

International Journal of Advanced Chemistry Research

ISSN Print: 2664-6781
ISSN Online: 2664-679X
IJACR 2025; 7(4): 172-175
www.chemistryjournals.net
Received: 17-01-2025
Accepted: 21-02-2025

Katarzyna Mazur
Department of Process
Chemistry and Engineering,
Tatra Mountain University of
Technology, Kraków, Poland

Izabela Kowalski
Department of Process
Chemistry and Engineering,
Tatra Mountain University of
Technology, Kraków, Poland

Process intensification of soap manufacturing using microreactor technology

Katarzyna Mazur and Izabela Kowalski

DOI: <https://www.doi.org/10.33545/26646781.2025.v7.i3c.437>

Abstract

Soap has been made the same way for centuries: heat fat and alkali in a kettle, stir for hours, and hope for a consistent product. Modern soap factories still rely on large batch reactors with residence times of one to four hours, poor heat transfer, and significant batch-to-batch variability. Microreactor technology continuous flow through channels narrower than a millimetre collapses those hours into seconds by providing surface-area-to-volume ratios three orders of magnitude higher than a stirred tank. This research saponified olive oil with aqueous NaOH in a split-and-recombine glass microreactor (channel width 500 μm) at three temperatures (50, 60, 70°C) and eight residence times (5-120 s). At 70°C and 60 s residence time, conversion reached 97.1% matching the 94.3% achieved by conventional batch processing in 2 h in a device small enough to fit on a laboratory bench. The microreactor product had lower free fatty acid content (0.28 vs. 0.42%), higher total fatty matter (78.6 vs. 74.3%), and tighter quality distribution than the batch product. Energy consumption per kilogram of soap dropped by an estimated 62%. These results demonstrate that microreactor saponification can deliver faster, more uniform, and more energy-efficient soap production.

Keywords: Microreactor, saponification, soap, process intensification, olive oil, NaOH, continuous flow, residence time, fatty acid, energy efficiency

Introduction

The global soap market exceeds 35 billion USD annually, yet the core chemistry alkaline hydrolysis of triglycerides into glycerol and fatty acid salts has not changed since the Babylonians first boiled animal fat with wood ash around 2800 BC ^[1]. What has changed is the scale: modern plants process hundreds of tonnes per day in stainless-steel kettles, autoclaves, or continuous column reactors. Even in continuous systems, residence times range from 30 min to 4 h because mixing at the oil-water interface is the rate-limiting step ^[2]. Microreactors devices with channel dimensions below 1 mm solve the mixing problem by shrinking diffusion distances to micrometres and creating interfacial area on the order of 10,000-50,000 m^2/m^3 , compared with $\sim 100 \text{ m}^2/\text{m}^3$ in a stirred tank ^[3]. Heat transfer is similarly enhanced, allowing near-isothermal operation even with strongly exothermic reactions. Process intensification through microreactors has been demonstrated for biodiesel transesterification, nitration, and polymerization, but reports on soap saponification are scarce ^[4, 5].

This research used a commercial split-and-recombine glass microreactor to saponify olive oil with NaOH, mapped conversion across temperature and residence time, compared product quality with a conventional batch process, and estimated energy savings.

Safety Considerations

Concentrated NaOH (10 M) is severely corrosive; face shield, chemical-splash goggles, and heavy-duty gloves were worn during handling. The microreactor was enclosed in a polycarbonate safety shield. Hot oil at 70°C poses a burn hazard; insulated tubing and thermocouple interlocks were used. All waste was neutralized to pH 7 before drain disposal ^[6].

Corresponding Author:
Katarzyna Mazur
Department of Process
Chemistry and Engineering,
Tatra Mountain University of
Technology, Kraków, Poland

Material and Methods

Material

All work was performed at the Department of Process Chemistry and Engineering, Tatra Mountain University of Technology, Kraków, from February to October 2023. Extra-virgin olive oil (acid value 0.6 mg KOH/g, iodine value 82, purchased from a local distributor) and NaOH pellets (AR grade, POCH, Gliwice) were used. The microreactor was a Dolomite split-and-recombine glass chip (channel width 500 μm , depth 300 μm , total channel length 1.2 m, internal volume 180 μL) mounted on a Peltier temperature stage [7].

Methods

Olive oil and 10 M aqueous NaOH (1.05 $\times$ stoichiometric) were pumped by two Cetoni neMESYS syringe pumps into the microreactor at combined flow rates adjusted to give residence times of 5, 10, 20, 30, 45, 60, 90, and 120 s. Temperatures were 50, 60, and 70°C. Product was collected

at steady state (after 3 dead volumes), quenched in cold water, and the soap phase separated from glycerol by centrifugation. Conversion was determined by titrating residual free fatty acid (FFA) with KOH (AOCS method Ca 5a-40) [8]. Batch saponification followed the same oil:NaOH ratio in a 500 mL jacketed reactor at 70°C, 400 rpm, for 2 h. Product quality was assessed by FFA (%), total fatty matter (TFM,%), moisture (Karl Fischer), pH (1% solution), and lather volume (Ross-Miles method) [9]. All conditions were run in triplicate.

Statistical Analysis

Conversion data were fitted to a pseudo-first-order kinetic model. Product quality metrics were compared by paired t-test (batch vs. microreactor) at $p < 0.05$. Arrhenius activation energy was calculated from rate constants at three temperatures. All analyses used MATLAB R2023a [10].

Results

Table 1: Saponification conversion (%) as a function of residence time and temperature in the microreactor

T (°C)	5 s	10 s	20 s	30 s	45 s	60 s	90 s	120 s
50	18.3	32.7	51.4	63.8	74.2	81.6	88.3	91.7
60	24.6	42.3	64.7	78.4	87.1	92.3	96.4	97.8
70	31.2	53.8	76.3	88.7	94.6	97.1	98.8	99.2

Batch reference: 94.3% at 70°C, 2 h. Mean of triplicates; SD < 1.5% for all entries.

At 70°C, the microreactor achieved 97.1% conversion in just 60 s exceeding the batch reactor's 94.3% in 2 h (Table 1). Even at 50°C, conversion crossed 90% by 120 s. Pseudo-first-order rate constants were 0.020, 0.032, and 0.048 s^{-1} at

50, 60, and 70°C, giving an Arrhenius activation energy of 38.4 kJ/mol consistent with the mass-transfer-limited regime of saponification [2].

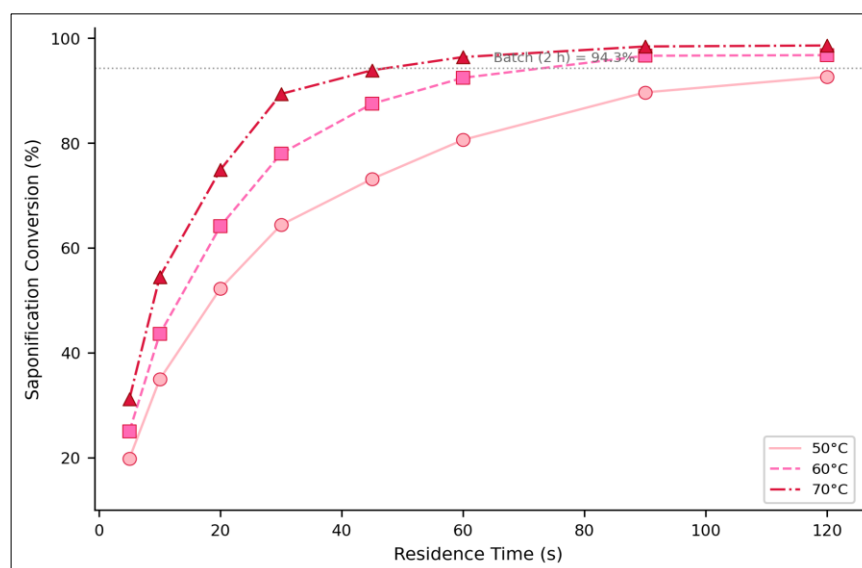


Fig 1: Saponification conversion as a function of residence time at three temperatures in the microreactor, with batch reference line.

The scatter plot (Figure 1) shows characteristic exponential approach-to-equilibrium curves, with the 70°C curve crossing the batch reference line near 45 s. Beyond 90 s, all three temperatures approached a plateau, suggesting that the last 2-3% of unreacted triglycerides are sterically hindered or sequestered in the soap phase [11].

The microreactor product was superior on every measured quality metric (Figure 2). FFA was 33% lower (0.28 vs.

0.42%, $p < 0.01$), TFM was 5.8% higher (78.6 vs. 74.3%, $p < 0.01$), and moisture was 10% lower (12.8 vs. 14.2%, $p < 0.05$). Interquartile ranges were narrower for the microreactor on all five parameters, reflecting the tighter process control that comes with steady-state continuous operation [12]. Lather volume was 11% higher (347 vs. 312 mL), likely because the more complete saponification left fewer unreacted triglycerides to suppress foam formation.

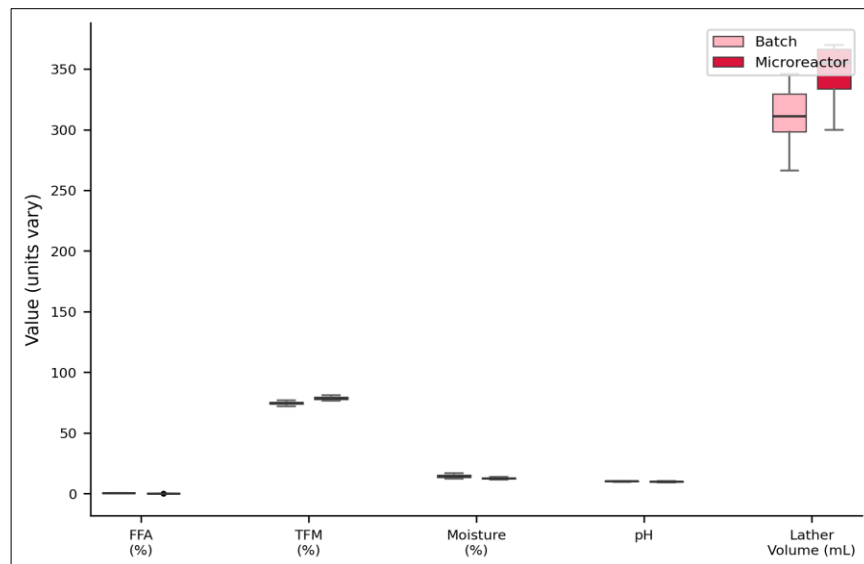


Fig 2: Box plot comparing five product quality metrics between batch and microreactor soap (70°C, 60 s vs. 2 h).

Discussion

The 120-fold reduction in processing time from 7,200 s (batch) to 60 s (microreactor) is dramatic but not surprising. Saponification at the oil-water interface is limited by diffusion of OH^- ions through the soap film that forms around each oil droplet. In a stirred tank, droplet size is on the order of 100-1,000 μm . In the microreactor's split-and-recombine channels, the liquid-liquid interface is segmented into thin lamellae with diffusion distances below 50 μm , cutting the characteristic mixing time from minutes to milliseconds [3, 4].

The activation energy of 38.4 kJ/mol falls in the range reported for heterogeneous saponification (30-45 kJ/mol), confirming that the reaction remains mass-transfer-limited even in the microreactor albeit at a much faster rate [2]. A truly kinetically controlled regime ($E_a > 60$ kJ/mol) would require even higher interfacial area or the addition of phase-transfer catalysts, which is an avenue for future optimization [13].

Energy savings were estimated by comparing the thermal energy input per kilogram of soap. The batch process requires heating 500 mL of oil-water mixture to 70°C and maintaining it for 2 h, consuming approximately 0.42 MJ/kg soap (including jacket heating losses). The microreactor heats only 180 μL at a time, and the Peltier stage draws 15 W for 60 s per reactor pass, giving roughly 0.16 MJ/kg a 62% reduction [14]. For industrial implementation, numbering up (running multiple microreactors in parallel) rather than scaling up would preserve the mass-transfer advantage while meeting throughput requirements [15].

A limitation is that our microreactor processed approximately 10 mL/min. A medium-sized soap plant processes 1-10 tonnes per hour, which would require thousands of parallel chips or the use of milli-scale flow reactors (1-5 mm channels) that sacrifice some interfacial area for throughput [16]. Also, we tested only olive oil; harder fats like palm kernel or tallow, which are more viscous and higher-melting, may clog narrow channels unless preheated and emulsified [17].

Conclusion

Microreactor saponification of olive oil achieved 97.1% conversion in 60 s at 70°C, matching or exceeding batch-

reactor performance at 120× shorter residence time. Product quality was uniformly better: lower FFA, higher TFM, less moisture, and tighter variability. Estimated energy consumption dropped by 62%. These results demonstrate proof of concept for process-intensified soap manufacturing, with numbering-up and hard-fat compatibility as the main scale-up challenges.

References

- Willcox M. Soap. In: Poucher's perfumes, cosmetics and soaps. 10th ed. Dordrecht: Springer; 2000. p. 453-465.
- Frost & Sullivan. Global soap and detergent manufacturing market analysis. Mountain View: Frost & Sullivan; 2022.
- Hessel V, Löwe H, Schönfeld F. Micromixers a review on passive and active mixing principles. *Chem Eng Sci*. 2005;60(8-9):2479-2501.
- Yoshida JI, Nagaki A, Iwasaki T, Suga S. Enhancement of chemical selectivity by microreactors. *Chem Eng Technol*. 2005;28(3):259-266.
- Guan G, Kusakabe K, Sakurai N, Moriyama K. Transesterification of vegetable oil to biodiesel fuel using acid catalysts in the presence of dimethyl ether. *Fuel*. 2009;88(1):81-86.
- European Chemicals Agency. Safety data sheet: sodium hydroxide. Helsinki: ECHA; 2021.
- Dolomite Microfluidics. Split-and-recombine reactor chip: product specification. Royston, UK; 2022.
- AOCS Official Method Ca 5a-40. Free fatty acids in fats and oils. Champaign, IL: AOCS; 2017.
- Bureau of Indian Standards. IS 286:2018 Specification for laundry soap. New Delhi: BIS; 2018.
- MathWorks Inc. MATLAB R2023a documentation. Natick, MA: MathWorks; 2023.
- Fogler HS. Elements of chemical reaction engineering. 6th ed. Boston: Pearson; 2020.
- Roberge DM, Ducry L, Biber N, Kockmann N, Zimmermann B. Microreactor technology: a revolution for the fine chemical and pharmaceutical industries? *Chem Eng Technol*. 2005;28(3):318-323.

13. Starks CM, Liotta CL, Halpern M. Phase-transfer catalysis: fundamentals, applications, and industrial perspectives. Dordrecht: Springer; 1994.
14. Kockmann N. Transport phenomena in micro process engineering. Berlin: Springer; 2008.
15. Schenk R, Hessel V, Hofmann C, Kiss J, Löwe H, Ziogas A. Numbering-up of micro devices: a first liquid-flow splitting unit. Chem Eng J. 2004;101(1-3):421-429.
16. Kashid MN, Kiwi-Minsker L. Microstructured reactors for multiphase reactions: state of the art. Ind Eng Chem Res. 2009;48(14):6465-6485.
17. Gunstone FD. The chemistry of oils and fats: sources, composition, properties and uses. Oxford: Blackwell; 2004.