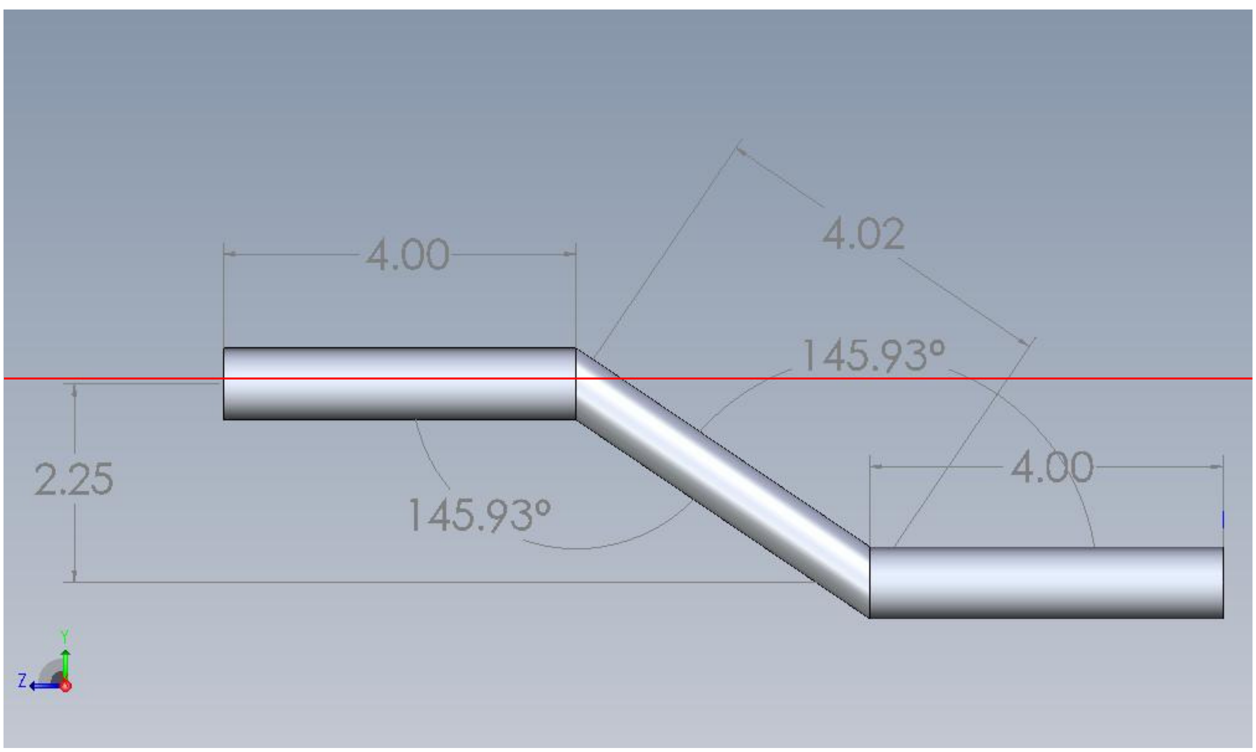
ASTM testing literature will be vital to the assurance in quality of recovered methanol distillate. Such testing literature will be obtained either at the library, or online sources when necessary.

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

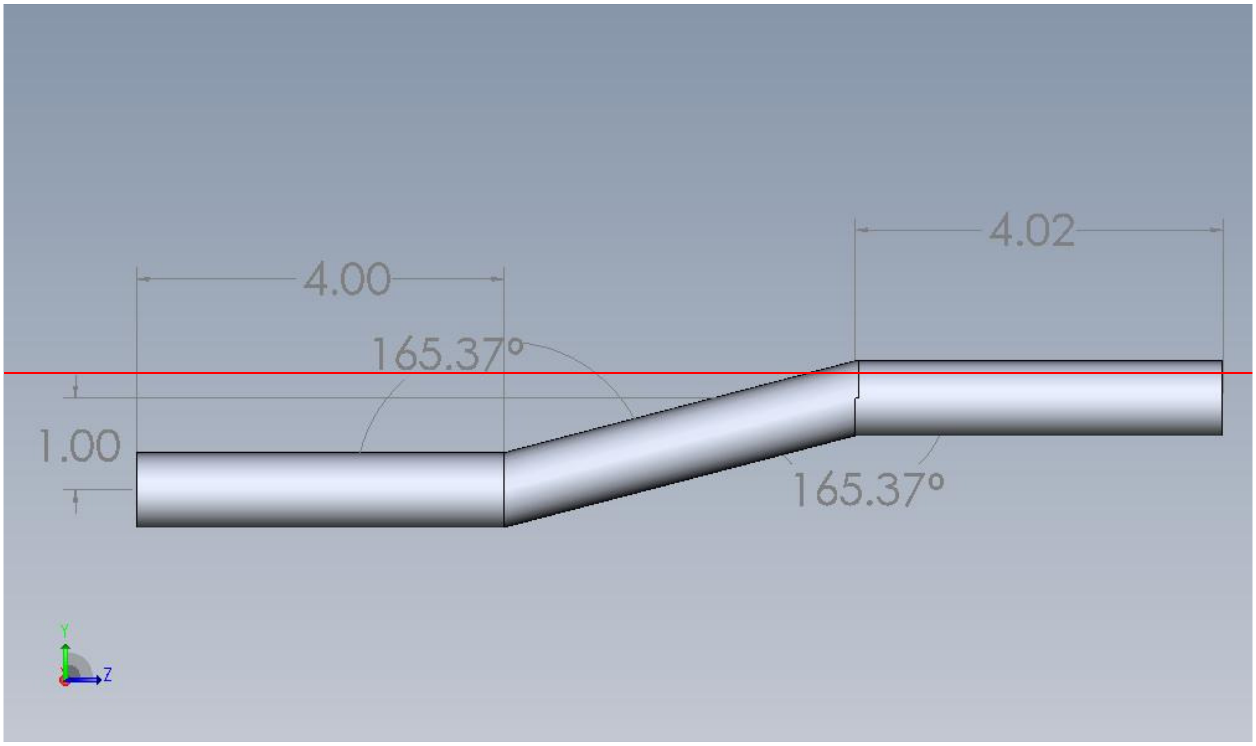
Coupling					
Adapter	3/4" Threaded to Pipe	6	Free	Free	Andy Derr
		Tax	$ 0.98		
		Total	$ 17.38		
Miscellaneous					
Stainless Steel Hose Clamps	1/2"	3	Free		Biodiesel Team
Steel Mesh Screen	1" x 1"	2	Free		Biodiesel Team
Drum	55 gallon	1	Free		John Meyer
Heater Core	4500 W	1	Free		Biodiesel Team
Bucket	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