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MICROREACTORS - PORTABLE FACTORIES FOR BIODIESEL FUEL PRODUCTION

Abstract

Nowadays, when fossil fuel recourses are decreasing rapidly, biodiesel fuels are becoming more and more interesting. Being environmentally beneficial, biodegradable, non-toxic and with low pollution emission makes biodiesel interesting for consumption. Various methods have been reported for biodiesel production such as microemulsification, pyrolysis and transesterification. Main problems of this traditional approach in biodiesel production are long residence time, high operation costs and energy consumption and low efficiency. To overcome these issues new technologies for process intensification are being developed. Main features of these processes are enhanced reaction rate, reduced molar ratio of alcohol to oil, and low energy input realized by intensification of mass and heat transfer.

Microreactor technology is finding increasing application in many fields, from the chemical industry and biotechnology to the pharmaceutical industry and medicine High volume/surface ratio, short diffusion distance, fast and efficient heat dissipation and mass transfer are just some of microreactors advantages. Up to now these characteristics have been successfully used in organic synthesis where in comparison to traditional reactor systems, higher conversions and productivities have been achieved. Researchers believe that implementation of this technology into biodiesel production could be a next big breakthrough not only in process intensification but also in process economics. In this paper, application and possibilities of microreactor technology in biodiesel production have been discussed.

1. Introduction

Biodiesel referees to a diesel-equivalent, processed fuel derived from biological sources [1]. Main advantage of biodiesel is that it is increased biodegradability, with inherent lubricity and superior flash point, non-toxic and reduced emission of hydrocarbons, sulfur, carbon monoxide and pollutant particles. Therefore biodiesel is environmentally beneficial fuel [2-5]. Important disadvantages of biodiesel include high feedstock cost, increased emission of NO_x, inferior storage and oxidative stability, low volumetric energy content and inferior low-temperature operability [6-9].

There are different ways to produce biodiesel that include microemulsification [7] (reduces the high viscosity of vegetable oils by mixing them with solvents, such as methanol, ethanol and ionic or non-ionic amphiphiles and simultaneously form two normal immiscible liquids), pyrolysis [10-14] (conversion of one substrate into another using heath, or heath and a catalyst typically in absence of air or oxygen) and transesterification [15-19]. Today, the majority of biodiesel is produced by transesterification (also called alcoholysis) of vegetable oils, waste kitchen frying oils and animal fats with short chained alcohols such as methanol or ethanol in stirred tank reactors in the present of alkalis, acids or enzymes as catalysts or under supercritical methanol conditions to form esters and glycerol [20]. There are some challenges related to this processes such as:

- reaction rate can be limited by mass transfer between the oils and alcohols because they are immiscible;
- transesterification itself is a reversible reaction and therefore there is an upper limit to conversion in the absence of product removal;
- most commercial processes are performed in a batch mode and thus do not gain some of the advantages of continuous operation [21];
- biodiesel costs 1.5 – 3 times more than fossil diesel [2, 22];
- acid-, alkaline-, and enzyme-catalyzed production processes may take 2 – 24 h to obtain very high oil conversion and fatty acid methyl ester yield [20].

There are different approaches to overcome this problems like prolonging reaction time, increasing molar ratio of alcohol to oil and catalyst concentration, working under supercritical conditions (temperatures higher than 300 °C pressures higher than 40 MPa) or by adding a co-solvent, such as tetrahydrofuran in the reaction [23-25]. By adding co-solvents biodiesel could be produced in few minutes but this approach increases number of process steps and energy consumption.

Another problem in the production of biodiesel is downstream processing. High operating cost and energy consumption are required to purify biodiesel and recover excess amount of alcohol and catalyst together with significant amounts of toxic wastewater that is produced. To resolve these problem different technologies have been developed and proposed. Main goal of those methods is to improve mixing and mass/heat transfer rate between two liquid phases and enhance reaction rate so residence time may be reduces. Some of process intensification technologies are presented in Table 1 together with their comparison. The static mixers can accomplish effective radial mixing as fluids pass trough it. Their biggest advantage is low maintenance and operating cost and low space requirement because they have no moving parts. However, the mixing process relies mainly on slow, unforced molecular diffusion in the laminar regime and therefore reactions in them are still slow. Microchannel reactors improve heat and mass transfer due to short diffusion distance and high volume/surface area so reaction rates achieved in them are rapid. Beside that, because of microreactor size, they offer reductions in construction and operating costs. Oscillatory reactors enhance radial mixing and transport processes by independent and controlled oscillatory motion. In cavitational reactors, the collapse of cavity or bubbles produces high temperature and pressure turbulence locally resulting in rapid reaction rate. Rotating/spinning tube reactors take the advantage of Couette flow formation where the two liquids are mixed instantaneously and moved trough the annular gap between two tubes. Under those conditions a coherent thin liquid film is formed that leads to high mass transfer rate and very short mixing time. Microwave reactors utilize microwave irradiation to transfer energy directly into reactants and thus accelerate the rate of chemical reaction. Membrane reactors integrate reaction and membrane-based separation into single process. They achieve high reaction rate by selective removal of bound glycerin from products via a membrane. Reactive distillation combines chemical reactions and product separation by distillation also in one unit. Combination and implementation of centrifugal separation of product and reaction is the main principle of the centrifugal contractor. As the rotor in the contactor rapidly rotates within a stationary cylinder, intense mixing and good mass transfer are achieved together with quick phase separation by high centrifugal force [21].

Table 1: Comparison of process intensification technologies for continuous biodiesel production with conventional stirred thank reactor [21]

Technology	Residence time	Energy efficiency (g/J)	Operating and capital cost	Temperature control	Current status	Reference
Static mixer	~ 30 min	14.9 – 384	Low	Good	Lab scale	[26]
Microchannel reactor	28 s – several minutes	0.018	Low	Good	Lab scale	[27]
Oscillatory flow reactor	30 min		Low	Good	Pilot plant	
Cavitational reactor	Microseconds – several seconds	1×10^{-4} to 2×10^{-4} (hydrodynamic cavitation) 5×10^{-6} to 2×10^{-5} (acoustic cavitation)	Low	Good	Commercial scale	
Spinning tube in tube reactor	< 1 min		Low	Good	Commercial scale	
Microwave reactor	Several minutes	~ 0.038 L/kJ	Low	Good	Lab scale	[28]
Membrane reactor	1 – 3 h		Lower	Easy	Pilot plant	
Reactive distillation	Several minutes	$\sim1.6\times10^{-6}$	Lower	Easy	Pilot plant	[29]
Centrifugal contactor	~ 1 min		Lower	Easy	Commercial scale	

Main focus of this review paper will be placed on microchannel reactors and related technologies.

2. Microreactors

Microreactors are defined as miniaturized reaction systems fabricated by using, at least partially, methods of microtechnology and precision engineering [30]. The term “microreactor” is the name that is generally used to describe a great number of devices that have small dimensions. Reducing dimensions of the reaction system (typical dimensions of microchannel are in the range of 10 to 500 μm) they exploit rapid reaction rates and minimize heath and mass transport limitations that usually present a problem in conventional reactor systems.

2.1. Basic characteristics

Microreactors exhibit numerous practical and performance advantages when compared to traditional, equivalent macroreactors [31]. The small dimensions of microchannel (submicrometers and submillimeters size) allow usage of minimal amounts of reagent under precisely controlled conditions and make it possible to rapidly screen reaction conditions and improve the overall safety of the process [32]. Beside that, excellent mass and heath transfer, shorter residence time, smaller amount of reagents, catalyst and waste products comparing to macroscale reactors, lightweight and compact system design, laminar flow, effective mixing, short molecular diffusion distance and better process control, and small energy consumption are just some of the microsystem advantages [30,33]. Furthermore, they could be easily coupled with numerous detection techniques together with the pretreatment of the samples on the one single chip. Having in mind all those benefits one of the main motivations for the use of microreactor technology is the gain in the yield and safety.

Because of the high surface to volume ratio of microreactors (microchannel: 10,000 – 50,000 m^2/m^3; laboratory vessel: 1000 m^2/m^3 and production vessel: 100 m^2/m^3), heat transfer is very efficient and reaction temperatures in microreactors can be regulated by very effective heat removal or application [34-35]. As a result, heath transfer coefficient measured in microreactor goes up to 25,000 $W/(m^2\ K)$. This exceeds heath transfer coefficients of conventional heath exchangers by at least one order of magnitude [30,36]. The excellent heath transfer characteristics of microfabricated devices also avoid the risk of potential significant industrial accidents caused by thermal runaway [37]. Comparison between micro- and macro-heat exchange systems is presented in Table 2.

2.2. Scale-up of microreactors

Another advantage of microreactor systems is so called “numbering up” (Figure 1). Connecting microreactors of the same proven dimensions to operate in parallel or in series, higher capacities can be achieved and compact microplants could be build-up [38]. In comparison, increasing the size of conventional reactors requires plant designers to increase the size of each reactor unit. This makes scale up expensive, time-consuming and sometimes extremely difficult.

In contrast, the microchannel reactors can be shop-fabricated, and the microchannel based plants can be constructed more quickly and easily with guarantees that desired feathers of basic unit will remain unchanged when increasing total system capacity. Beside that, in microeractor plant continuous operation is uninterrupted with the replacement of the failed microreactor, while the other parallel units continue production [34].

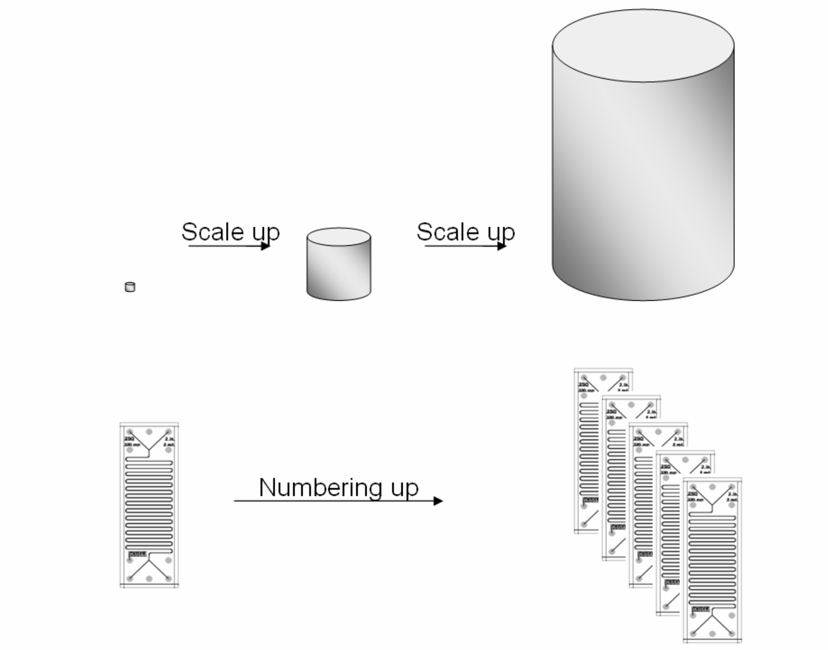

Figure 1: Comparison of scale-up methodology in macroreactor and microreactor system

Table 2: Comparison of macro- and micro-heat exchange system

Parameter	Shell and tube heat exchanger	Compact heat exchanger	Microchannel heat exchanger
Surface to volume ratio, m^2/m^3	50 - 100	850 - 1500	> 1500
Heat transfer coefficient, $W/(m^2 K)$ (liquid)	~ 5000 (tube side)	3000 - 7000	> 7000
Heat transfer coefficient, $W/m^2 K$ (gas)	20 - 100	50 - 300	400 - 2000
Approach temperature,°C	~ 20 °C	~ 10 °C	< 10 °C
Flow regime	Turbulent	Turbulent	Laminar

2.3. Micro-total-analysis-systems

In last few years, together with microplant development, a great part of research investigation is focused on integrated micro-systems, so called micro-total-analysis-systems (*µ-TAS*; Figure 2). *µ-TAS* includes pumps, valves, mixers, reactors and separators [39] and other process equipment necessary for all production steps such as for example purification of produced products. Optimally, such devices would automatically perform sampling, sample preparation, separation, detection and data processing in a fully integrated manner.

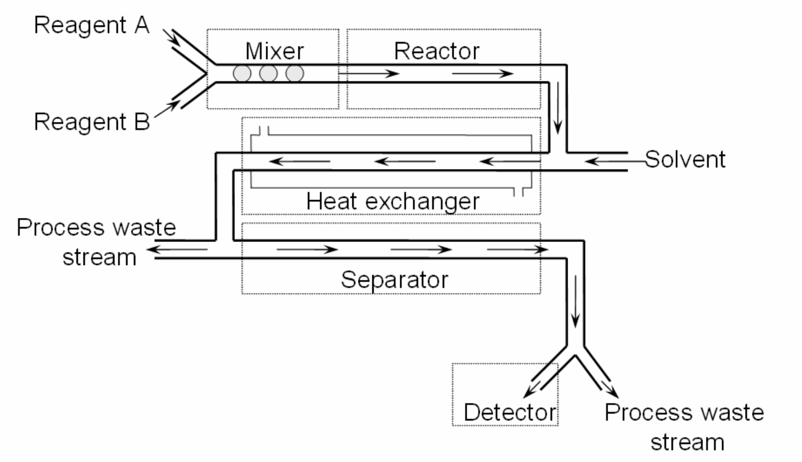

Figure 2: Micro-total-analysis-system diagram (µ-TAS)

3. Biodiesel production in microreactors

3.1. New technologies for biodiesel production on macroscale

How to promote efficiency of biodiesel production and how to reduce corresponding process costs become an increasing worldwide concern. Implementing new technologies and scientific research a great progress has been made. Up to now, taking in consideration all microreactor advantages, different approaches and different technologies were implemented for biodiesel production on macroscale. Results obtained in such processes were promising and optimistic. Before implementing microreactors in biodiesel production several new processes have been developed recently to improve production on macroscale. Using ultrasound irradiation way for biodiesel synthesis, Stavarache et al. (2007) [40] exceeded 90 % yield during the residence time of 20 min. Supercritical transesterification with co-solvent was sufficient to achieve 97 % conversion within 10 min [41]. Demirbas (2008) [42] used MgO as solid catalyst on supercritical conditions (methanol/oil molar ratio 4:1, T = 252 °C, p = 24 MPa) for biodiesel production and reached nearly complete conversion within 6 ~ 7 min. Azcab and Danisman (2008) [43] reported that using microwave system, the transesterification rate reached 93.7 % at a residence time of 5 min.

3.2. Biodiesel production on microscale

In comparison to previously mentioned macroscale processes, when microreactors were implemented even shorter residence time was necessary to perform production process. Canter (2006) [44] reported that biodiesel could be produced in a microreactor of the size of a conventional credit card and that reaction could occur at mild conditions. When the residence time was about 4 min, over 90 % yield was obtained. Recently, the yield of methyl esters of 99.4 % was obtained at the residence time of 5.89 min with KOH concentration of 1 % and methanol to oil molar ratio of 6 at 60 °C in capillarity microreactor with inner diameters of 0.25 mm [20]. Authors also observed that the reaction temperature was the minimal factor in the yield of methyl ester but that the inner diameter of the microchannel reactor had a strong influence on the transesterification reaction. For the same conditions, but using microchannel of inner diameter of 0.53 mm, 95 % methyl yield could be obtained within a residence time of about 6 min. Thus, it could be concluded that higher methyl ester yield could be obtained at even shorter residence time for the microchannel reactor within the smaller inner diameter.

Different types of micromixer units with various operating principles and parameters have been designed and successfully applied in microreactor technology. They are used to decrease diffusion lengths and to increase interfacial area between phases in microchannel. Combination of micromixer and microchannel were also applied in biodiesel production. As presented in Figure 3 oil and methanol/KOH streams were premixed before entering the microchannel.

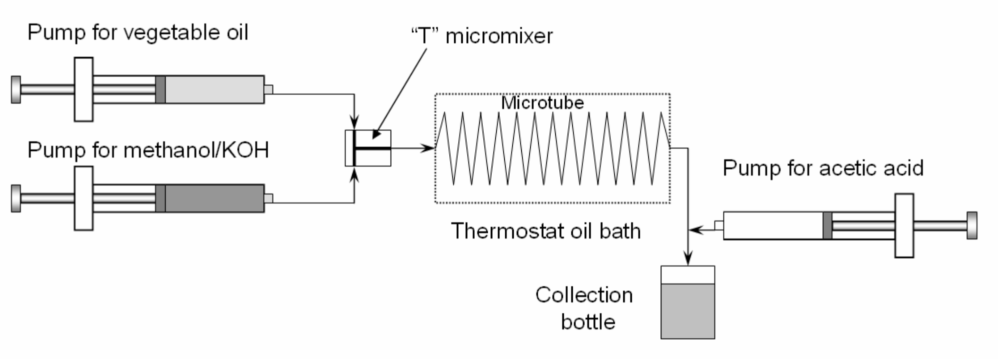

Figure 3: Microreactor system for biodiesel production

Using this approach sunflower oil was completely converted to biodiesel even in the residence time as short as 112 s at a reaction temperature of 60 °C in a system composed of a micromixer (T-type) and microreactor tube (inner diameter = 1 mm; length = 160 mm) [45].

Significant improvement was made when zigzag microchannel reactor was implemented for continuous alkali-based biodiesel synthesis [27]. The configuration of zigzag microchannel reactor with narrower channel size and more turns is showed in Figure 4. This type of reactor was shown to intensify the biodiesel production by obtaining smaller droplets/slugs to those microchannel reactors with "T" and "Y"-flow structure. Slug flow in a microchannel is a flow characterized by a series of a liquid slug of one phase separated by other. Main advantage of this kind of flow is complex transport mechanism: convection within the individual slag of each phase and interfacial diffusion between two adjacent segments. At residence time of 28 s and temperature of 56 °C, yield of methyl ester reached 99.5 % using a 9:1 molar ratio of methanol to oil and a catalyst concentration of 1.2 wt % sodium hydroxide. Additional advantage of this system is energy consumption. Authors reported that under these conditions less energy consumption was needed for the same amount of biodiesel then in the case of conventional stirred reactor.

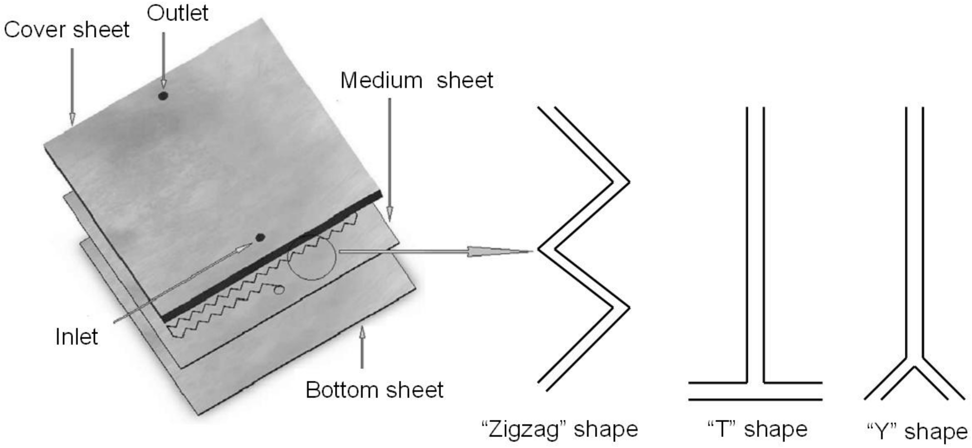

Figure 4: Comparison of different micorcahnnel configuration types [27]

3.3. Portable biodiesel production factory

Jovanovic et al. (2009) [46] developed and patented biodiesel process production in microchannel. Figure 5 presents illustrated scheme for biodiesel production together with digital image of reaction system with a single chip having plural microchannels extending axially along the long axis of the microchip (Figure 6a). They used two syringes pumps to deliver substrates (soybean oil and stock solution of methanol with dissolved NaOH) in microchannel. Effects of channel thickness (100 µm and 200 µm) and residence time on biodiesel production were investigated. Transesterification products biodiesel and glycerol were collected in cold trap, which allowed effective separation of the two phases. Working with microreactors with a 100 µm microchannel thickness and soybean oil and a transesterification processing temperature of about 25 °C, conversion of soybean oil to biodiesel ranged form about 12 % at 0.4 minute residence time to about 91 % at 10 min residence time.

Total methyl ester concentration ranged from about 0.3 mol/L at about 0.4 minute residence time to 2.5 mol/L at about 10 minutes residence time. When microchannel of 200 μm thickness was used, at residence time of 0.4 minute, oil conversion of 4 %, with total ester concentration of 0.1 mol/L was achieved while 86 % conversion was achieved for residence time of 10 minutes with 2.4 mol/L methyl ester concentration. Like in some previous reports [20] influence of the microreactor inner diameter on the transesterification reaction was clearly demonstrated.

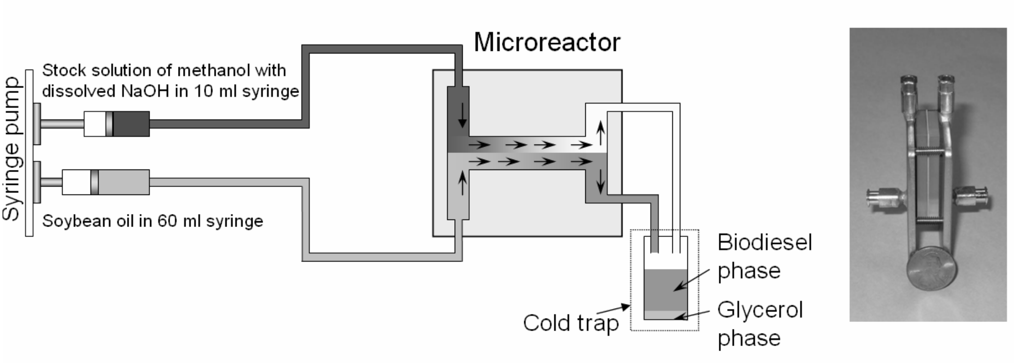

Figure 5: Schematic diagram and digital image providing side perspective view of one embodiment of a microreactor used to produce biodiesel [46]

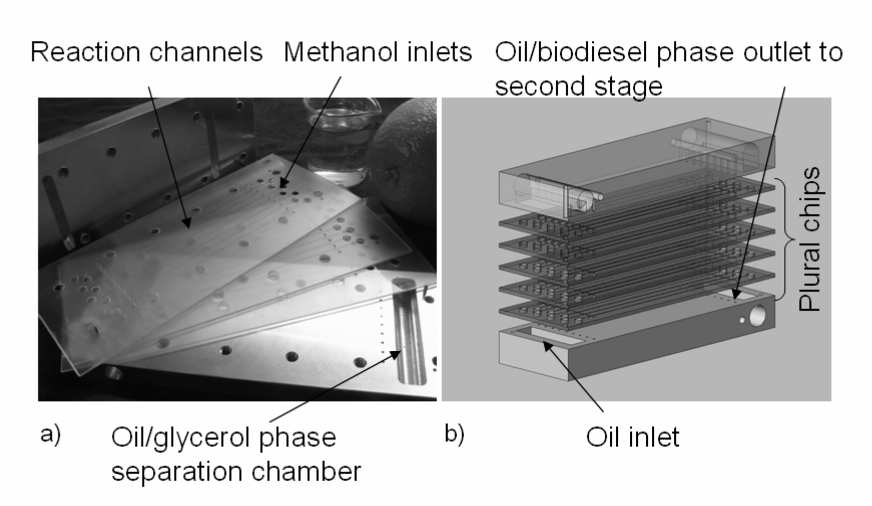

Figure 6: Biodiesel synthesis in microreactor:
a) single chip with plural fluid microchannels;
b) plural chips with plural fluid microchannels [46]

Next step in the process development was developing of a microreactor with plural microchips each with plural fluid microchannels and plural fluid ports for delivering fluid to the microchannel (Figure 6b). They are combined in such form to increase fluid throughput and hence increase biodiesel production. It is realized by numbering up approach and each microchip has the same microfeatures. Finally, Jovanovic et al. (2009) [46] developed stage plant for biodiesel production. Typical dimensions of such portable plant were length : height : depth = 50.8 cm : 40.64 cm : 20.32 cm. Capacity of such system for biodiesel production was reported to be 12 ml/min or 17.28 L of biodiesel daily.

4. Conclusion

Biodiesel is biodegradable, non-toxic, low pollutant emission and environmentally beneficial fuel. Majority of biodiesel is produced by transesterification in stirred tank reactors and different waste streams containing oils and fats are used as substrates. To overcome the main bottlenecks of traditional processes for biodiesel production such as long residence time, high operation costs and energy consumption, low efficiency, and downstream processing different new technologies are being developed. In comparison to conventional macroreactors, microreactors have significant potential for enhancement of biodiesel production due to excellent mass and heath transfer, shorter residence time, smaller amount of reagents, catalyst and waste products comparing to macroscale reactors, lightweight and compact system design, laminar flow, effective mixing, short molecular diffusion distance, better process control, and small energy consumption. Additionally, microreactors are scaled-up easily which reduces the capital and operating costs and increases profit. Portable biodiesel production factory could satisfy all household fuel needs just by using different waste oils as substrates.

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