THE USE OF ULTRASONIC REACTORS IN A SMALL SCALE CONTINUOUS BIODIESEL PROCESS

Graham Towerton

G&M Global Enterprises Inc., 1207 NW 1st Street, Amarillo TX 79107, U.S.A, www.customchempack.com

EXECUTIVE SUMMARY

Two 1 kW ultrasonic reactors UIP1000hd were obtained from Hielscher USA (Ringwood, NJ, USA, www.hielscher.com) for use in converting a 500 gallon batch biodiesel process to a continuous process with a proposed design flow rate of 1-2 gpm. The reactors were installed in a privately designed and owned biodiesel plant using PBSY cottonseed oil.
The reactors were installed in a two-stage process and evaluated for operational performance for a period of two months. Changes in operational parameters were tested to determine performance impacts including vegetable oil flow rate, methanol to oil ratio, catalyst to oil ratio, reactor inlet temperature and reactor pressure.
The study of these reactors provided the facility operators with the following conclusions:

- The use of Hielscher's ultrasonic reactor technology is a highly cost-effective means of converting a batch biodiesel reaction process to a continuous process.
- High quality biodiesel can be produced on a continuous basis with greatly reduced operating costs in terms of methanol and catalyst usage
- The ultrasonic reactors provide significant operational flexibility improved recovery times from operational upsets caused in other areas of the process
- Key operating parameters may be easily and quickly adjusted to optimize process performance with the aim of continuous control of biodiesel quality

1. PROCESS DESCRIPTION

The Hielscher ultrasonic reactors obtained were two 1kW UIP1000hd models used for the purposes of evaluating operational performance with the aim of determining key operating parameters for later plant capacity expansion.
The reactors were installed according to the following basic process design (Fig. 1).
Each stage of reaction and separation equipment consisted of the following key equipment items:

- An oil storage tank with a vegetable oil circulation pump. The vegetable oil is circulated through a heater to maintain a temperature of 150-160 deg F at the reactor inlet temperature (prior to catalyst and methanol injection. A hot water boiler provided the heating medium for the vegetable oil heaters.
- The Hielscher 1 kW reactor. Oil flowrate to the reactor was controlled by feeding a slipstream off the circulating loop to the reactor. Pressure at the reactor inlet was typically controlled at 13-15 psi. A recirculation line (not shown) was also provided from the reactor outlet to the vegetable oil tank to allow recirculation of the reactor product until suitable reaction conditions were achieved at each stage of the process during start-up of the process. Reactor product sample points were installed at the inlet to each settler.
- A methanol and catalyst injection pump. Electric diaphragm pumps were used to control chemical injection rates.
- A settling tank to receive the mixed reactor product sized to allow up to 2 hours residence time in the settling tank at maximum plant capacity. Glycerin is periodically drawn by gravity into a collection tank from each settling tank while biodiesel from the settler top outlet was transferred to the next stage of processing.

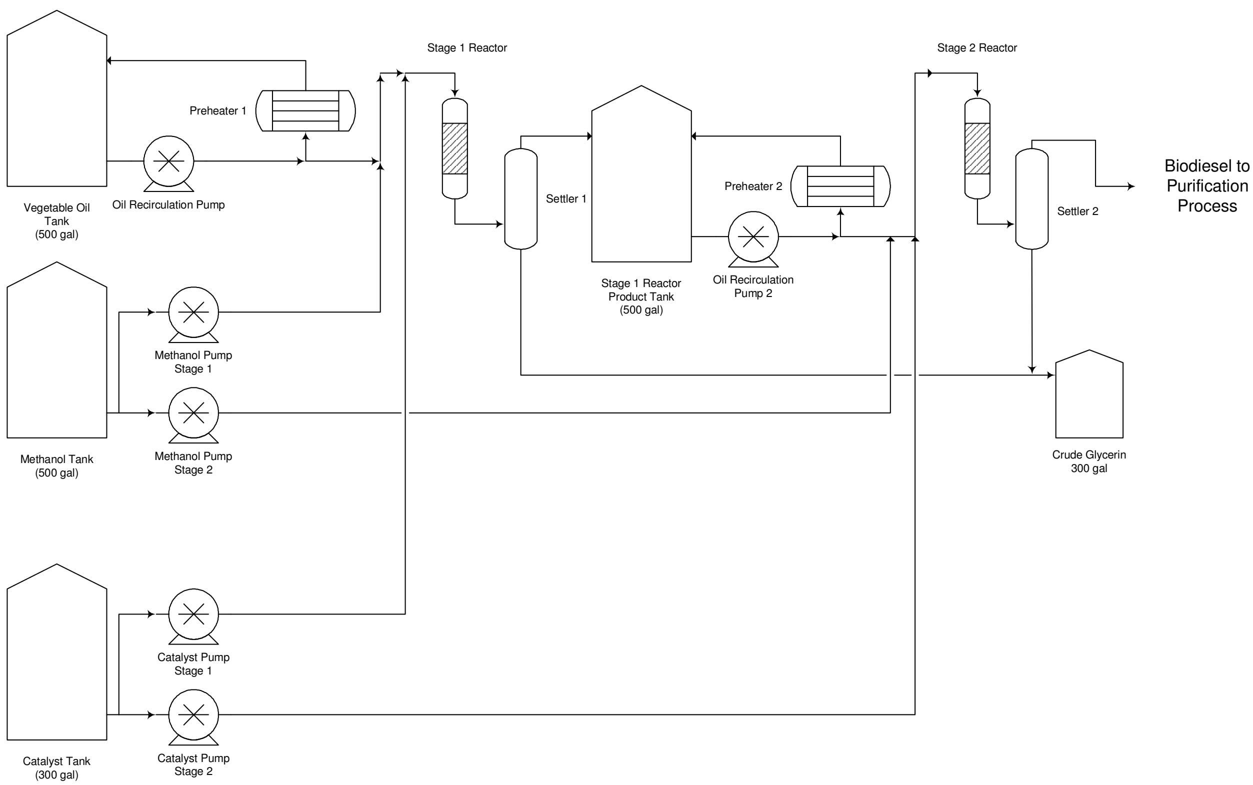

Figure 1 - Process Flow Diagram – 2-stage biodiesel process

2. PROCESS OPERATION

The process was operated each day for a period of 8-12 hours while performing evaluation of the reactors. At the start of each day the sequence of start-up was as follows:

1. Commence circulation and heating of the vegetable oil (Stage 1) and biodiesel (Stage 2) through the heaters and reactors until the process achieved target temperatures.
2. Commence injection of methanol
3. Commence injection of catalyst
4. Start the reactor controller and activate the reactor sonotrode
5. Discontinue circulation of reactor product to the vegetable oil tank and direct the reactor product to the settling vessel (typically 30-60 seconds after reactor start-up)
6. Sample the reactor product and observe glycerin fall-out time, glycerin yield after 30 minutes and glycerin color.
7. Adjust operating parameters as needed for optimizing reactor yield and product quality.

Variables to be adjusted included methanol and catalyst injection rates, reactor pressure and vegetable oil flow rate. Reactor sonotrode intensity was not adjusted once the correct sonotrode configuration was achieved to provide optimum power. The first stage reactor typically operated at 400-600 W and the second stage at 350-500W depending on flowrate of oil.

80% of catalyst and methanol requirements were injected into the first stage reactor, while the remaining 20% of catalyst and methanol requirements were injected to the second stage.

Flowrate of oil was typically controlled at 1.5-2.0 gpm on each stage. Periodically, fresh oil would be transferred from the main holding tank in order to maintain oil levels in the first vegetable oil tank to Stage 1.

3. KEY OBSERVATIONS AND BENEFITS

Upon converting from batch reaction (with no Hielscher reactors used) to continuous reaction with the Hielscher reactors, the following significant changes were observed:

- Methanol usage was dramatically decreased. In batch mode the process typically used a 100% excess of methanol compared to the stoichiometric requirements. In continuous mode, as low as 30% excess was required, but typically 40% excess was used to maintain a good conversion. The reductions in methanol were such that injection of methanol was almost discontinued at the second stage where catalyst only was injected to force the reaction to completion. Methanol carryover in the first stage biodiesel (both dissolved in biodiesel and entrained with glycerin) allowed sufficient methanol for the second stage reaction to proceed.
- Glycerin drop-out was significantly quicker due to lower amounts of methanol usage reducing the tendency to emulsify glycerin in the biodiesel. Typically 90% of the glycerin would drop out in less than 15 minutes, while 95-98% of the glycerin would fall out in under 30 minutes.
- Catalyst usage was decreased by approximately 25-30% compared to batch operation. We believe that this was achievable due to increased mixing caused by the Hielscher reactors which greatly increased the reaction kinetics allowing reductions in catalyst requirements.
- Ideal reaction pressure was typically 13-15 psi in each stage. Higher or lower pressures tended to cause incomplete reactions and improper glycerin drop-out due to higher levels of unreacted methanol and vegetable oil in the reactor product.
- The second stage reactor could be operated at a much higher flowrate (4 gpm) than the first stage (1.5 to 2 gpm). The second stage flow could be safely increased on a continuous basis without exceeding the power capacity of the ultrasound reactor controller. This was clearly due to the majority of the reaction taking place in the first stage, placing less power demand on the second stage. Future designs of the plant could incorporate a second parallel reactor at the first stage allowing a doubling of the plant capacity for very low incremental capital cost.
- Downstream biodiesel purification operations (water wash, centrifuge and resin polishing) all performed at significantly improved levels due to better glycerin removal, less excess methanol and less excess catalyst in the biodiesel. Yields of soap by-products at the water wash stage were greatly reduced as a result of better reaction conversion.
- Process upsets could be very quickly corrected. After a few weeks of using the Hielscher reactors, operators could quickly judge reactor performance by listening to changes in the sound of the reactor sonotrodes as well as observing glycerin drop out

in test samples. Changes in sonotrode power and sound levels would typically be indicative of methanol or catalyst pump failure (causing loss of reaction) and changes in glycerin drop-out would typically indicate excess or deficiency of methanol and/or catalyst. Upsets in process performance could be quickly corrected in 2-3 minutes minimizing off-spec product to the downstream processes.

- Offspec product could be easily recycled from the back end purification system into the inlet of the second stage reactor. Several months' worth of off-spec product from failed batch reactions could be quickly reprocessed in the second stage while also processing fresh oil from the first stage. This allowed a significant recovery of working capital tied up off-spec product storage.

4. CONCLUSIONS

The owners and operators of the plant were pleased with the overall performance of the Hielscher ultrasonic reactors due to the significant increase in plant capacity, product quality and yield improvements achieved. The cost of the Hielscher reactors and the conversion from batch to continuous operation was achieved for incremental capital costs of just 5% compared to the original investment in plant equipment, with estimated operating cost reductions of 3-5% and capacity improvement from 500 (batch) to 2800 gallons per day (continuous).