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CONTINUOUS FLOW BIODIESEL PRODUCTION

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Summary: A continuous flow esterification process for producing biodiesel from rapeseed oil and ethanol was investigated. The process consists of an oil metering pump, centrifugal mixing pump, alcohol and catalyst metering pump, static mixers, "ladder" type retention reactor, water injection system, and continuous flow centrifugal separating system. The system has met the proposed ASTM standard for free and total glycerol.

Keywords: Biodiesel, Transesterification, Continuous Flow Transesterification

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Continuous Flow Biodiesel Production

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Abstract

Biodiesel is a potential replacement for a portion of the diesel fuel used in transportation. It is produced from both waste and new vegetable oils. It has several advantages. Among these are its classification as a renewable resource, its ability to reduce HC, CO and CO_2 exhaust emissions, its non-toxic character, and its biodegradability. Plant oils grown around the world can be harvested and converted to biodiesel using current esterification processes. One of the keys to making biodiesel a viable and profitable energy source is the use of a continuous flow transesterification process to reduce time and cost, thereby increasing production and profit. Most biodiesel is produced in manual or automatic batch processes. Research and development into continuous flow processes is relatively unperfected in the United States, and therefore warrants further investigation.

In this study a continuous flow esterification process for producing biodiesel from rapeseed oil and ethanol was investigated. The process consists of an oil metering pump, centrifugal mixing pump, alcohol and catalyst metering pump, static mixers, "ladder" type retention reactor, water injection system, and continuous flow centrifugal separating system. The system was assembled and tested. Oil flow rate used was 0.38 L per minute which would be about 3 times the production per week of our existing batch type transesterification system which produces 945 L (250 gal) per batch.

The system in its present configuration has met the proposed ASTM standard for free and total glycerol; it is anticipated that if methanol were used instead of ethanol that it could do so at a much faster flow rate than with ethanol. The centrifugal separation also results in excessive alcohol vapors coming off. A vapor recovery and condensing system will be installed. This latter system should help further reduce costs of the biodiesel produced by recycling some of the alcohol used.

Keywords: Biodiesel, Transesterification, Continuous Flow Transesterification.

Ethanol is the preferred alcohol because it is derived from agricultural products and is renewable and biologically less objectionable in the environment (Peterson et al., 1990).

Production cost and time for producing a small amount of fuel is high using current batch reactor methods. Therefore, a continuous flow process for trans-esterification of vegetable oils is in development. A continuous flow process would allow more fuel to be produced and, on a larger scale, reduce cost. Also, using previous design considerations and equipment, modifications and additions have been made to increase the potential for producing a higher quality fuel.

Problem Definition

Current batch methods of producing Biodiesel are slow, tedious, labor intensive, and not well adapted to automation. It is desired to construct and test a continuous flow transesterification process for the conversion of vegetable oils into Biodiesel.

Objectives

The objective of this project was to plan, construct, and test a continuous flow transesterification apparatus for producing biodiesel. The literature was reviewed to determine probable flow-rates, probable raw product quantities, and previous experiments in order to determine a process which would have a good chance of operational success.

Other objectives of the project were to:

- Develop design specifications and build a prototype system.
- Modify the prototype based on initial tests for better performance.
- Analyze biodiesel produced for comparison with batch reactor and ASTM standards.

Literature Review

Typical fuel properties of both raw rapeseed oil and modified esters derived from rapeseed oil are included in Table 1 for comparison with Diesel No. 2.

University of Idaho personnel are producing biodiesel using ethanol and KOH in the transesterification reaction instead of methanol as is used in most biodiesel production plants. Benefits of using ethanol are:

- Ethanol is an environmentally friendly and a product of renewable resources;
- Ethyl and methyl esters have similar fuel characteristics;
- Compared to methanol, ethanol is much safer to handle because toxic effects to personnel from exposure to the fumes are reduced.

Biodiesel composed of hydrogenated soy ethyl esters has been termed HySEE (Lowe et al., 1998). The transesterification process consists of four steps: 1) raw material pre-treatment; 2) ethyl ester transesterification; 3) separation of the ester and glycerin phases; and 4) purification of the ethyl esters. The first two steps of this procedure require the addition of heat to the oil and its reacting apparatus, because the waste oil is solid at room temperature. A similar procedure can be used in the continuous transesterification process for the conversion of used potato processing oil into HySEE biodiesel fuel in the future.

A patent search was performed to determine whether any patents applied to the proposed design process. Two patents relating to biodiesel production were found. Braden (1996) covers a method for producing biodiesel fuel however continuous flow was not mentioned and Bam and Drown (1995) discusses a method for purifying alcohol esters in a continuous batch process. No patents were discovered which covered a continuous flow biodiesel production process as discussed in this article.

Materials and Methods

Transesterification requires the basic materials including vegetable oil (in this case canola or rapeseed), ethanol (EtOH), potassium hydroxide (KOH), and tap water.

Equipment used in the project included two different Alfa Laval continuous flow centrifuges (Alfa Laval Model WSB 103B-74-60 and Alfa Laval Model Gyro-Tester, this latter centrifuge was on loan from the J. R. Simplot alcohol plant); oil, ester, and glycerol containers; a Fluid Metering, Inc. (FMI) metering pump (A. O. Smith , Model QDX1, 0 to 550 ml/min); a hydraulic motor driven centrifugal mixing pump (Delaval Mfg.); an oil metering pump (Teel, model 1P771 gear pump with 1/3 hp Pacific Scientific DC permanent magnet motor and a Dart Controls, Inc. model 253G-200E controller) and oil flow meter (Omega model FL-2100). In-line static mixers were obtained from Cole-Parmer (stock no. H-046667-08, unmounted OD ½", 12 elements per 6 ¼ inch length). Water flow was controlled with a Parker #600S needle valve monitored with an M and M, model 406 PX gasoline flowmeter.

Two different continuous flow set-ups were examined in this project. These will be referred to as CF-1 and CF-2. Figure 2 is a schematic of the components and flow path of the final continuous flow system.

Continuous Flow System 1 (CF-1)

CF-1 consisted of the mixing pump, alcohol/catalyst metering pump, static mixers placed in Clear polyvinyl chloride tubing followed by 100 feet of 1-in. I.D. Clear polyvinyl chloride tubing arranged in a circular pattern around a plastic barrel (Fig. 3) and either the gyro-tester centrifuge or a settling tank to remove the glycerol. The settling tank was used or test runs before the centrifuge became available.

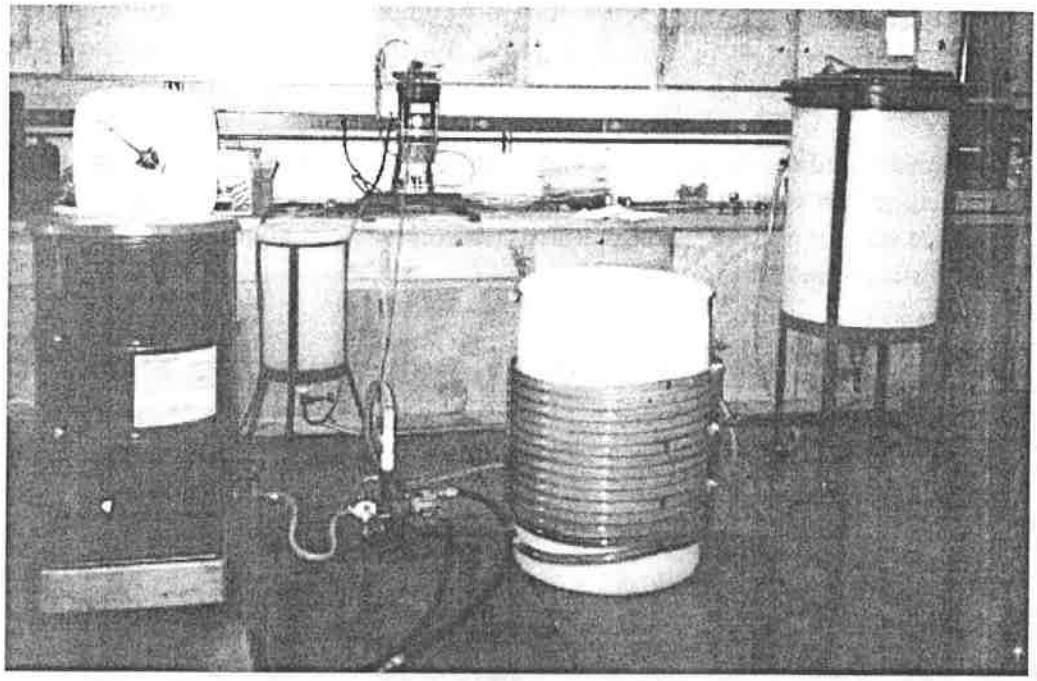

Figure 3. First attempt at a reactor for the continuous flow system. It consisted of about 30.5 m (100 ft) of 25.4 mm (1-in) clear polyvinyl chloride tubing in a circular pattern. Also shown in this picture are the centrifugal mixing pump to the left of the reactor and the FMI metering pump on the shelf to the left and rear of the reactor.

Continuous Flow System 2 (CF-2)

CF-2 was a second generation continuous flow system which was constructed using ideas learned during the development of CF-1.

Two major changes distinquish CF-2 from CF-1. First, a second metering pump was placed ahead of the centrifugal mixing pump to more accurately meter the flow of vegetable oil into the mixer. Second, a redesigned retention reactor was constructed. For this reactor eleven, 2.9 m (9.6 ft) sections of 31.8 mm (1 1/4 inch) schedule 40 PVC pipe were assembled into a vertical "ladder" shaped reactor. Each pipe had a slope of 25 mm (1-inch) over it's 3 m (10-ft) length. Sections were connected with two 90 degree elbows and a short section of pipe causing the individual lengths to be separated by 120.6 mm (4.75 in) (figure 5). At the bottom rung of the network a vertical length of 22 mm (½ in) PVC was used to make the outlet of the ladder at the same elevation as the inlet. This not only helped assure that the glycerol would not accumulate at the bottom but made sure that the reactor was filled with reactant.

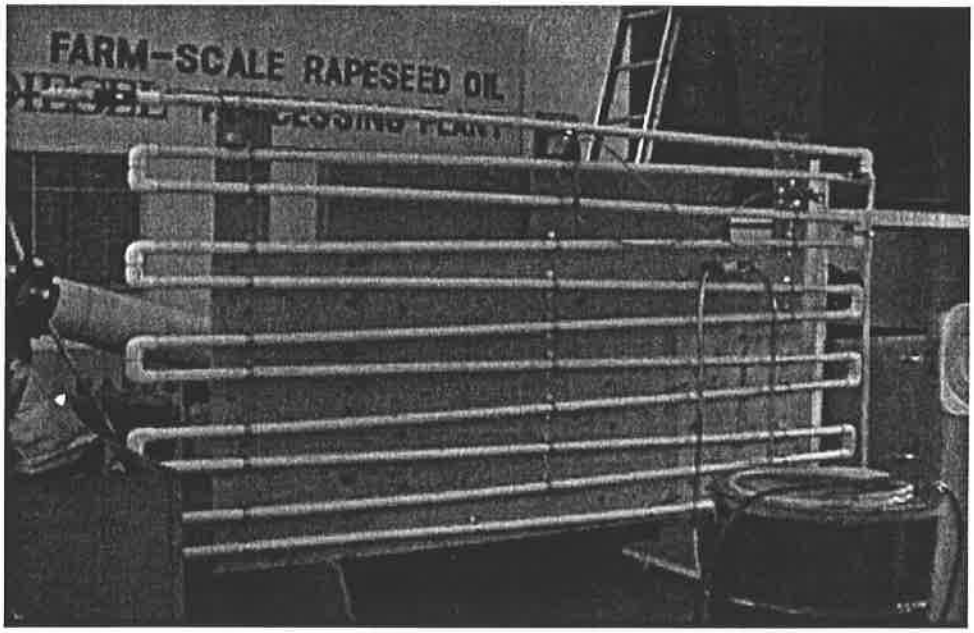

Figure 5. "Ladder type" retention reactor used in the final design for the continuous flow biodiesel production system. The reactor consists of 33.5 m (110 ft) of 31.8 mm (1 1/4-in) PVC arranged in 11 steps with 1.8 m (6-ft). of 22 mm (½ in) PVC vertical exit section.

mixture exits the mixing pump, passes through a series of static mixers and into the retention reactor. As the biodiesel exits the retention network, it passes through a series of static mixers and then a spray valve adds water to the mixture at a rate of 57 ml/min. The mixture then passes through another series of static mixers and is separated in the centrifuge into ester and glycerol phases.

The Teel positive displacement pump with a Dart Controls Inc. controller was used to meter oil from the supply to the centrifugal mixing pump. In addition to moving the oil in a positive feed in to the mixing pump, the metering pump produces a back pressure to prevent any of the catalyst from flowing back into the barrel of oil. It was discovered from calibration of the metering pump and controller that it would not sustain a consistent pumping rate when the motor driving the pump was operated at speeds less than 20 rpm. Therefore, the motor was geared down using a chain drive to allow the motor to operate at higher RPMs while allowing the pump to operate at the desired rate.

Table 2

Calibration Data of FMI Pump

Trial #	Time (Sec)	Flow (mL/min)
1	58	110
2	59	111
3	59	111
4	59	111
5	59	111
6	59	111

The final calibration was performed as the Biodiesel was produced during the trial run. The water insertion valve was initially set at 22.7 (6 gal/hr), but resulted in a foamy product in the exit tubing. Flow was eventually reduced to 9.1 L/hr (2.4 gal/hr), resulting in a product which was similar in appearance to that produced with the batch process.

Start Up and Shutdown Procedure – One of the problems with a continuous flow system as described in this report is the amount of material required to fill the system. Approximately 22.7 L (6 gal) of mixture is required to fill the system and once the mixture is in the system it is difficult to empty on shut down.. The following procedure was used for starting and stopping the production of biodiesel with the continuous flow system.

Start Up:

1. A brass air bleed valve at the top, entrance end of the retention network was opened to allow air to escape while the system was filling.
2. The flow regulator oil pump and FMI pump were primed with vegetable oil and ethananol/catalyst, respectively.
3. The hydraulic driven centrifugal mixing pump was started.
4. The flow regulator oil pump and ethanol/catalyst metering pump were turned on at appropriate flow rates.
5. Filling of the retention network took approximately one hour. Once product began to exit the bleed valve, it was closed and reaction products start to flow out the exit of the reactor network toward the water spray nozzle.
6. The water injection and Alfa Laval Centrifuge were primed and turned on.
7. Collection of separated glycerol and ester began.
8. The system flow rates required monitoring to assure a quality product.

produced. The results for the three test runs are shown in Table 3. Analysis of the biodiesel for the best run showed biodiesel contained 0.016 percent free glycerol and 0.236 total glycerol, both of which are within the proposed ASTM specification for biodiesel. Potassium analysis provide a means to determine the amount of catalyst remaining in the product (ester/biodiesel). It should be mentioned that current batch methods, using ethanol, do not always meet ASTM quality standards for free and total glycerol without additional processing.

A second consideration is the amount of ester remaining in the glycerol phase. This is easily recovered because the ester will be the top layer and can be recycled through the centrifuge.

Table 3
Laboratory Analysis of Process Sensitive Parameters
for Biodiesel Produced in Continuous Flow Process Configuration CF-2

Analysis	ASTM Limit	Run 1	Run 2	Run 3
Free Glycerol (%)	0.02	0.036	0.021	0.016
Total Glycerol (%)	0.24	1.49	0.267	0.236
Viscosity @ 40° C (mm^2/s)	1.9 - 6.0	5.81	5.9	6.0
Potassium (ppm)		170	16	27
Ethanol (%)		0.12	0.09	0.09

Cleanup and Maintenance -- The cleanup process for the retention network works well. The valve at the bottom of the network at the pipe junction allow flow to continue from the network after the oil source is removed. The tube at the top exit of the network is removed and attached at the lower valve. Once the lower valve is opened, gravity forces the remaining reaction mixture out the tube and on to the rest of the process. Overall, cleanup takes about 1 ½ hours.

Following each use, the retention network should be flushed first with water then with ethanol to provide a pure test each time. In addition, the centrifuge should be cleaned each time it is used. An emulsive material builds up on the inner sections, potentially reducing long-term separation effectiveness.

Problems -- Some problems were noticed during the first run. The chief problem was associated with pressure build up inside the retention network as it was filling with reactants. A brass air bleed valve was inserted at the top end of the network. A small tube was inserted onto the valve, leading into a beaker. The network was full when product came out of the tube into the beaker. The valve was then closed.

Conclusions

The continuous flow reactor successfully produced ester from canola oil. The ester was within the limits of the proposed ASTM standard specification for biodiesel. It is anticipated that refinement of the system could further improve the final product. At a flow rate of 0.38 L /min or 22.8 L per hour, the continuous flow system would produce as much biodiesel in 41.5 hours as one batch from the existing 945 L per batch system . Ordinarily, the batch system produces one batch per week so the small, pilot scale, continuous flow system would increase our present production capacity by three fold if operated 24 h/day.

Other conclusions of these tests are:

1. Capital cost of the system is reasonable, the addition of a computer process control system would be desirable.
2. Production of biodiesel at a rate three times the existing batch system appears feasible.
3. Alcohol vapors coming from the centrifuge should be recovered system to improve safety and environmental impact.

Recommendations

Additional testing of the continuous flow reactor should include studies of the following:

1. a computer monitoring and control system for sensing flow of oil and control of the alcohol/catalyst mixture injection rate.
2. further studies on ester flow rate and centrifuge adjustments on ester purification.
3. a method for recovering the excess ethanol from the ester and glycerol phases and an apparatus to capture the ethanol vapor leaving the centrifuge.
4. secondary treatment including consideration of a second centrifuge step for further purification.

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