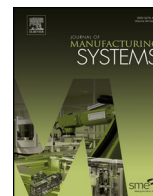




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A cellular manufacturing process for a full-scale biodiesel microreactor

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ABSTRACT

A cellular manufacturing process was developed for fabrication and assembly of a full-scale biodiesel microreactor capable of producing biodiesel fuel at the rate of 2.47 L/min and at a capacity of over 1.2 million liters of fuel per year. The scale-up of the microreactor was done through fabrication of over 14,000 individual microchannel laminae, and assembly of these laminae into a hierarchical system of modules and manifolds, thus duplicating many times over the intensification of the reaction rate per reactor volume that occurs in a single microchannel lamina. The work describes the design of the microreactor, the production process to fabricate and assemble the microreactor, the design of the manufacturing cell, and the testing of the microreactor to verify its performance.

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1. Introduction

In recent years, the synthesis of biodiesel fuel by transesterification of vegetable oil with methanol in the presence of a homogeneous base catalyst has been experimentally studied in different types of microreactors including capillary reactors and microchannels [1–5]. Through this research, it has been shown that the large surface-to-volume ratio offered by microchannel geometries can intensify mass and heat transfer. For mass transfer limited reactions like transesterification, this intensification serves to increase the reaction rate and thus increase the rate of product synthesis per reactor volume. For the production of biodiesel by transesterification, use of a microreactor can result in higher chemical reaction rates than what has been observed through conventional biodiesel batch reactor technology [6], able to reduce residence time from hours to minutes [1–5].

When used for biodiesel production, a microreactor has several advantages over a batch process including the ability to produce

a greater amount of this alternative fuel in a shorter period of time per reactor volume, the associated reduction in labor costs through higher throughput, the ability to dramatically reduce the size of a production biodiesel reactor, mild pressure and temperature operating conditions, low energy usage, and low capital costs [7].

These advantages create the potential for distributed small-scale production of biodiesel fuel, rather than large-scale centralized production. Through the reduced footprint of the entire production system made possible through the microreactor, the portable biodiesel system could potentially be mounted on a vehicle and taken to fields where oil-producing feedstocks exist, rather than transporting such feedstock to a large central production facility, incurring transportation cost and risking the loss of oil yield due to inevitable drying of the feedstock oils while in transit.

However, in reviewing the published research in microreactors for biodiesel production, no researcher has reported the development of the microreactor technology beyond a proof-of-principal test, which is limited to low volume flow rates only. The work does not appear to have been developed beyond laboratory bench top scale, with the greatest amount of reported production rate limited to a microreactor-based system described by Jovanovic [8] producing 17 L/day.

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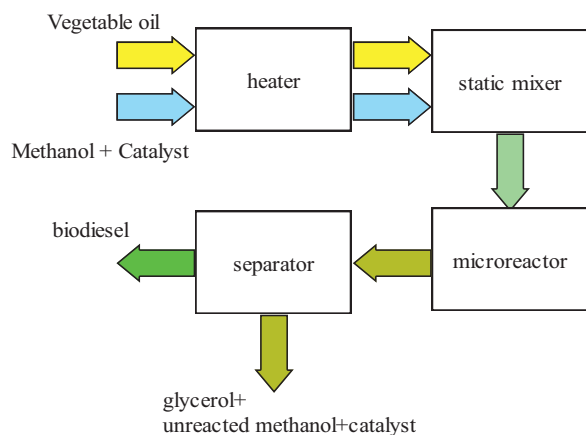


Fig. 1. Continuous biodiesel synthesis process using a microreactor.

2. Purpose of research

This research describes the cellular manufacturing process that was developed and used for fabrication and assembly of a full-scale biodiesel microreactor capable of producing fuel at the rate of 1.9 L/min and at a capacity of 950,000 L per year. The work describes the scale-up of the microreactor to such capacity through a numbering-up or replication of individual microreactor microchannel laminae into subassemblies and assemblies, thus duplicating many times over the fast chemical reaction rates observed through an individual microchannel lamina. The work includes the design of the microreactor, the production process to fabricate and assemble the microreactor, a description of the manufacturing cell that

was developed, and the testing of the microreactor to verify its performance.

3. Overview of microreactor biodiesel processing

Biodiesel is typically produced using the transesterification reaction that involves mixing triglycerides (TG), such as vegetable oils, with methanol (MeOH) and a mineral base powder. The mineral base, such as potassium hydroxide (KOH) or sodium hydroxide (NaOH), acts as a catalyst and in powder form it readily dissolves in the MeOH in low concentrations. The use of a base catalyst allows the reaction to proceed at atmospheric pressure even at room temperature, though temperatures up to 70 °C are used to accelerate the reaction. The reaction is reversible, so MeOH amounts beyond the required stoichiometric molar value are used. The MeOH: TG molar ratios ranging from 6:1 to 10:1 are typically used [6].

The mild operating conditions for this reaction make it easy to perform in large tanks in a batch fashion. Agitation in the form of stirring is usually employed in these batch systems to enhance the contact between the immiscible oil and alcohol reactants. In large tanks only slow stirring is practical so the time required to reach a complete reaction could be several hours [6]. Since production capacity depends on the number and size of tanks, a high capacity plant could require significant capital and land investment.

The microreactor system described in this paper aims to dramatically improve production capacity per reactor volume by decreasing the required reaction time from hours to minutes. The process is continuous, which allows for more consistent product quality compared to the batch process. The microreactor based process is illustrated in Fig. 1.

The reactants are first heated to the desired reaction temperature and are passed to a static mixer. The mixer contains packing that aids in forming an emulsion of the reactant streams. The

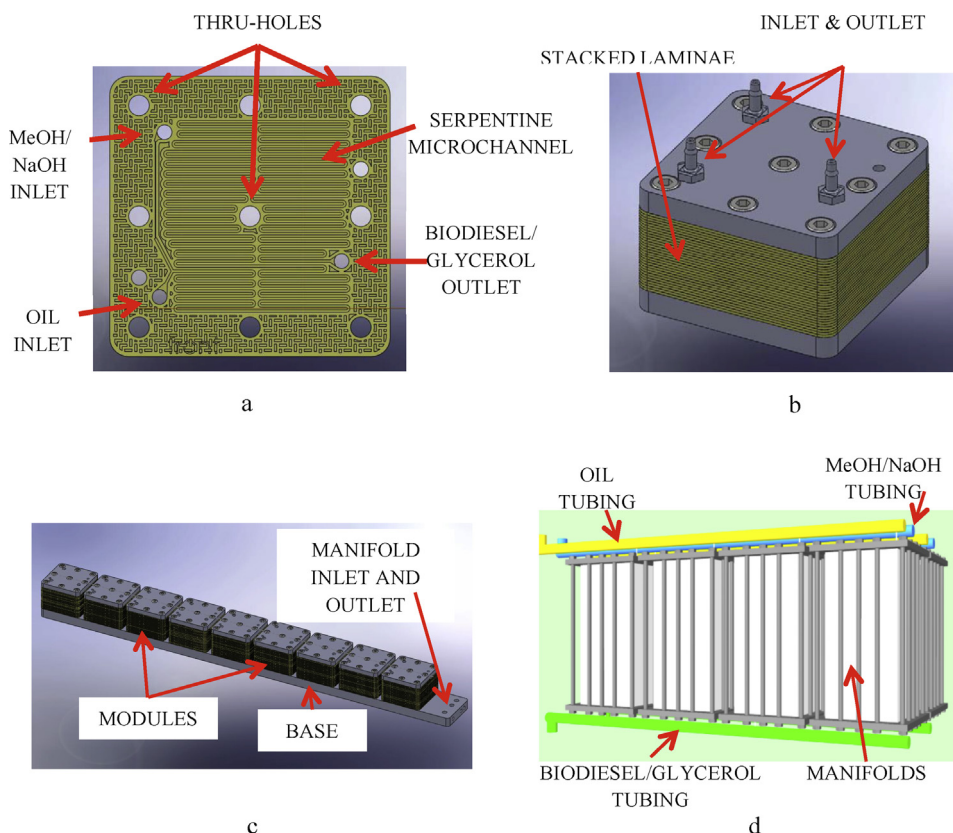


Fig. 2. Modular microreactor design: (a) lamina; (b) module; (c) manifold; and (d) full-scale microreactor.

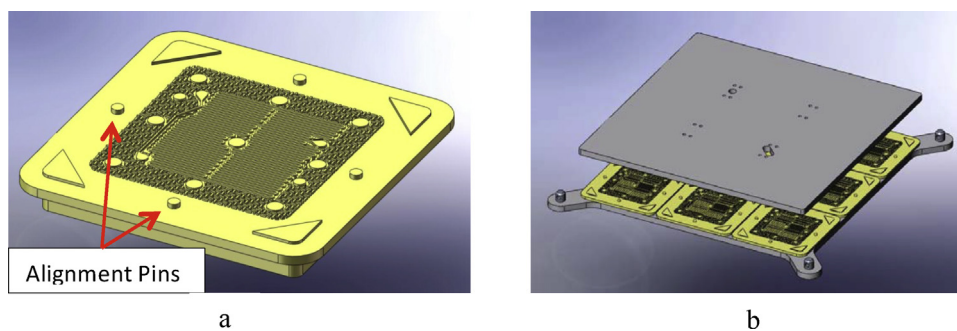


Fig. 3. (a) Lamina die and (b) die fixture assembly.

emulsion is then directed into the microreactor unit. The microreactor unit contains thousands of microchannels that organize the bulk flow into parallel streams of alternating reactant slugs that are laminar [1–4]. This flow pattern intensifies mass transfer due to the high surface area to volume ratio of the slugs and essentially accelerates the reaction [5].

The product stream exiting the microreactor is an emulsion of biodiesel and glycerol. There will also be unreacted MeOH and catalyst being present in the glycerol phase. The biodiesel and glycerol phases are immiscible and readily separate by gravity due to the large difference in the specific gravities.

Though not shown in Fig. 1, additional downstream processing might be performed on the products. The glycerol phase could be processed to remove the MeOH for recycling and to purify the glycerol for sale as a co-product. The biodiesel could be washed with water to remove residual glycerol and fatty acid salts (soaps).

4. Microreactor design

The full-scale microreactor was designed to be modular as illustrated in Fig. 2. First, individual microreactor laminae were fabricated (Fig. 2a); stacks of laminae were then assembled into modules (Fig. 2b); modules were assembled into manifolds (Fig. 2c); and manifolds then assembled into the final full-scale microreactor (Fig. 2d). The research team designed and fabricated the items in Fig. 2a–c, and the industrial research sponsor was responsible for developing the final full-scale microreactor unit.

The representative design for a single microreactor lamina is shown in Fig. 2a. It consists of inlets for the oil and MeOH/NaOH process reactants; microchannels where the reaction takes place, and an outlet for the resultant biodiesel and its glycerol byproduct. Other features on the lamina are thru-holes for later assembly of the laminae into modules and manifolds. The lamina had 0.76 mm thickness and 5.7 cm × 5.7 cm planar dimensions. The

microchannel array in the lamina had a serpentine flow pattern, the length of which was designed to correspond with the flow rate and residence time of the biodiesel process to ensure a complete conversion of the reactants to biodiesel fuel. The microchannel had a 500 μm × 500 μm depth and width, respectively. The slug-flow pattern of the biodiesel process allowed for a 99% oil conversion with a short residence time thus allowing larger sized microchannels to be designed for the process [1]. Use of larger microchannels also resulted in low pressure drops compared to other microreactor biodiesel processes that use smaller microchannel dimensions that have been reported [2]. In addition, the larger channel size reduces the chance for fouling and the need for high-resistance filters upstream of the reactor.

For laminae, the material substrate that was selected was required to be chemically inert and low-cost. To meet these criteria, several polymer substrates were considered. Early tests were conducted with polycarbonate, poly(methyl methacrylate) (PMMA), and polysulfone. Polycarbonate and PMMA were both found to be hygroscopic, absorbing and thus expanding after exposure to MeOH and NaOH causing stress cracks at fitting joints fabricated in the substrate. Polysulfone, when exposed to these biodiesel process chemicals, exhibited outgassing which interfered with the reaction process [9]. All of these materials were abandoned in favor of high density polyethylene (HDPE). HDPE had multiple advantages of being highly chemically resistant to the biodiesel process reactants and products, and was inexpensive with material costing only about \$0.02 per lamina. Unfortunately, as will be described later in this paper, HDPE's superior material properties for this process also made it infeasible to directly laminate or fuse together laminae made from this material.

The laminae were stacked and sealed to one another in an assembly referred to as a module. The module was made of 50 laminae sealed between two stainless steel cover plates using nine #8 socket head cap screws. Each module contained 3.2 mm inlet

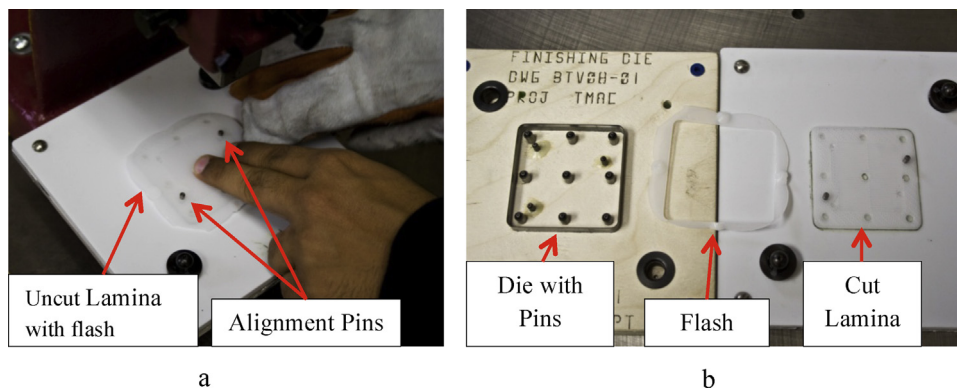


Fig. 4. (a) Bottom die cutter fixture with alignment pins and (b) top die cutter fixture with pins for making thru-holes and removing flash; excess flash; and a resultant cut lamina with thru-holes and inlets.

and outlet for oil and MeOH/NaOH injection, and biodiesel/glycerol emission. The module is shown in Fig. 2b.

A number of modules were placed in parallel on a manifold assembly (Fig. 2c). The manifold was made up of 1.3 cm thick HDPE material with a 1.3 cm aluminum base which served as a support for the modules. A thin layer of polypropylene was placed between the HDPE and the aluminum as a gasket. The underside of the HDPE base had a machined slot (not shown in Fig. 2c) along its length and thru-holes for directing the reactants and products to/from the modules. At one end of the manifold were threaded inlet and outlet holes for delivery of fluids to/from the manifold.

Manifolds were assembled in parallel into a frame representing the full-scale microreactor, and connected to reactant tubing (Fig. 2d). To obtain the production rate of 1.9 L/min of biodiesel fuel, the final full-scale microreactor consisted of 35 manifolds connected in parallel, containing over 14,000 individual laminae.

5. Process design

A manufacturing process was designed to produce the microreactor. There were seven major operations in the manufacturing process which included (1) hot pressing of HDPE blanks to emboss the microchannel features, thus creating the laminae; (2) a cooling operation for the laminae; (3) die cutting of flash from the laminae; (4) pressure testing individual laminae; (5) assembling laminae into modules; (6) assembling modules into manifolds; and (7) leak testing the manifolds. Final assembly of the microreactor given the 35 manifolds that were produced and delivered was carried out by the industrial sponsor of the project.

5.1. Hot pressing

The process begins with HDPE blanks that served as the substrate for the laminae. The blanks were 5.7 cm × 5.7 cm × 0.76 mm, with tolerance of +1.3 mm thickness. An aluminum die fixture assembly was produced to hold the blanks for hot pressing (see Fig. 3). The die fixture assembly consisted of a die fixture, cover plate and nine dies that contained the negative pattern of the microchannel geometry previously shown in Fig. 2a. Each die contained four alignment pins to help center a blank. Individual blanks were loaded into the nine dies and covered with a top plate.

A 147 kN Carver AutoFour™ heated bench top press was used to hot press the lamina pattern onto the HDPE blanks. The die fixture assembly containing the HDPE blanks was loaded into the press. After the press is preheated, the assembly was pressed for 15 s with both the top and bottom plate having temperatures of 185 °C and a force of 9.8 kN.

5.2. Cooling

The die fixture assembly was then placed in a cooling operation. As the HDPE laminae cooled, a hydraulic press was used to minimize deformation of the substrate and features due to thermal stress. Cooling was done in a modified Jack X-Change™ hydraulic press. Modifications included a bottom plate on the press with alignment pins that would accept and register the die fixture assembly. A force of 245 kN (862 kPa of pressure) was applied for 15 min as the assembly cooled.

5.3. Die cutting

After cooling, individual laminae were then removed from the die fixture assembly and sent to die cutting. The die cutting operation was designed to remove excess flash from the lamina and place thru-holes and inlets in the lamina (see Fig. 4). A single lamina was centered on a die with alignment pins. This was done by manually



Fig. 5. Lamina pressure test fixture.

forcing the alignment pins through the material on the part. The die cutter made use of a die with pins corresponding to thru-holes, inlet/outlets and borders. A Tippeman™ 69 kN die cutter was used for this operation.

5.4. Pressure testing

Defects or inconsistencies in the geometry in microchannels on individual lamina due to fabrication variation will impact the performance of the microreactor. A method had to be developed to indirectly compare the internal geometry of the individual laminae.

To achieve this, a mechanism was developed to measure the variation in the pressure required to achieve a set flow rate of fluid inside a single lamina. The inference here was that if two laminae have the same internal geometry then the pressure required at a constant given flow rate will be the same.

The mechanism designed to test a lamina worked on a simple principle of using a source of fluid, a pump, tubing and an interface that was mechanically joined to the lamina to measure the pressure inside the channels. A Cole Parmer™ syringe pump was used with a 30 mL plastic Bic™ syringe. HDPE tubing was connected to the syringe pump, a mass pressure sensor, and an interfacing fixture to hold each lamina. The fixture is illustrated in Fig. 5 in the unloaded position. The mechanism to fixture the lamina to a cover plate used a pneumatic actuator.

To perform a test, a single lamina was placed on alignment pins and slid under the cover plate activating the testing process. Two seconds after the cover plate actuated, sealing a device, hot water at a temperature of 70 °C was pumped through a lamina at a flow rate of 39 mL/h. A pressure measurement was then taken. If microchannel geometry was intact as originally designed, then pressure was expected to be approximately 35 kPa. Higher or lower pressure indicated a flaw in the microchannel geometry. This situation resulted in inspection of the lamina to deduce the defect followed by corrective action. Typical problems seen by the research team included wear or defect in a lamina die, wear of the die cutter, or needed modifications to the heating schedule in the hot press as HDPE material properties typically change by lot.

5.5. Lamina joining and module assembly

Over a 12-month period, much experimentation was done to find an effective technology to join laminae into a module. HDPE is highly chemically resistant. This made it an ideal substrate for withstanding the corrosive biodiesel reactants as the microreactor did not erode or deform over time using this material. However, this same chemical resistance also made it difficult to seal laminae together to form a leak-proof unit with features that retained their original shape [10].

Common techniques for joining individual devices using traditional microreactor polymer materials such as PDMS are unsuitable for HDPE due to differing material properties. For example PDMS and HDPE have Young's modulus (E) of 800 kPa and 800,000 kPa, respectively, and PDMS is a thermoset while HDPE a thermoplastic. Therefore, low pressure, low temperature methods of joining, common for PDMS, are not suitable for HDPE.

Tested technologies to seal laminae included laser sealing, adhesive sealing, hot rolling, hot pressing, planar force pressure sealing, and fastener pressure sealing, most of which were unsuccessful for one reason or another [11]. Other technologies such as ultraviolet curing [12] heat staking [13], and microwave sealing [14] were reviewed but were determined to be inappropriate for the small feature sizes of the current microchannel lamina design or were shown to have success with polymers other than HDPE.

Of the methods tested, high pressure fastener sealing was shown to be robust for HDPE microreactors. This sealing approach relied on bolt fasteners joining two plates on the top and bottom of a stack of laminae. The module was made of a bottom and top stainless steel plate with nine #8 socket head cap screws that passed through the plate and the stack of 50 laminae as shown in Fig. 2b. Washers were used to help distribute the pressure evenly, and Viton® gaskets of 0.51 mm thickness were placed between each lamina. Screws were first placed 50 mm apart and later, through a parameter optimization study, it was found that they needed to be 25 mm apart to obtain even pressure across the module. A screw through the center of the structure was also required to help provide even pressure across the device. Evenly distributed pressure of 35 kPa adequately sealed the module both between individual laminae and between the stack of laminae and the sealing plates.

5.6. Manifold assembly

Eight modules were assembled in parallel on a 71 cm × 5.7 cm × 1.3 cm HDPE base (see Fig. 2c). As stated previously, the base had a slot and thru-holes machined into it to accept the nine bolts used to seat the modules to the manifold. Finally, a bottom 71 cm × 5.7 cm × 1.3 cm aluminum plate was added to the HDPE base to provide strength and stability to the manifold assembly and the bolts were tightened for each module.

5.7. Leak testing

Each manifold was leak tested by having hot water at 70 °C temperature with a flow rate of 1.9 L/min cycled through it. If leaks were found, the bolts were tightened to prevent the modules and the manifold from leaking. Once the manifold had stopped leaking, hot water was run through it for at least 1 h to ensure a reliable seal.

6. Manufacturing cell

A manufacturing work cell was developed for production of the microreactor process described above (see Fig. 6) [15]. The cell consisted of a 3.7 m × 3.7 m area. This area allowed movement of the parts along a U shaped path. The flow of operation moved counter-clockwise from the heated press to the chilling stations, to the die cutter to the manifold assembly at which point the process moved to assembly. Recycling was provided near the die cutter for flash. In front of each cooling station, which must have an air line running behind it, was a heat resistant mat. This mat served as protection for the die assembly fixture and as a work surface. Needed tools were placed in bins near each cooling station. A mat was provided across the work area to reduce fatigue. Two tables were used both of which were height adjustable for ergonomic relief. The heated press was on a standard stand from the supplier but the platens could be adjusted for height to reduce neck and shoulder incidents.

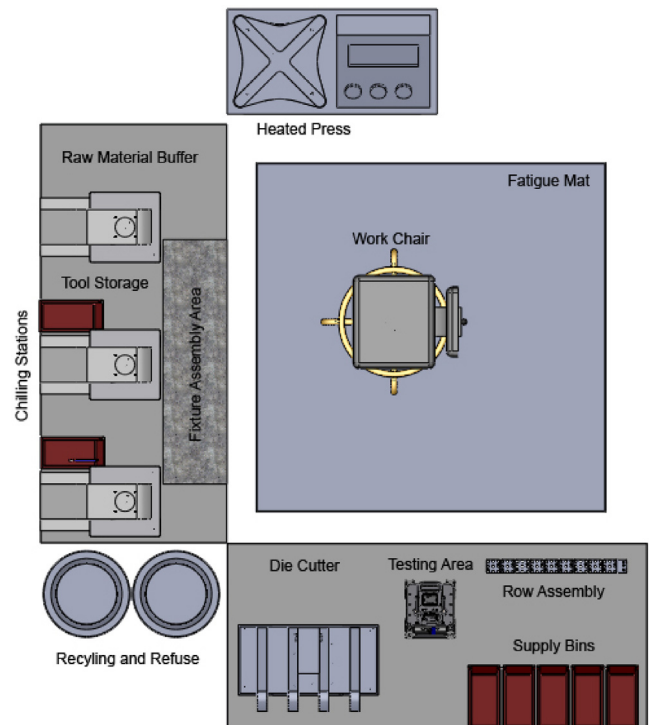


Fig. 6. Cell layout.

Fig. 7 shows a three-dimensional representation of the work cell. Factory controls for input and output were designed as part of the operation of the cell. A CONWIP approach was used to manage the work-in-progress so that it stayed at a balanced level. This was achieved by allowing only parts required for the manifold being worked on to be in the work cell. As a manifold was finished, empty and near-empty bins and staging areas alerted the operator for the need for more HDPE blanks and a manifold kit of materials.

To determine the production schedule needed to produce the 35 manifolds and the 14,000 laminae, the bottleneck operation was

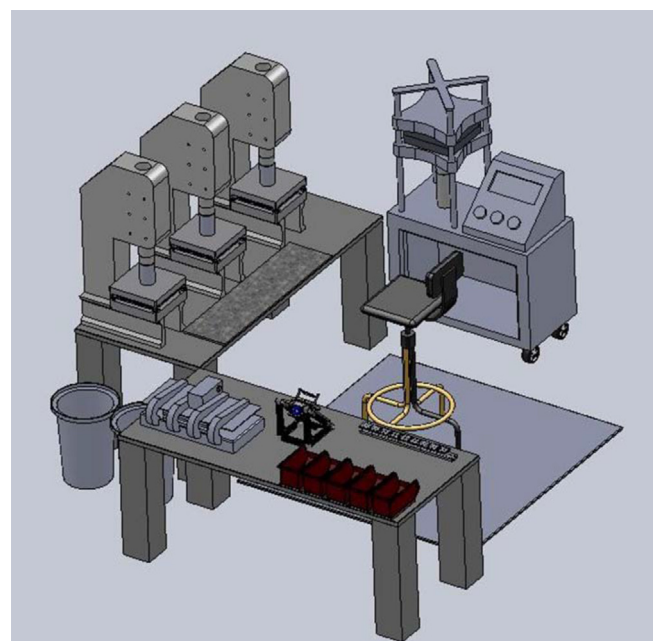


Fig. 7. Three-dimensional view of cell.



Fig. 8. Completed manifolds.

identified in the cell. This was the heated press operation which had a production rate of 1 lamina every 5 min. Using this limiting operation as a basis, three workers were assigned to the cell so as to operate at full capacity rather than the single worker configuration illustrated in Figs. 6 and 7. This assignment of three workers to the cell allowed the team to output one lamina every 5 min. Operations were then setup as two 8-h shifts for 5 days per week. Using this operating scheme, it was calculated that 3.17 months were needed to produce the entire biodiesel processing unit (e.g., 14,000 laminae @ 5 min/lamina @ two 8-h shifts/day @ 5 days/work week @ 4.6 work weeks/month = 3.17 months).

In actuality, the team of three workers in the cell completed the 35 assembled manifolds in a 3-month period. Completed manifold units were delivered to the sponsor upon completion of assembly so the entire completed unit cannot be shown the reader. However, Fig. 8 does depict 18 of the 35 finished manifolds that were produced. When stacked two high, the final delivered unit of 35 manifolds had principal dimensions of 61 cm × 91 cm × 31 cm. Total cost to produce the 35 manifolds of this prototype unit was approximately \$29,000 including material costs of \$17,500 and labor costs of \$11,500.

7. Validation

A one-time test loop was designed and constructed to validate the proper operation and longevity of a module composed of 50 microreactor laminae. This test loop was not considered part of the cellular manufacturing process described above, but was intended to assure that the biodiesel processing unit was able to achieve

its fuel production projections. Specifically, the loop served to test different oil-to-methanol and catalyst-to-methanol ratios as well overall flow rate to achieve an oil conversion of 99%. The schematic for the test loop is shown in Fig. 9.

The reactants were stored in stainless steel tanks under nitrogen (N_2) pressure supplied by a gas cylinder. The MeOH and KOH catalyst (0.9 M) were premixed and stored in a single 19 L tank. Soybean oil purchased from a local grocery store was stored in another 19 L tank.

The oil was pumped by nitrogen pressure (70 kPa) while the flow rate was controlled with a needle valve and monitored with a rotameter. The MeOH/KOH solution flow was controlled with an adjustable peristaltic pump. A rotameter was also used to monitor the flow rate. The reactants were directed into a static mixer composed of a 0.318 cm inner diameter Viton[®] tube containing two 15.2 cm long polypropylene static mixing elements. The tubing was immersed in a heated water bath that was temperature controlled with a thermostat. A bath temperature of 65 °C was used.

The loop piping was composed of HPDE tubing with 316 stainless steel, HDPE, and polypropylene fittings. On the oil side, an inline heater was used to continuously heat the oil as it flowed. The oil heater was made from a steel pipe with 7.62 cm inner diameter that is wrapped by a 1500 W electric band heater. The heater was connected to a variac that can be adjusted to maintain an oil temperature of 65 °C. The outside of the heater was wrapped with insulation to improve the heating efficiency and also guard against short circuits and exposure to possible chemical leaks. The reactor module inlet tube was modified so that a thermocouple could be used to monitor the inlet temperature of the reactants.

Product samples were collected periodically in water to quench the reaction. The biodiesel phase was then tested by gas chromatography to determine the relative amount of free and bound glycerol in the sample. From this information, the conversion of the oil was calculated.

The microreactor laminae were tested in stacks of 50. The plates were held in place with a stainless steel compression fixture with nine bolts and were sealed mechanically by applying torque to the nine bolts. Viton[®] gaskets (0.51 mm thick) were placed on each plate and the modules would seal with the bolts turned only hand tight. Under these conditions, we found the laminae would survive temperatures of 75 °C for several hours without deforming the channels.

The modules with Viton[®] gaskets were used to produce biodiesel continuously for more than 4 days using the test loop. At the end of the test, the laminae were found to have no damage or soap deposits. The Viton had not degraded as well.

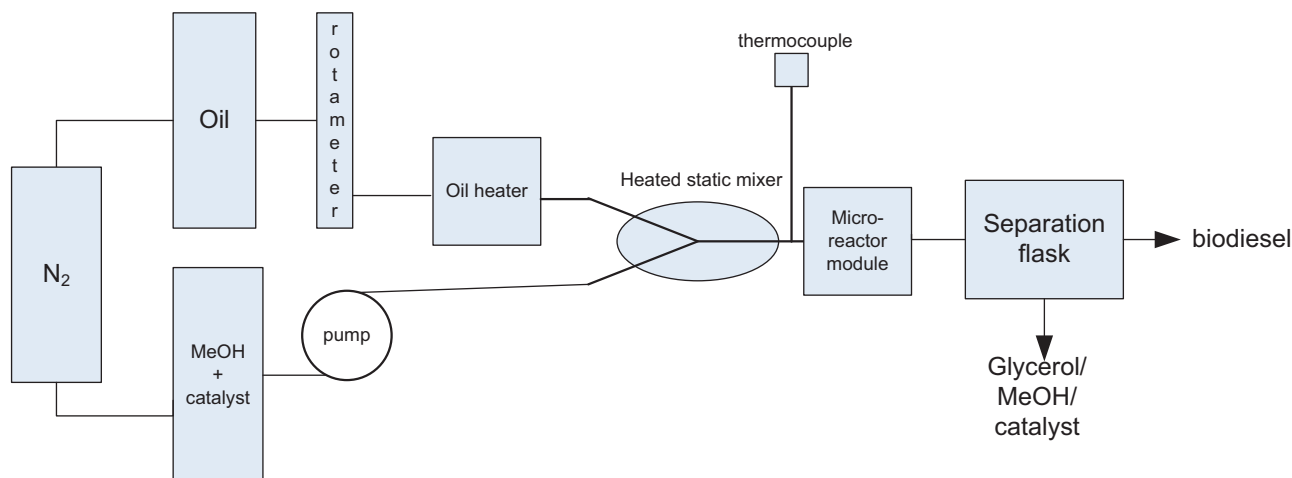


Fig. 9. Single stage test loop with static mixer schematic.

Table 1
Operating conditions for microreactor module (50 laminae) to achieve 99% oil conversion.

Property	Value
Reactant temperature	65 °C
Maximum oil flow rate	0.53 L/h
Flow rate ratio (MeOH:oil)	1:3
Catalyst concentration	0.9 M

The stack was tested for maximum flow rate to achieve 99% conversion of oil to biodiesel. The results are summarized in Table 1.

Under the conditions given in Table 1, unwashed biodiesel with less than 1% free and bound glycerol was produced at the rate of 0.53 L/h, which corresponds to an overall residence time of 2.6 min including the time spent for static mixing. With 35 manifolds containing eight modules each, a biodiesel production rate of 148 L/h or 2.47 L/min is made possible, exceeding the research team's original intended hopes of 1/9 L/min for the unit.

8. Conclusions

This research showed how bench scale biodiesel microreactors developed in the research laboratory could be designed and fabricated for high flow rate production. The modular subassembly and assembly design allowed the principal dimensions of each component – lamina, module, manifold – to remain small to allow the intensified mass transfer effects to be fully realized, while also allowing readily available, low-cost production equipment to be used for manufacturing of the full-scale units. The process was shown it could be implemented using modern manufacturing cell technology resulting in production of one lamina every 5 min, and operating with classic pull manufacturing control procedures.

With a predicted biodiesel fuel production rate of 2.47 L/min, the full-scale microreactor should exceed its goal of producing 1.9 L of fuel per minute. With continuous operation, this system is capable of producing over 1.2 million liters of fuel per year.

As inferred in the paper, the most difficult challenge the research team faced was the joining of the HDPE laminae to each other. It took well over a year for us to find a reliable mechanism to prevent leakage between laminae. In the end, we resorted to mechanical fasteners with gasket material between laminae as the only solution to reliably eliminate the leakage. Clearly, this was expensive both in terms of added material cost in the use of Viton gaskets, as well as the additional labor needed to apply gasket material and join the HDPE laminae with nut and bolt fasteners. Further research needs to be done to develop technology for making inexpensive and reliable hermetic seals between thermoplastic polymer materials.

At the time of this writing, the biodiesel production system is being evaluated by a sponsor for implementation in the South American country of Paraguay. With transportation fuel costs exceeding \$8 per gallon, and a ready supply of non-food jathropa growing wild in this country to serve as a low-cost oil feedstock to the microreactor, it is planned that a number of portable units can be manufactured and distributed near this feedstock, providing a low-cost clean fuel alternative to current petroleum-based diesel fuel.

This research for large-scale production of microreactors is extensible to other types of microreactors that can be “numbered up” to achieve high-volume flow rates. Work is underway by the research team to determine its use for transport limited reactions that are used in other synthetic fuel production processes. Examples include converting gases like methane or carbon dioxide into valuable liquid chemicals.

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