Shahrekord University

Journal of Microfluidic and Nanofluidic Research

Journal homepage: https://jmnr.sku.ac.ir

JMNR

Review Article

A Review of Biodiesel Synthesis in Microchannel Reactors: From Fundamentals to Process Intensification

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ARTICLE INFO

Article history:
Received: 2025-08-04
Revised: 2025-09-13
Accepted: 2025-09-14

Keywords:
Microreactor;
Biodiesel;
Review;
Microfluidics;
Micromixer.

ABSTRACT

Biodiesel production in conventional batch reactors is limited by long residence times and mass transfer constraints due to the immiscibility of oils and alcohols. Microreactor technology offers process intensification through high surface-area-to-volume ratios and enhanced mixing. This review synthesizes seventeen key studies on biodiesel synthesis in microchannel reactors. Passive micromixers with optimized geometries significantly enhance performance. T-channels with alternate circular obstructions achieved mixing indices of 0.99 and conversions of 99.09%. Uniflow comb micromixers achieved high mixing quality with pressure drops ten times lower than cross-shaped designs. Tesla-shaped microreactors produced 96.7% yields, outperforming omega (95.3%) and T-shaped (93.5%) configurations. Key challenges include developing biphasic kinetic models, managing the mixing-pressure drop trade-off (up to 178 bars), fabricating reliable seals for polymer reactors, accommodating lower-quality feedstocks, implementing online monitoring, and ensuring uniform flow distribution in scaled-up systems. Addressing these gaps will enable distributed, small-scale biodiesel production.

1. Introduction

Biodiesel has emerged as a viable alternative to fossil diesel due to its renewable nature, biodegradability, and reduced environmental impact. It consists of mono-alkyl esters of long-chain fatty acids derived from vegetable oils or animal fats, typically produced through transesterification [1]. While conventional batch reactors require several minutes to hours to achieve high yields, microreactor technology has recently demonstrated remarkable potential for process intensification in biodiesel synthesis [1, 2, 3].

Microreactors are generally defined as devices with interconnected microchannels where small amounts of reagents are handled and reacted. Their advantages include high surface area-to-volume ratios, enhanced heat transfer, precise temporal and spatial control of reagents, and the ability to generate concentration gradients [1, 4]. These characteristics are particularly beneficial for transesterification reactions, which are limited by mass transfer due to the immiscibility of vegetable oils and alcohols [5, 6].

A critical component of these microreactors is the micromixer, which must perform quick and efficient mixing of reactants. The laminar flow regime typical of microchannels presents a significant challenge for mixing [4]. Consequently, various passive micromixer designs have been proposed to enhance mixing through geometric modifications that promote flow splitting, recombination, and vortex formation [1, 3, 7]. The use of microreactors for biodiesel synthesis has attracted considerable attention because they enable continuous production with higher yields and shorter residence times compared to conventional batch processes [8, 9].

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Cite this article as:

Nourbakhsh, A., 2025. A Review of Biodiesel Synthesis in Microchannel Reactors: From Fundamentals to Process Intensification. *Journal of Microfluidic and Nanofluidic Research*, 2(3), pp. 173-179. https://doi.org/10.22034/jmnr.2025.116662

2. Fundamentals

The transesterification reaction consists of three consecutive reversible steps. Triglycerides (TG) react with an alcohol to form diglycerides (DG) and esters, followed by conversion of DG to monoglycerides (MG), and finally MG to glycerol (GL). The overall reaction can be represented as TG + 3A ↔ GL + 3E, where A represents alcohol and E represents ester [1, 4, 10]. The reaction rate for triglyceride conversion in the presence of a base catalyst can be expressed as $(r_{TG}) = k\ C_{TG}^{\alpha}$, with kinetic parameters varying according to oil type, alcohol, and catalyst concentration [1, 11]. For Jatropha curcas oil ethanolysis, the reaction order for triglycerides was determined to be 1.266 with a kinetic constant of 0.1830 using KOH as a catalyst [1].

At low Reynolds numbers (typically below 100), flow in microchannels is laminar, and mixing occurs primarily by molecular diffusion [1, 3]. The mixing efficiency is characterized by a mixing index MI, calculated from the standard deviation of the mass fraction of oil at the channel outlet: $MI = 1 - \sqrt{(\sigma^2/\sigma_{max}^2)}$, where σ is the variance of the mass fraction and σ_{max} is the maximum variation over the data range [1, 12]. For T-channels, mixing is dominated by molecular diffusion, whereas micromixers with obstructions promote split-and-recombine flow patterns that increase interfacial area [3].

The modified Villermaux-Dushman method has been successfully used to characterize micromixing efficiency in monophasic systems. This method is based on the mixing-sensitive conversion of two competing reactions, where high absorption of triiodide at 286 nm indicates insufficient micromixing [13]. Using this method, Schwarz et al. ranked three mixers and found that the LTF-MX micromixer (split-and-recombine principle) showed the best micromixing performance [13].

Three characteristic times govern the reactive process: residence time ($\tau_R = L_m/\bar{u}$), diffusion time ($\tau_D = (w/2)^2/2D$), and reaction time ($\tau_r = 1/k$). The Damköhler numbers, defined as the ratios between these times, indicate whether the process is controlled by mixing, reaction kinetics, or both [1, 4]. For values of $D_{aI} > 1$, chemical species have enough time to react and the reaction will be complete. When $D_{aI} \rightarrow 0$, reactants do not have sufficient time to react [1]. The flow patterns in microtube reactors significantly affect biodiesel synthesis. Guan et al. observed that at the entrance region of a microtube, two-phase segmented flow of methanol and oil segments occurs [5]. Fine droplets containing produced glycerol and unreacted methanol form at the interface due to the stabilization effect of surfactants such as monoglyceride and diglyceride [5]. As the reaction progresses, the segmented flow converts to a quasi-homogeneous phase due to intense aggregation of fine droplets. When the methanol/oil molar ratio was 23.9 at 60°C, oil conversion reached 100% within a residence time of 100 seconds, corresponding to the point where segmented flow just converted to quasi-homogeneous phase [5].

3. Advance in the Field

3.1 Micromixer Geometries for Enhanced Mixing

Numerous studies have demonstrated that micromixer geometry significantly influences biodiesel synthesis efficiency. Santana et al. numerically investigated T-channel, T-channel with circular obstructions, and T-channel with alternate circular obstructions for Jatropha curcas oil-ethanol mixing and reaction [1]. The T-channel with alternate circular obstructions showed the highest mixing index (0.99) and maximum conversion of 99.09%. The presence of obstacles improved species conversion by promoting split-and-recombine flow patterns, increasing interfacial area, and reducing diffusion pathways. The pressure drop increased almost linearly with increasing flow rate, but higher oil conversions were obtained at low flow rates (high residence times) and consequently low pressure drops [1].

Similarly, Sabry et al. [3] developed a uniflow comb micromixer based on the multi-lamination principle for biodiesel production at low Reynolds numbers. Their design achieved high mixing quality with significantly lower pressure drops compared to other publications. The introduction of a contraction section nearly doubled mixing quality, while the addition of optimized ribs (three ribs extending to half channel width) increased mixing quality by 36% with a 131% increase in pressure drop. The final micromixer configuration was tested for different fluid mixtures and systematically gave better results compared to recent publications [3].

Martinez Arias et al. [6] experimentally compared T-shaped, omega-shaped, and Tesla-shaped microreactors for castor oil ethanolysis. The Tesla-shaped microreactor exhibited the highest ethyl ester yields (96.7%) due to superior mass transfer intensification. The chaotic flow patterns generated by the Tesla geometry promoted effective collision between fluid streams, enhancing interfacial contact. The omega-shaped microreactor achieved 95.3% yield, while the T-shaped reactor reached 93.5% under the same conditions [6].

Santana et al. [4] also numerically studied T-micromixer, Cross-micromixer, and Double-T-

micromixer for Jatropha curcas oil-ethanol reaction. The Cross-micromixer showed the highest mixing index, with average values of 0.99. The residence time played an important role in oil conversion, with maximum conversion reaching 98.48%, 98.55%, and 96.59% for T-, Cross-, and Double-T-micromixers, respectively, after 40 seconds of residence time.

3.2 Static Elements and Wire Coils

Santana et al. [7] investigated micromixers with static elements for sunflower oil ethanolysis. The micromixer with static elements (MSE) showed superior mixing index (0.99 at Re=100) compared to the T-micromixer (0.32). The static elements induced flow direction changes, boundary layer disturbance, and vortex formation, which enhanced contact area and reduced diffusion paths. Experimentally, the MSE achieved 99.53% FAEE at 50°C with an ethanol/oil molar ratio of 9 and 1% catalyst concentration at a residence time of approximately 12 seconds.

Aghel et al. [8] employed a wire coil insert in a microreactor for soybean oil transesterification with methanol. Using response surface methodology for optimization, they achieved 99% methyl ester conversion at a residence time of 180 seconds under optimal conditions (methanol/oil molar ratio 9:1, 1.2% KOH, 60°C). The wire coil promoted intense mixing and enhanced mass transfer, although pressure drop increased linearly with flow rate.

Rahimi et al. [14] studied transesterification of soybean oil with methanol in a T-shaped microreactor without inserts. Using Box-Behnken experimental design, they achieved 89% FAME at 26 seconds residence time and 98% at 180 seconds under optimal conditions (methanol/oil molar ratio 9:1, 1.2% KOH, 60°C). The pressure drop increased linearly with flow rate, and the energy dissipation rate analysis showed that residence time was more important than mixing time for this slow transesterification reaction.

3.3 Zigzag Microchannel Reactors

Wen et al. [9] developed zigzag microchannel reactors for continuous alkali-catalyzed biodiesel synthesis. Smaller channel sizes and more turns resulted in smaller droplet diameters and higher efficiency. The optimized reactor (Zigzag-1) achieved 99.5% methyl ester yield at a residence time of only 28 seconds, with conditions of 9:1 methanol/oil molar ratio, 1.2% catalyst, and 56°C. The droplet size distribution analysis revealed that the mean droplet size was 1.93 μm for Zigzag-1, approximately one third that of the batch stirred reactor after 1 hour of agitation. The energy consumption per gram biodiesel was 54.5 J/g for Zigzag-1 compared to 133.2 J/g for the stirred reactor.

Dai et al. [15] designed a novel microchannel reactor with integrated mixing and reaction layers [15]. Using response surface methodology, they optimized conditions to achieve 99.5% biodiesel yield at a residence time of 14.9 seconds, with a methanol/oil molar ratio of 8.5, 1.17% KOH, and 59°C. The reactor demonstrated higher moisture tolerance than traditional stirred reactors (93.7% yield at 0.2% added water) but remained sensitive to free fatty acid content (yield below 90% at acid value of 0.37 mg KOH/g).

3.4 Process Scale-Up

Billo et al. [16] reported the development of a cellular manufacturing process for a full-scale biodiesel microreactor capable of producing 2.47 L/min (1.2 million liters per year). The scale-up was achieved through numbering-up, fabricating over 14,000 individual microchannel laminae assembled into modules and manifolds. Each lamina had 500×500 μm microchannels with serpentine flow patterns. The manufacturing cell produced one lamina every 5 minutes, with 35 manifolds containing eight modules each completing production in 3 months. A module of 50 laminae achieved 99% oil conversion at a flow rate of 0.53 L/h with a residence time of 2.6 minutes. The authors identified laminae joining as the most difficult challenge, ultimately using high-pressure fastener sealing with Viton gaskets.

3.5 Micromixer Comparison Studies

Sun et al. [12] compared four micromixers (T-mixer, J-mixer, rectangular interdigital micromixer RIMM, and slit interdigital micromixer SIMM-V2) for cottonseed oil transesterification. The multilamination micromixers RIMM and SIMM-V2 produced superior FAME yields (approximately double those of T and J mixers) due to the formation of many small methanol droplets (50-500 μm) dispersed in the oil phase, greatly increasing interfacial area. Using a Dixon-ring-packed PTFE tube as a delay loop, they achieved 99.5% FAME yield at a flow rate of 10 mL/min with a residence time of 17 seconds. The pressure drop in this system was 0.7 MPa at 10 mL/min, lower than the 0.8 MPa at 2.5 mL/min in a 0.6 mm i.d. delay loop.

Jachuck et al. [17] developed a continuous process for biodiesel production using a narrow channel reactor with 1.5 mm internal diameter. Using canola oil and methanol with 1% NaOH catalyst at 60°C and 80 psig pressure, they achieved greater than 98% conversion in a residence time of 3 minutes. The flow regime changed from slug flow at the inlet to stratified

flow at the outlet, indicating in-situ separation of biodiesel and glycerol phases within the reactor. This immediate separation hindered the reversible reaction and maximized conversion.

3.6 Kinetic Modeling

Richard et al. [10] developed a comprehensive model for the transesterification of sunflower oil with ethanol in microreactors, considering both mass transfer and reaction kinetics. Their model successfully represented the evolution of all reaction components for ethanol to oil molar ratios ranging from 6.0 to 45.4. The mass transfer coefficient was determined to be 1.0×10^{-5} m/s, and the model was validated in both reaction-controlled (microreactors) and mass transfer-controlled (millireactors) regimes. The model was subsequently used to simulate glycerol removal, showing that continuous glycerol removal could shift the equilibrium toward maximum ethyl ester formation.

Santacesaria et al. [2] proposed a simplified kinetic model for biodiesel synthesis in microreactors with static elements. They demonstrated that monophasic pseudo-homogeneous kinetic models are unsuitable for describing continuous reactors with intense micromixing, as the partition of catalyst, reactants, and products between polar and apolar phases dramatically changes with conversion. Using ChemCAD software with the UNIFAC LLE model, they showed that the volume ratio between polar and apolar phases changes significantly with conversion, affecting catalyst distribution and reaction rate.

Schwarz et al. [13] performed base-catalyzed ethanolysis of soybean oil in seven different microreactor configurations. They showed that the yield of fatty acid ethyl esters depends strongly on mass transfer limitations and correlates with micromixing efficiency. Kinetic modeling revealed that the first consecutive reaction is highly reversible in continuous reactors compared to batch processes, and that mass transfer coefficients depend on flow rate following an exponential relationship. The LTF-MX micromixer (split-and-recombine) showed the best performance, achieving higher yields than batch processes at otherwise identical conditions.

4. Challenges, Research Gaps, and Future Directions

4.1 Mass Transfer Limitations and Kinetic Modeling

Despite significant advances, mass transfer limitations remain a central challenge. Richard et al. demonstrated that the base-catalyzed ethanolysis of vegetable oils is strongly influenced by mass transfer, with the yield correlating with micromixing efficiency [10]. Their kinetic modeling revealed that the first reaction step (TG to DG) is highly reversible in continuous reactors compared to batch processes, indicating that unconsidered mass transfer effects influence observable reaction rates [10, 13].

A significant research gap is the development of comprehensive kinetic models that properly account for biphasic behavior, mass transfer, and the changing phase equilibria as the reaction progresses [2, 13]. Santacesaria et al. [2] emphasized that the catalyst initially present as potassium methoxide is mainly distributed in the form of mono and diglyceroxides, and that successive transesterification steps occur preeminently in the oil phase. Future models should incorporate these mechanistic insights.

4.2 Pressure Drop and Energy Efficiency

A common trade-off exists between mixing enhancement and pressure drop. Santana et al. [1] reported pressure drops ranging from 2 to 178 bars for micromixers with circular obstructions. Aghel et al. [3] defined a performance ratio to evaluate energy efficiency, finding that wire coil inserts are most advantageous at lower flow rates where their role in increasing mixing intensity is more significant relative to the baseline [8]. Sabry et al. achieved low pressure drops through careful design of the uniflow comb micromixer.

Future work should focus on optimizing geometric parameters to maximize mixing while minimizing energy consumption, potentially through computational fluid dynamics coupled with multi-objective optimization algorithms [3, 7]. The use of larger microchannels (500 μm) as demonstrated by Billo et al. [16] may offer a practical compromise between mixing efficiency and pressure drop.

4.3 Material and Fabrication Challenges

Billo et al. [16] identified laminar joining as the most difficult challenge in scaling up microreactors for biodiesel production. High-density polyethylene (HDPE) proved chemically resistant but difficult to seal. After testing laser sealing, adhesive sealing, hot rolling, and other methods, high-pressure fastener sealing with Viton gaskets was the only reliable solution, adding material and labor costs. Developing cost-effective, reliable hermetic sealing methods for thermoplastic polymers remains an important research direction.

Additionally, most microreactors reported in the literature were fabricated from PDMS using soft lithography [6, 7]. While suitable for laboratory-scale studies, PDMS may swell in contact with

organic solvents, limiting long-term stability. Alternative materials such as stainless steel [5, 9, 15], PFA [10], and FEP [5] tubes have been successfully used and may be more suitable for industrial applications.

4.4 Feedstock Quality and Catalyst Reusability

Most microreactor studies use refined vegetable oils with low free fatty acid and water content. Dai et al. [15] showed that while the microchannel reactor had greater moisture tolerance than traditional reactors (93.7% yield at 0.2% added water), it remained sensitive to free fatty acids (yield below 90% at acid value of 0.37 mg KOH/g). Jachuck et al. [17] successfully used refined canola oil, while Santana et al. used Jatropha curcas oil, a non-edible feedstock [1, 4].

Research is needed on microreactor performance with lower-quality feedstocks such as waste cooking oils, animal fats, and algae oils. Additionally, the integration of heterogeneous catalysts into microchannel reactors could eliminate downstream separation steps and enable catalyst reuse [2, 15]. The use of supercritical conditions without catalyst has been reported [9], but requires high temperature and pressure.

4.5 In Situ Monitoring and Process Control

Martinez Arias et al. [6] evaluated near-infrared spectroscopy with a fiber-optic probe for in situ monitoring of transesterification in microreactors. While ethyl esters showed a characteristic absorbance at 5000-5500 cm^{-1}, baseline instability due to the small optical path length of microchannels presented challenges. Future work should develop robust online monitoring techniques suitable for continuous microreactor operation, potentially through improved probe design or alternative spectroscopic methods such as Raman spectroscopy.

Richard et al. [10] successfully used gas chromatography for off-line analysis but noted that online monitoring would be valuable for process control. The development of in situ sensors for measuring conversion, temperature, and pressure within microchannels would enable real-time optimization.

4.6 Numbering-Up and Industrial Implementation

Although Billo et al. [16] demonstrated that numbering-up is feasible for large-scale production, challenges remain in ensuring uniform flow distribution among thousands of parallel microchannels. Flow maldistribution can lead to varying residence times and conversion efficiencies. Research on manifold design and flow distribution optimization is essential for reliable industrial implementation.

Furthermore, economic analyses comparing microreactor-based biodiesel production with conventional processes under realistic conditions are needed to guide commercialization efforts. Jachuck et al. [17] demonstrated that the intensified narrow channel reactor achieved 99.8% conversion in 3 minutes compared to 3 hours for batch operation, but detailed cost analysis was not provided. Future work should include techno-economic assessments that account for capital costs, operating costs, and potential revenue from glycerol byproduct.

5. Conclusions

The body of research reviewed demonstrates that microreactor technology offers significant advantages for biodiesel synthesis compared to conventional batch processes. Passive micromixers with optimized geometries, including circular obstructions [1], static elements [7], wire coils [8], zigzag channels [9], multilamination designs [12, 13], and Tesla/omega shapes [6], consistently achieve high mixing indices (0.95-0.99) and oil conversions (95-99.5%) at residence times drastically reduced from hours to seconds or minutes.

The intensification mechanism relies on increasing interfacial area between immiscible reactants through droplet size reduction (as low as 1.93 μm) [9], promoting split-and-recombine flow patterns [1], inducing vortex formation at low Reynolds numbers [7], and generating chaotic advection [6]. Smaller channel hydraulic diameters (down to 240 μm) [9] and more frequent flow reorientations [9] produce smaller droplets and higher conversion efficiencies.

Several studies have successfully scaled up microreactor technology through numbering-up, with one full-scale system demonstrating production capacity of 1.2 million liters per year [16]. Response surface methodology has proven effective for optimizing reaction conditions [8, 14, 15], with typical optima including methanol/oil molar ratios of 6-9:1, catalyst concentrations of 1-1.2%, and temperatures of 50-60°C [1, 5, 7, 8, 9, 14, 15, 17].

Kinetic modeling has advanced from simple pseudo-homogeneous models [2] to comprehensive biphasic models incorporating mass transfer [10, 13]. However, challenges remain in developing models that accurately predict behavior across different reactor geometries and operating conditions [2, 10, 13].

Key challenges for future research include: developing accurate biphasic kinetic models that account for changing phase equilibria [2, 10];

managing the trade-off between mixing enhancement and pressure drop [1, 3, 8]; fabricating reliable, cost-effective seals for polymer-based microreactors [16]; accommodating lower-quality feedstocks containing water and free fatty acids [15]; implementing robust online monitoring techniques [6]; ensuring uniform flow distribution in numbered-up systems [16]; and conducting comprehensive techno-economic analyses to guide commercialization.

Addressing these research gaps will be essential for the widespread industrial adoption of microreactor technology for biodiesel production. The potential benefits, including drastically reduced reaction times, continuous operation, enhanced mass and heat transfer, improved safety, and the possibility of distributed, small-scale production systems, make this technology a promising pathway toward more sustainable and economically viable biodiesel manufacturing. It should be pointed out that numerous passive and active micromixer designs can be employed to produce biodiesel [18-23].

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