

Purpose:

To evaluate the effectiveness of separating biodiesel from glycerin using a high voltage/low amperage current in a continuous flow system.

Background:

As biodiesel production technology changes, all aspects of the process are being tested and refined for faster, easier, and more efficient production. One such piece of the production process is separating crude biodiesel glycerin from biodiesel. Using gravity settling is the most often used method for separating glycerin and biodiesel. Effective and easy, using gravity is not a good solution for production facilities looking to move to a continuous flow setup. Centrifuges are faster than gravity, and can be used in a continuous flow design. However, centrifuges are expensive both upfront and in the power they consume to run and maintain. Ultimately, other methods need to be found to make separation easy, fast, and less time consuming.

The idea for using a high voltage current to separate biodiesel from glycerin came from Graham Laming, a well known biodiesel enthusiast from the UK. Companies also exist who use electrostatic charge to separate insoluble liquids, but that technology is propriety and made mostly for the oil filtration industry. Graham's simple table top experiments showed that passing a high voltage (around 1000V) low amperage current through a recently-reacted biodiesel glycerin mix worked to separate the layers almost completely in the span of about 30 seconds. Others used this idea on slightly larger scales in their backyards with success, though there has not yet been a good, continuous design utilizing this technique which is both safe and effective. Also, almost no hard data is publicly available describing the effectiveness of this process.

Experiment Design and Setup:

The first set of tests were to determine the the effectiveness of a high voltage current on glycerin separation in a batch setting and to evaluate container size, placement and design of electrodes, and electrode distance. The second set of tests evaluated the effectiveness of using high voltage current in a continuous flow process.

1.1 – see “Batch separation using 100ml graduated cylinder – point to point” -

This test is to confirm the tests done by Graham Laming. 100ml of freshly reacted biodiesel/glycerin mix was poured into a 100ml graduated cylinder. Wires were connected to a transformer which at 110 amps produced 7500V of AC current. One wire was placed near the bottom of the container, and the other near the top, both completely submerged. The tips of the wires were stripped of insulation.

1.2 – see “Batch separation using 100ml graduated cylinder – line to line” -

This test is to determine if separation will occur faster by having the exposed wire running the full length of the liquid with both electrodes parallel and equidistant to each other. Wires were connected to a transformer which at 110 amps produced 7500V of AC current. The wires from the transformer were connected to thick gauge copper wires which were then placed on either side of the container lengthwise, equidistant to each other.

1.3 – see “Batch separation using 100ml graduated cylinder – plate to plate, wire mesh” -

This test was to determine if separation would occur faster using plates or mesh for the electrodes. Wires were connected to a transformer which at 110 amps produced 7500V of AC current. The wires from the transformer were connected to carbon steel wire mesh placed on either side of the container lengthwise, equidistant to each other.

2.1 – see “ Continuous flow separation – point to point” -

This test used the successful results from tests 1.1 – 1.3 and applied them to a continuous flow design. The system was connected to a 4000 gallon settle tank which receives reacted biodiesel/glycerin mix at about 120 degrees (made from chicken fat using potassium hydroxide catalyst). After making a 2000 gallon batch, the mix was sent to the settle tank and left to recirculate. The separation apparatus was connected to the sample port on the 4000 gallon tank, and the test occurred with the tank recirculating to ensure the mix was homogeneous.

The separation apparatus has a 2" PVC line input with 2 electrodes, in the form of spark plugs, inserted into schedule 80 PVC couplings (one could also use schedule 80 PVC for the entire piece of 2" input line). The flow then passed through the settle chamber, a custom made 12"x12"x36" plastic container. The output stream from the two output lines, the 2" biodiesel output on top and 1" glycerin output on the bottom, were regulated by adjusting the valve on the 1" glycerin output line.

Before turning on the current, the 2" input line was filled completely with biodiesel/glycerin mix to order to completely submerge the electrodes. After starting the current 20 minutes passed before taking any samples.

Tests were run at two different times, both using biodiesel made from chicken fat and using potassium methylate as the catalyst. However, the first test (test 1) had a prewash (5% water) while the second did not.

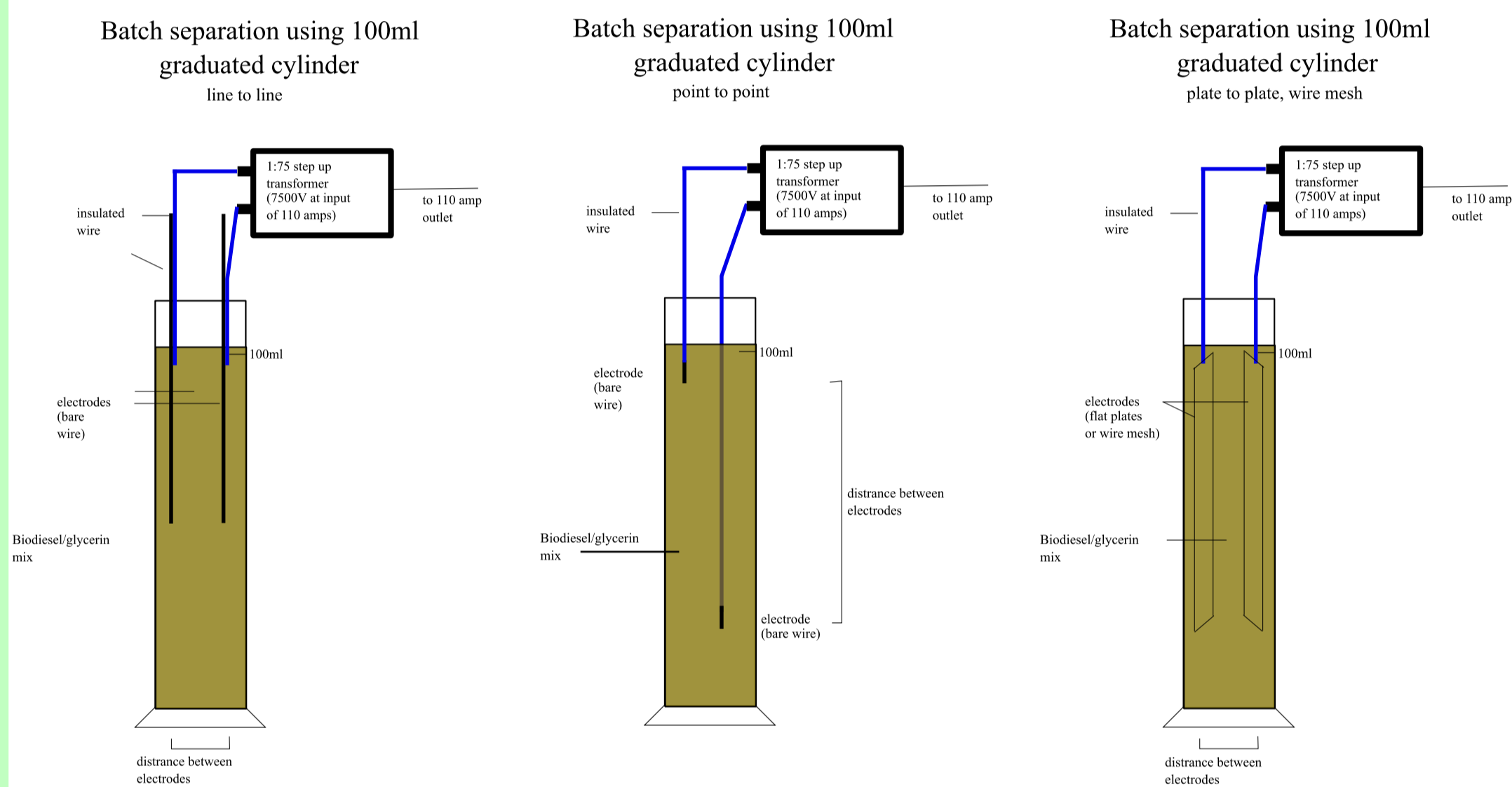
Glycerin amount was determined by letting samples of biodiesel taken from the output stream gravity settle for 9 days, and then measuring the amount of extra glycerin that settled out.

2.2 – see “ Continuous flow separation – point to point” -

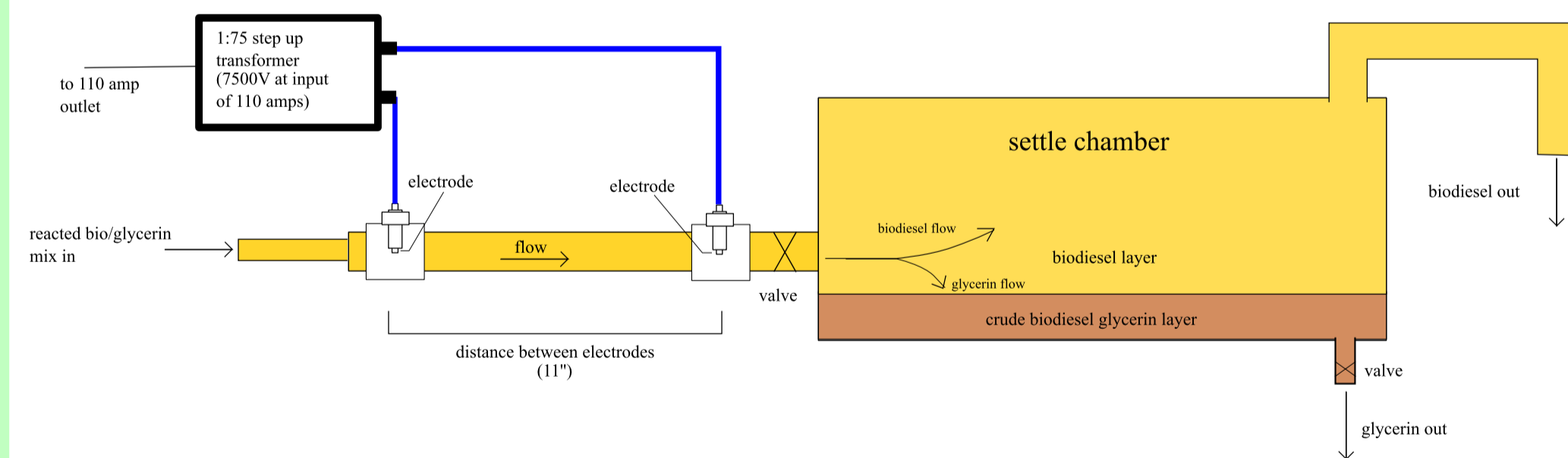
This test was set up exactly like test 2.1, but the input stream came from a continuous flow cavitation reactor instead of the settle tank. Additionally, the feedstock used to make the biodiesel was waste vegetable oil instead of chicken fat. The cavitation reactor was set to flow at near 6L/m, which was the slowest speed possible as a result of its design of the reactor, and the biodiesel/glycerin mix came in at around room temperature.

Using high voltage current for continuous separation of glycerin from biodiesel

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Continuous Flow Separation point to point



Brief Explanation of Process

The incoming biodiesel/glycerin mix passes through the 2" PVC with the electrodes (spark plugs) placed in them 11" apart. It has about 30 seconds of residency time between the electrodes before passing on into the settle chamber. Glycerin "clumping" occurs primarily in the 2" PVC between the electrodes. As it enters the settle chamber, the now large glycerin bubbles are heavy enough to fall out of solution. Residency time in the settle chamber (12"x12"x36") at 4L per minute is approximately 30 minutes. By the end of the chamber, most of the glycerin has settle and exits through the glycerin out 1" outlet, while biodiesel is pushed out the top 2" port. Flow is regulated by adjusting the glycerin out valve (which should be set to approximately 22%, based on expected amount of glycerin). Free glycerin in the outgoing biodiesel stream ranged from .25% to 1.25% glycerin depending on flow rate.

Results:

1.1 – see “Batch separation using 100ml graduated cylinder – point to point” -

The point to point method was most effective at settling glycerin quickly. Within seconds of running current through the mixture at 7500V the glycerin begins to “clump” into pin-sized balls. Those clumps were then heavy enough to drop quickly through the biodiesel and settle on the bottom of the container. The electrodes tended to buzz slightly, and if left in too long would bubble (probably heating the liquid around them to the point of boiling off the methanol in solution). Also, because the conductivity of glycerin is so much higher than that of biodiesel, if the electrode was placed too close to the glycerin layer it arced into the glycerin.

Complete settling occurred within 30 seconds when the electrodes were placed 9" apart. Testing with other, larger containers showed that electrode separation greater than 12" resulted in a noticeable increase in settle time, and that a container width greater than 4" also resulted in significant increases in the separation time.

1.2 - see “Batch separation using 100ml graduated cylinder – line to line” -

The line to line method was ineffective at settling glycerin quickly. Though settling occurred faster than gravity settling, it was still very slow with complete settling occurring in the span of minutes, not seconds. After repeat trials, this method was abandoned.

1.3 - see “Batch separation using 100ml graduated cylinder – plate to plate – wire mesh” -

The plate to plate method was ineffective at settling glycerin quickly. Though settling occurred faster than gravity settling, it was still very slow with complete settling occurring in the span of minutes, not seconds. After repeat trials, this method was abandoned.

2.1 - see “ Continuous flow separation – point to point” -

The parameters for the design of the continuous flow apparatus were taken from the results of tests 1.1 – 1.3. The electrode distance of 11" in a 2" diameter container were shown to be effective at clumping glycerin almost immediately and dropping it within 30 seconds. A flow rate of 5L/m was estimated in order to give the liquid 30 seconds of residency time between the electrodes. In addition, it was clear from the initial set of tests that after glycerin clumping all that was needed was time for the clumps to fall out of solution. During this settling phase, no current was needed. As a result, the design incorporated a settle chamber which slowed the flow rate enough to allow the glycerin to fall to the bottom. The results are recorded in the graph below.

note: samples tested at a given flow rate were sample at least 10 minutes after that flow rate was set. 10 minutes is probably not adequate time to get a really accurate number from a change in flow rate (i.e., the liquid that passed through the electrodes will probably take more than 10 minutes to reach the end of the settle chamber where they are sampled). Therefore, this graph can be viewed as changes in % glycerin while increasing flow rates generally, though perhaps not exactly specific to the flow rates associated with them.

2.2 - see “ Continuous flow separation – point to point” -

The high voltage had no effect on the income stream of biodiesel glycerin mix. There was no noticeable clumping, and the stream remained milky colored throughout the 2" input pipe and did not effectively settle through the settle chamber.

Discussion:

Positive Results:

From this experiment it is clear that using high voltage, in this case 7500 volts AC at about 20 milliamps, significantly affects glycerin drop out from biodiesel by causing sudden clumping of the glycerin. In addition, the continuous flow system described here is effective at causing similar clumping and can remove 99.7% of glycerin at flow rates of 2 – 4L/m, and around 99% glycerin at flow rates of 5 – 6L/m.

Concerns:

The benefits of a continuous flow separation device seem greatest when using a continuous flow reactor, and the separation apparatus was ineffective when used in conjunction with our cavitation reactor. One possible explanation of the poor performance of the separation apparatus when used with the cavitation reactor is that the cavitation reactor emulsifies the methoxide and fat/oil stream much more than a traditional batch reactor. This extreme emulsion may inhibit the separation effects that we see when using a biodiesel glycerin stream from a batch reaction. More testing is required on this issue.

Safety is also a concern. The system must be designed around safety because of the high voltages required. An especially disconcerting issue is the bubbling which occurs around the electrodes. Those bubbles can build up in the 2" input pipe and could even build up around the electrodes which is an explosion hazard. In addition, because the current is passing through the biodiesel stream, the entire stream up and down the line is charged, which means arcing could occur anywhere along that stream. Finally, it is possible that passing through the current increases polymerization of the biodiesel or has other quality effects not measured in this test. All these concerns must be addressed before implementing a system like this.

Future Testing:

All of the listed concerns need to be tested – from creating a safe design, to testing the output stream for the full spectrum of ASTM specs (especially cold flow and viscosity, which might indicate polymerization). Varying voltage and correlating that to electrode distance, and testing the system on a wider variety of types of biodiesel (from different feedstocks) would be useful in understanding the depth of applicability of this technology. Finally, achieving successful separation with a continuous flow reactor would be an important step towards a simple, fully continuous plant design.