CONTINUOUS FLOW BIODIESEL PRODUCTION

C. L. Peterson, J. L. Cook, J. C. Thompson, J. S. Taberski

ABSTRACT: Biodiesel, which consists of the fatty acid esters of simple alcohols, is a potential replacement for a portion of the diesel fuel used in transportation. It is produced from both used (oil that has been utilized for frying and discarded) and new vegetable and animal fats and oils. It has several advantages. Among these advantages are its classification as a renewable resource, its ability to reduce HC, CO, and CO₂ exhaust emissions, its non–toxic character, and its biodegradability. 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.

A continuous flow esterification process for producing biodiesel from rapeseed oil and ethanol was investigated. The equipment consisted of an oil metering pump, centrifugal mixing pump, ethanol and catalyst metering pump, static mixers, “ladder” type retention reactor, water injection system, and continuous flow centrifugal separating system. The oil feed rate was 0.38 L/min that yields about three times the weekly production of the existing batch type transesterification system that produces 945 L (250 gal) per batch. It was anticipated that if methanol were used instead of ethanol that the flow rate could be considerably increased.

The system in its present configuration has met ASTM PS121–99 standard for free and total glycerol. The centrifugal separation resulted in release of excessive alcohol vapors. A vapor recovery and condensing system should be installed. This latter system could help further reduce costs of the biodiesel produced by recycling some of the alcohol used.

Keywords: Biodiesel, Transesterification, Continuous flow transesterification.

Use of biodiesel, which consists of the fatty acid esters of simple alcohols, derived from vegetable oil and animal fats as a renewable fuel source may provide benefits to the environment. Among its advantages are reduced HC and CO exhaust emissions, non–toxicity, and biodegradability. Carbon dioxide production in biodiesel combustion is offset by the carbon dioxide use of the plant in photosynthesis. Nitrous oxides and particulate matter changes depend on the particular test conditions. However, exhaust particulate matter toxicity and mutagenicity have been shown to be reduced. Biodiesel is biodegradable with potential less harm to the environment. Among 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.

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less objectionable in the environment (Peterson et al., 1996). Compared to methanol, ethanol is safer to handle because toxic effects to personnel from exposure to the fumes are reduced. Ethanol is more expensive than methanol and cannot be derived as readily as methanol. For those reasons methanol is more widely used for biodiesel production than is ethanol. Benefits of using a continuous flow system include:

- Production cost and time for producing a small amount of fuel is high using current batch reactor methods.
- A continuous flow process would allow more fuel to be produced per unit of labor and, on a larger scale, reduce cost.
- With design improvements to optimize equipment, continuous flow has the potential for consistently producing a higher quality fuel.

**OBJECTIVES**

Current batch methods of producing biodiesel are slow, tedious, labor intensive, and not well adapted to automation. 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 to determine a process that 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.

Noureddini et al. (1996) detailed an approach for a continuous process for the transesterification of triglycerides to methyl esters. This process is based on a combined high shear and motionless reactor system. Their experimental studies explored the available variations in the mixing intensity, stoichiometry, and catalyst concentration on the overall conversion of vegetable oils to esters.

The reaction materials used by Noureddini et al. (1996) to produce biodiesel were refined and bleached grade soybean oil, anhydrous methyl alcohol, and sodium hydroxide. A solution of 25 g of sodium hydroxide per 1 L of methanol was prepared for mixing with the soybean oil in the continuous flow system. The article describes the setup of the pilot plant in detail. The major operations of the system included a pump station, preheating/mixing, mixing, a residence tube, cooling, and settling.

Peterson et al. (1991) discussed the characteristics of the batch type transesterification process for winter rapeseed oil. The main variables in the process were: catalyst type, catalyst concentration, oil/alcohol ratio and stirring rate. They found that 1% NaOH or KOH effectively enhanced the reaction rate at room temperature (a 60–min reaction time was allowed). A 6:1 molar ratio of methanol to oil gave the best conversions in a batch system. In addition, the rate of reaction was satisfactory if the stirring action was vigorous with some splashing.

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 glycerol 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.

Two patents were found relating to biodiesel production. Bradin (1996) described a method for producing a unique biodiesel fuel that is outside the normal definition of biodiesel being a fatty acid ester of simple alcohols. In his patent he discloses a fuel additive composition that includes fatty acid alkyl esters and glyceryl ethers, as well as a method for preparing the composition. The fuel additive composition can be added to diesel fuel to provide an alternative diesel fuel composition. Continuous flow production was not a claim of the patent. Bam et al. (1995) discussed a method for purifying alcohol esters in a continuous batch process. No patents were discovered that 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, used hydrogenated soy oil (used cooking fat), or rapeseed oil], ethanol, potassium hydroxide. Tap water was employed to facilitate product separation from other reaction components. The ethanol was 200 proof industrial grade ethanol supplied by the J. R. Simplot Company (Caldwell, Idaho). The J. R. Simplot Company produces ethanol from waste potato products and generally supplies the ethanol into the motor fuel market for use in gasohol.

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); 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 speed 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 .5 in., 12 elements per 6.25 in. length). Water flow was controlled with a Parker no. 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 or CF–2 continuous flow system.

CONTINUOUS FLOW SYSTEM 1 (CF–1)

CF–1 consisted of the centrifugal mixing pump, FMI alcohol/catalyst metering pump, static mixers placed in clear polyvinyl chloride tubing followed by 100 ft 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 used to remove the glycerol. The settling tank was used on test runs before the centrifuge became available.

The first test made with CF–1 was with used frying oil. Since this oil is solid at room temperature the reservoir was heated with a 120–V barrel heater. It was placed on a spring scale in order to monitor flow rate by weight loss. The mass flow rates of oil and alcohol/catalyst were based on calculations made using the batch process recipe. After being mixed with alcohol/catalyst and fed into the retention network, flow was not a problem. Water was added to the reactants at the exit end of the retention tubing to increase the separation rate of the glycerol from the ester. The mixture was then fed into the midpoint of a settling tank. When the settling tank was full and had reached equilibrium, ester was taken off the top and glycerol was drained off the bottom in proportion equal to the inflow rate.

Subsequent testing with CF–1 involved the use of an Alpha Laval gyro–tester continuous centrifuge (fig. 4) to separate the ester from the glycerol phase. Using this system, a second test was conducted with used frying oil and a third with canola oil. The canola oil, because of its lower pour point, eliminated the requirement for heating. Oil flow rate was difficult to monitor and control and the retention time of the system was only 30 min resulting in less than desirable conversion efficiency.
Figure 4. Alfa Laval model WSB 103B–74–60 continuous flow centrifuge used for ester and glycerol separation.

CONTINUOUS FLOW SYSTEM 2 (CF–2)

CF–2 was a second–generation continuous flow system that was constructed using ideas learned during the development of CF–1. Two major changes distinguish 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 as shown in figure 5. For this reactor eleven, 2.9–m (9.6–ft) sections of 31.8–mm (1.25–in.) schedule 40 PVC pipe were assembled into a vertical “ladder” shaped reactor. Each pipe had a slope of 25 mm (1 in.) over its 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). At the bottom rung of the network a vertical length of 12.6–mm (.5–in.) PVC was used to make the outlet of the ladder at the same elevation as the inlet. This vertical length of PVC not only helped assure that the glycerol would not accumulate at the bottom but also ensured that the reactor was filled with reactant. Quick–release hydraulic couplers were used at the inlet and outlet for connection into the system. Nine feet of static mixers in 12.6–mm (.5–in.) clear polyvinyl chloride tubing were integrated into the new system.

A platform was constructed for the pumping and metering portions of the network. The centrifuge was mounted on a movable frame and an explosion proof controller was attached.

A separate movable self–supporting wall held the retention reactor network. Figures 4 and 5 are photographs of the various components.

CF2 Operation

For convenience in these tests canola oil [density = 0.915 g/mL (7.61 lb/gal)] was used to produce biodiesel. Canola oil is liquid at room temperature and is pure enough to prevent settling of foreign matter within the retention network and connections. Just prior to beginning a run, the catalyst was mixed using a ratio of 11.6 L (3.07 gal) ethanol [density = 0.789 g/mL (6.565 lb/gal)] to 2.2 kg (1 lb) KOH.

The process started with oil metered into the inlet of the centrifugal mixing pump with the positive displacement oil–metering pump at a flow rate of 380 mL/min (0.10 gal/min). At the same time, the FMI metering pump regulated the flow of alcohol/catalyst at a rate of 111 mL/min (0.029 gal/min). The catalyst and oil were mixed with high shearing action in the hydraulically driven centrifugal mixing pump. The mixture exited the mixing pump, passed through a series of static mixers, and into the retention reactor. As the biodiesel exited the retention network, it passed through a series of static mixers. A spray valve then added water to the mixture at a rate of 57 mL/min. The mixture then passed through another series of static mixers and was separated in the centrifuge into ester and glycerol phases.

Explosion Proof Considerations

It is necessary to use explosion–proof switches and service entrance connections since ethanol and methanol are highly flammable. The centrifugal mixing pump was powered with a hydraulic motor, which is a convenient method for providing both speed control and prevention of explosion hazards.

Calibration Data

Calibration was performed on the flow–regulating pump and a straight line fit to the data provided a model for the
Table 1: Oil Pump Calibration Curve

<table>
<thead>
<tr>
<th>Flow Gage Reading</th>
<th>Flowrate (mL/min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>18</td>
<td>140</td>
</tr>
<tr>
<td>21</td>
<td>200</td>
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<tr>
<td>21</td>
<td>300</td>
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<td>21</td>
<td>330</td>
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<tr>
<td>22</td>
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</tr>
<tr>
<td>22.5</td>
<td>355</td>
</tr>
<tr>
<td>23</td>
<td>380</td>
</tr>
<tr>
<td>23</td>
<td>410</td>
</tr>
<tr>
<td>23</td>
<td>500</td>
</tr>
<tr>
<td>25</td>
<td>600</td>
</tr>
</tbody>
</table>

Figure 6. Calibration data for the Teel positive displacement oil flow regulating pump.

pump flow adjustments necessary during biodiesel production (fig. 6). Calibration was also performed on the FMI pump for modeling the flow of the ethanol/catalyst mixture resulting in 111 mL/min at the setting used in these tests.

The final calibration was performed as the biodiesel was produced during the trial run. The water insertion valve was initially set at 22.7 L/h (6 gal/h), but this flow rate resulted in a foamy product in the exit tubing. Flow was eventually reduced to 9.1 L/h (2.4 gal/h), resulting in a product that 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, like this, is the amount of material required to fill the system. Approximately 22.7 L (6 gal) of mixture is required to fill the system. Once the mixture is in the system it is difficult to empty. 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 ethanol/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 1 h. Once product began to exit the bleed valve, it was closed and products started 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.

**Shutdown**

1. The flow regulator oil pump, centrifugal mixing pump, and ethanol/catalyst metering pumps were turned off.
2. A valve at the entrance to the retention reactor network was turned off.
3. A valve at the exit to the retention reactor network was turned off and the exit tubing was disconnected and reconnected at the lowest point of the network.
4. A valve at the bottom outlet position of the retention reactor network was turned off and the air bleed valve was opened allowing the remaining product to drain from the network and through the water injection and centrifuge.
5. After product stopped flowing from the retention network, the water and centrifuge were turned off.

Glycerol will settle from the mixture if left in the system. An alternative to draining is to fill the system with ester using the oil–metering pump. Filling with ester would take about an hour and 22.7 L (6 gal) of ester. When the system is restarted the ester would pass through the centrifuge and be reclaimed.

**Analytical Measurements**

Free and total glycerol were determined using the Iodometric–Periodic Acid method described in AOCS Ca 14–56, Total, Free and combined Glycerol (AOCS, 1989). Potassium was measured by Atomic Absorption Spectrophotometer by the Analytical Sciences Laboratory, University of Idaho. Other fuel parameters were measured according to methods recommended in ASAE Standard EP552 (ASAE, 2000) by the analytical laboratory in the Department of Biological and Agricultural Engineering at the University of Idaho. Only process sensitive parameters are reported here. Complete fuel properties have been reported in several earlier articles, see for example Peterson et al. (1997).

**Results and Discussion**

The first trials were conducted with the CF–1 configuration as previously described. The retention reactor was insufficient in size for complete reaction, thus the CF–2 configuration was designed, built, and tested. Data and discussion will be provided for both systems.

**Tests with the CF–1 Configuration**

Tests with the CF–1 configuration produced some biodiesel; however, free and total glycerol were excessively high. It was found that the centrifuge size limited flow rates and that the retention reactor was too short. Using a glycerol–settling tank to recover the ester worked better in this trial. However it required considerably more feedstock.
to begin the process and required more labor for clean up when the system was shut down for prolonged periods.

**Tests with the CF–2 Configuration**

Three tests were performed (called run 1, run 2, and run 3), each approximately 4 hours long. Run 1 was with a retention reactor with a vertical configuration, Runs 2 and 3 were with the longer “ladder type” retention reactor as shown in figure 5. Run 3 was essentially a continuation of Run 2 on successive days. Run 2 lasted approximately 3 hours on an afternoon. To begin Run 2 the system was shut down and reinitiated the next morning without flushing the system. It was determined that biodiesel production and quality were equal to runs performed after total cleaning and flushing. Only slight adjustments were made to water injection flow–rates due to uneven glycerol settling. Once the ester/glycerol from the previous day’s production had exited, all settings constant with only slight monitoring.

**Biodiesel Quality**

Free and total glycerol tests [total, free, and combined glycerol (Iodometric–Periodic Acid Method, AOCS Ca 14–56)] were performed on the resulting biodiesel produced. The results for the three test runs are shown in table 2. Analysis of the biodiesel for the best run showed it contained 0.016% free glycerol and 0.236 total glycerol, both of which are within ASTM PS121–99 specification for biodiesel. Potassium analysis provided a means to determine the amount of catalyst remaining in the ester phase. Current batch methods used in our department, using ethanol, have not been found to always meet ASTM quality standards for free and total glycerol without additional processing (Lowe et al., 1998).

Even though the pumps can be set to specific flow rates, it would improve the operation of the system if flow meters and computer controls were added. These improvements would allow the operator to better monitor the process and to have the capability of making informed adjustments to feedstock flow rates during operation.

A second consideration is the amount of ester remaining in the glycerol phase. The ester in the glycerol is easily recovered because the ester will be the top layer and can be recycled through the centrifuge. It was observed that the ester in the glycerol phase was very small but no specific measurements were made.

**Cleanup and Maintenance**

The cleanup process for the retention network worked well. The valve at the bottom of the network at the pipe junction allowed flow to continue from the network after the oil source was removed. The tube at the top exit of the network was removed and attached at the lower valve. Once the lower valve was opened, gravity forced the remaining reaction mixture out the tube and on to the rest of the process. Overall, cleanup took about 1.5 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 built 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.

A second problem was ethanol vapor. As the centrifuge separated the ester from the glycerol, the residual alcohol vaporized into the laboratory. The alcohol fumes were very irritating to those working in the area and potentially hazardous. To eliminate the fumes, the experiment was moved to a well–ventilated area. Future testing with the system will require an alcohol vapor capture system.

At a flow rate of 0.38 L/min or 22.8 L/h, the continuous flow system would produce as much biodiesel in 41.5 hours as one batch from an existing 945 L per batch system. Ordinarily, the batch system produces one batch per week (operation is during working hours, but non–working time is required for settling) so the small, pilot scale, continuous flow system would increase present production capacity by three fold if operated 24 h/day.

**Continuous Flow Ester Plant Capital Costs**

The total cost of the continuous flow ester plant as configured for these tests was determined to be $17,187. As shown in table 3, the major costs were for the centrifuge ($14,790), the ethanol and catalyst FMI metering pump ($915), the oil flow regulating pump and speed control ($604), the centrifugal mixing pump ($300), and the static mixers ($78). The remainder of the components were relatively inexpensive and were available locally. No estimate of the cost of construction and operation of a commercial plant was made.

| Table 2. 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 (mm²/s) | 1.9–6.0 | 5.81 | 5.9 | 6.0 |
| Potassium (ppm) | 170 | 16 | 27 |
| Ethanol (%) | 0.12 | 0.09 | 0.09 |

**Table 3. Estimated capital cost of equipment for constructing continuous flow system CF–2 as tested**, [a]

<table>
<thead>
<tr>
<th>Item</th>
<th>Cost ($)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Centrifuge</td>
<td>14,790</td>
</tr>
<tr>
<td>FMI ethanol/catalyst metering pump</td>
<td>915</td>
</tr>
<tr>
<td>Oil flow regulating pump and speed control</td>
<td>604</td>
</tr>
<tr>
<td>Centrifugal mixing pump</td>
<td>300</td>
</tr>
<tr>
<td>Static mixers</td>
<td>78</td>
</tr>
<tr>
<td>Miscellaneous</td>
<td>500</td>
</tr>
<tr>
<td>Total</td>
<td>17,187</td>
</tr>
</tbody>
</table>

[a] No labor charges are included.
CONCLUSIONS
The continuous flow reactor successfully produced ester from canola oil during one run. The glycerol content of the ester, as shown by results of one test, was potentially within the limits of the ASTM PS121–99 (ASTM, 2001) standard specification for biodiesel. It is anticipated that refinements to the system, such as computer controls and optimization of feedstock flow rates, could further improve the final product. Production of biodiesel at a rate three times the existing batch system as currently operated would result from 24-hour operation of this continuous flow system. Capital cost of the system was reasonable with the major expense being the costs of the centrifuge and metering pumps.

RECOMMENDATIONS
Additional testing of the continuous flow reactor should include studies of the following:
- alcohol vapors coming from the centrifuge should be recovered to reduce cost, improve safety, and reduce the negative environmental impact;
- a computer monitoring and control system for sensing flow of oil and control of the alcohol/catalyst mixture injection rate;
- further studies on ester flow rate and centrifuge adjustments on ester purification;
- a method for recovering the excess ethanol from the ester and glycerol phases and an apparatus to capture the ethanol vapor leaving the centrifuge; and,
- secondary treatment including evaluation of a second centrifuge step for further purification.

operation of the continuous flow system over a longer time to assess product quality variations over the duration of a long run.

- development of a real time assay to monitor the quality of the product and to provide for real time flow rate modifications as required to maintain high quality.

ACKNOWLEDGEMENTS
The authors express appreciation to the Idaho Department of Water Resources Energy Division for sponsoring the development of the continuous flow reactor for biodiesel production. Project manager was John Crockett, Bioenergy Specialist. Advice and assistance was also obtained from Craig Chase, consultant and Jeffrey James, manager, Department of Energy, Office of Transportation Technologies, Pacific Northwest and Alaska Regional Bioenergy Program.

REFERENCES