



Automated in-situ production of oil-in-water emulsions using the Temperature Adjustable Membrane Emulsification Device (TAMED)

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ABSTRACT

The Temperature Adjustable Membrane Emulsification Device (TAMED) was developed and evaluated as an automated and temperature-controlled system for laboratory-scale membrane emulsification. The device replaces repeated manual syringe extrusion with an automated sequence in which two pneumatic piston pumps alternately force the batch volume through a syringe filter. Oil-in-water emulsions containing 3.5 vol% or 30 vol% medium chain triglyceride oil and stabilized with polysorbate 20 or polysorbate 80 were processed at 23 °C and 60 °C. 15 mL emulsion batches with uniform droplet sizes were produced by adding the oil and aqueous phases directly to the same syringe without prior premixing, and applying 20 passes through a composite membrane with a nominal pore size of 10 µm.

Increasing the number of passes progressively reduced the Sauter mean diameter and narrowed the droplet size distribution (span $\approx$ 1). After 20 passes, Sauter mean diameters of approximately 2.0–2.9 µm were obtained. Similar final droplet size distributions were observed with and without prior mixing. Both surfactants produced significantly smaller droplets at 60 °C than at 23 °C. Importantly, this decrease occurred despite a lower transmembrane flux at 60 °C, indicating that the reduction in interfacial tension outweighed the opposing effects of the lower continuous-phase viscosity and transmembrane flux. Surfactant type and dispersed phase content also significantly affected the final droplet size. In addition, direction-dependent differences were observed in membrane resistance and transmembrane flux.

Within the investigated process and formulation range, TAMED enabled automated in-situ preparation and repeated extrusion of small emulsion volumes under defined temperature and cycle conditions. The device therefore provides a laboratory-scale platform for investigating the effects of process and formulation parameters on membrane emulsification.

1. Introduction

Droplet size is one of the key properties of emulsions that strongly influence digestibility and bioavailability [1], rheological and sensory properties [2], physical and chemical stability [3,4], and the release profile of ingredients encapsulated within the dispersed phase. As the present study focuses on how process and formulation conditions affect the resulting emulsion structure, droplet size can serve as a suitable primary response parameter for evaluating the performance of a newly introduced automated emulsification device [5].

Premix membrane emulsification is an energy-efficient alternative process. In conventional emulsification devices the energy density is

high with around 10^5 to 10^9 J m⁻³, with a substantial fraction of the applied energy being dissipated as heat [6]. By contrast, membrane emulsification operates at lower energy densities, typically ranging from 10^3 to 10^6 J m⁻³, reducing undesirable product heating and the risk of thermal degradation of heat-sensitive ingredients [7]. In premix membrane emulsification a pre-emulsion is forced through a porous membrane, which acts as a homogenizing valve [8]. In this process, the pre-emulsion is split into numerous streams and droplet disruption in each stream occurs through geometry-mediated droplet break-up within the pores [9]. Pore size, membrane resistance R_m , and porosity ϕ affect the final droplet size. A key operating parameter of the process is the transmembrane pressure, which determines the flow velocity through

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the membrane pores [10]. In addition to process parameters, interfacial tension, the viscosities of the continuous and dispersed phases and droplet stability influence the resulting droplet size [11]. Higher surfactant concentrations can reduce the dynamic interfacial tension during rapid droplet formation, resulting in smaller droplets and shorter droplet formation times down to approximately 10 ms [12].

Extrusion through a syringe filter is an attractive method of membrane emulsification since no special device is needed, apart from a syringe filter, which is disposable, and the emulsion can be made under aseptic conditions [10]. To produce defined particle size distributions of liposomes, Olson et al. introduced multiple extrusions of liposomal suspension through a membrane [13,14]. The process was expanded to conventional emulsions, nanoemulsions, and solid lipid nanoparticles [15]. The number of applied membrane extrusion passes ranged from 5 for conventional emulsions [16] to 20 for large unilamellar vesicles [17] and 21 for lipid nanoemulsions [14]. Typically, the transmembrane flux is higher in subsequent passes [9], due to reduced energy dissipation within the membrane when smaller droplets move through the pores.

Droplet breakup in membrane emulsification is promoted by lower interfacial tension, higher continuous-phase viscosity and higher flow velocity. Interfacial tension can be reduced by surfactant type and concentration or temperature, which supports droplet deformation and breakup [12,18,19]. Link et al. investigated the breakup of elongated droplets into two daughter droplets of equal size in a T-bifurcation microchannel and concluded that a droplet will break more easily at higher viscosity of the continuous phase, lower interfacial tension and higher flow rate [20]. This model can be supplemented by Hunter and Frisken's model postulating that a lubricating film of the continuous phase forms between the droplet plug flowing through the pore and the pore wall [21]. If the viscosity of the continuous phase is high and the interfacial tension is low, the lubricating film is thick, leading to long and narrow droplet plugs which are easier to break into smaller pieces.

Double syringe membrane emulsification has been used for the production of pharmaceuticals, micron-sized emulsion droplets [22], emulsion-templated porous polymer particles [23], polydimethylsiloxane powder [24], and polylactide microspheres [25]. Recently, double-emulsion templates for nanoparticle-stabilized water-in-air-in-water antibubbles have been produced using manually operated membrane extrusion [26], which highlights the broad application potential of this process.

Joseph, Gehrman and Bunjes showed that an emulsion premix can be prepared from initially separated oil and aqueous phases using two disposable syringes, an approach also discussed in the review by Nazir and Vladisavljević [14,15,27]. However, this syringe-based process was operated manually and did not combine in-situ preparation with automated, temperature-controlled repeated extrusion. Building on this concept, the present study implements in-situ emulsion preparation in an automated setup with controlled process temperature. To the best of our knowledge, automated and temperature-controlled double syringe membrane emulsification was only used by Reiner et al. to keep the dispersed phase molten during the production of solid lipid microparticles [28]. In this work, the novel Temperature Adjustable Membrane Emulsification Device (TAMED) was used for automated and temperature-controlled repeated extrusion of oil-in-water emulsions through a syringe filter. Its pneumatically controlled operation provides standardized extrusion under predefined temperature, pressure, and cycle conditions. This allows the effects of process temperature, transmembrane pressure, surfactant type, dispersed phase content, number of passes, and flow direction through the composite membrane to be investigated systematically. Temperature control further enables temperature-dependent changes in interfacial tension and phase viscosities to be related to transmembrane flux and droplet size. The study therefore demonstrates TAMED as an experimental platform for investigating process–formulation relationships in membrane emulsification. Using this platform, the direction-dependent transport behavior of the membrane was investigated, revealing direction-dependent differences

in membrane resistance and transmembrane flux. Moreover, smaller droplets were obtained at 60 °C despite the lower transmembrane flux, indicating that the reduction in interfacial tension can outweigh the opposing effects of lower continuous-phase viscosity and lower flux on droplet breakup. To the best of our knowledge, these effects have not previously been reported for repeated membrane extrusion.

2. Materials and methods

2.1. Materials

Medium chain triglyceride oil (MCT oil, Witarix, C8/C10 = 60/40) was provided by IOI Oleo GmbH (Germany). Polysorbate 20 (polyoxyethylene 20 sorbitan monolaurate, Tween 20) and polysorbate 80 (polyoxyethylene 20 sorbitan monooleate, Tween 80) emulsifiers were bought from Carl Roth (Germany). All materials were used without further purification. All samples were prepared with ultrapure water (Thermo Scientific Barnstead MicroPure, Waltham, USA).

2.2. Process characterization

2.2.1. Temperature adjustable membrane emulsification device (TAMED)

The continuous phase (2 wt% aqueous polysorbate solution) was prepared by dissolving pure polysorbate in water under stirring at 23 °C. For preparing an emulsion with a dispersed phase content of $\varphi = 30$ vol % by in-situ membrane emulsification, 10.5 mL of the polysorbate solution and 4.5 mL of MCT oil were added directly to the same 20 mL syringe without prior mixing. The second syringe was initially empty. For preparing an emulsion with $\varphi = 3.5$ vol%, 14.475 mL of the polysorbate solution and 0.525 mL of MCT oil were added to the same syringe without prior mixing. For preparing emulsions by premix membrane emulsification, the premix was made outside the TAMED using a high-performance dispersing instrument (T25 digital Ultra-Turrax, IKA, Staufen, Germany) at 18,000 rpm for 60 s resulting in the peripheral velocity of $v = 12.25 \text{ m s}^{-1}$. The prepared premix was transferred into a 20 mL syringe (Omnifix, B. Braun SE, Melsungen, Germany), which was then placed in the TAMED. For the experiments at 60 °C, the loaded syringes were pre-heated to 60 °C in a water bath before processing in the TAMED. During these experiments, the heat guns were set to 120 °C to limit heat loss from the syringes during the extrusion process. The heat guns were operated at a constant setting and did not directly control the liquid temperature. The liquid temperature was monitored separately with a custom-made connector between the syringes using a thermocouple. During the heated experiments, the monitored liquid temperature was 60 ± 2 °C. Experiments at room temperature were performed without active heating or cooling. The TAMED setup can be seen in Fig. 1. The syringes and the syringe filter were connected with a connector (Luer connector Rotilabo male/male, Karlsruhe, Germany). The assembled syringes and syringe filter were then placed in TAMED. Compressed air from the laboratory supply was controlled by the control unit and sent alternately to the right and left pneumatic piston pumps. The active piston pump pushed the plunger of the filled syringe and forced the liquid through the syringe filter into the empty syringe. After each pass, the control unit switched the air supply to the other piston pump and reversed the flow direction. After 20 passes the resulting emulsion was cooled to room temperature and analyzed. Each experiment was repeated three times. All emulsions were freshly prepared on the day of the measurements.

Temperature and pressure between the syringes were measured separately. To record the pressure or temperature, a pressure or temperature sensor was connected to the custom-made connector placed between the two syringes, Fig. 1(b). The transmembrane pressure was equal to the gauge pressure in the syringe upstream of the membrane since the gauge pressure in the downstream syringe was zero. The pressure in the upstream syringe (p_{syringe}) was defined as the peak pressure during each extrusion cycle and was recorded with a camera.

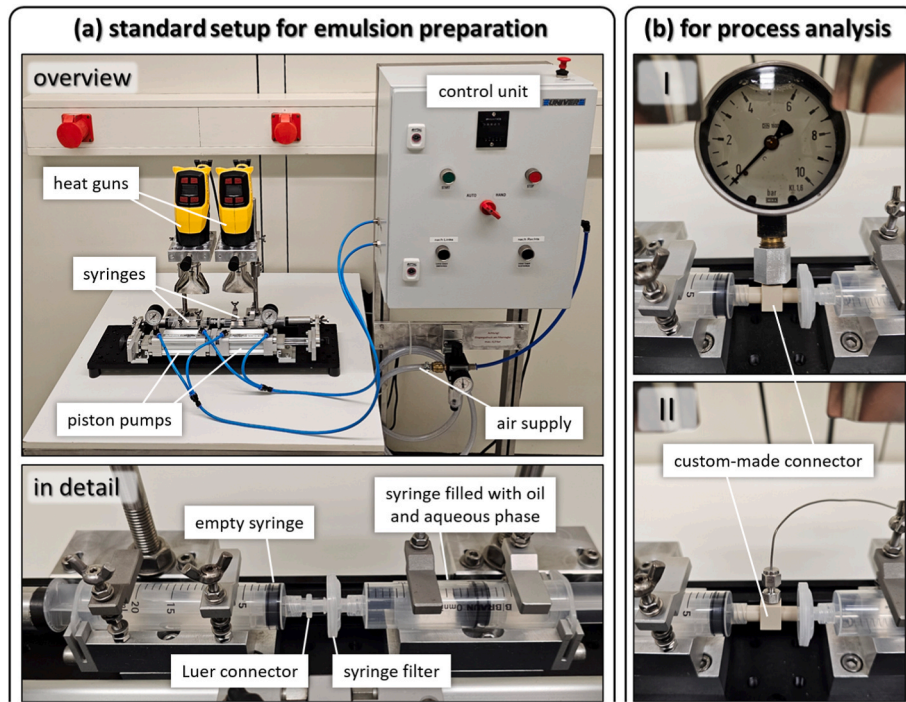


Fig. 1. Temperature Adjustable Membrane Emulsification Device (TAMED): (a) standard setup for emulsion preparation and flux measurement; (b) custom-made connector used for separate pressure (I) and temperature (II) measurements. Flux was determined from video recordings of the syringe-plunger movement.

The droplet sizes in these experiments were not measured because of the higher dead volume between the two syringes and different flow patterns. For pressure buildup and temperature, these influences were considered negligible.

To assess whether prior premixing affected the final droplet size distribution, samples prepared using an Ultra-Turrax premix were compared with samples in which the oil and aqueous phases were added directly to the same syringe without prior mixing. Both samples were subsequently processed in TAMED for 20 membrane passes under identical conditions. Similar final droplet size distributions were obtained, with Sauter mean diameters of $d_{\text{Sauter, in-situ}} = 4.02 \pm 0.20 \mu\text{m}$ without prior premixing and $d_{\text{Sauter, premix}} = 4.00 \pm 0.32 \mu\text{m}$ for the Ultra-Turrax premix, as shown in Fig. S1. Therefore, all subsequent experiments were performed by adding the oil and aqueous phases directly to the same syringe without a premixing step.

2.2.2. Membrane characterization

Experiments were carried out using Acrodisc 25 mm syringe filters with a Versapor membrane (Cytiva, USA). The maximum operating temperature specified by the manufacturer is 55 °C. This filter is composed of a polypropylene housing and composite membrane with a pore size of 10 μm containing hydrophilic acrylic copolymer cast on nonwoven nylon support, see Fig. 2. On SEM images, thick nylon fibers can be distinguished from much finer acrylic copolymer fibers. The membrane does not show a clearly separated skin layer and support layer. However, the nylon fibers appear to be more densely packed on the outlet side than on the inlet side. This local difference may result from the manufacturing process and may cause direction-dependent flow through the filter.

The membrane porosity ϕ at 23 °C and 60 °C was calculated using Equation (1):

$$\phi = \frac{m_{\text{water}}}{\rho_{\text{water}} V_{\text{total}}} \quad \text{Eq. (1)}$$

where m_{water} is the mass of water that has filled the membrane pores ($m_{\text{water, 23 °C}} = 21 \pm 0.5 \text{ mg}$ and $m_{\text{water, 60 °C}} = 23 \pm 1 \text{ mg}$), ρ_{water} is the

density of water ($\rho_{\text{water, 23 °C}} = 997.8 \text{ kg m}^{-3}$ and $\rho_{\text{water, 60 °C}} = 982.2 \text{ kg m}^{-3}$) [29], and $V_{\text{total}} = \pi r^2 h$ is the total volume of membrane disc with the radius $r = 10.7 \text{ mm}$ and the height h ($h_{23 °C} = 0.15 \text{ mm}$ and $h_{60 °C} = 0.151 \text{ mm}$). The mean membrane porosity based on three repeated measurements was $\phi_{23 °C} = 0.39 \pm 0.01$ and $\phi_{60 °C} = 0.43 \pm 0.02$.

To determine the membrane resistance of the syringe filters, R_m , an empty syringe housing with an inner diameter d_{syringe} was attached to the syringe filter, filled with 20 mL of water, and the time t needed for the volume of water in the barrel to decrease from 15 mL to 5 mL was measured. Fig. 3 shows a schematic setup of this experiment. The membrane resistance R_m was calculated from Darcy's law and the hydrostatic pressure difference according to Equation (2):

$$R_m = t \cdot \frac{A_{\text{membrane}} \rho_{\text{water}} g}{A_{\text{syringe}} \eta_{\text{water}} \ln\left(\frac{h_2}{h_1}\right)} \quad \text{Eq. (2)}$$

where h_2 and h_1 are the initial and final level of water in the barrel, A_{syringe} and A_{membrane} are the cross-sectional area of the syringe barrel and membrane disc, ρ_{water} and η_{water} are the density and viscosity of water, and g is the acceleration due to gravity. The measurements were carried out in triplicates at 23 °C and 60 °C. The membrane resistance was calculated in both flow directions.

2.2.3. Modelling of droplet disruption

During the first pass, the layered oil and aqueous phases were pushed together from one syringe through the membrane into the empty second syringe. This first pass formed the initial emulsion and did not match the conditions assumed by Link et al., where droplets are already present before entering the membrane pores [20]. The Link et al. model was therefore used to describe droplet deformation and breakup from the second pass onwards, see parameters for the investigated conditions summarized in Table 1. During these later passes, existing droplets can stretch inside the connected membrane pores and break at pore junctions, depending on the capillary number and their initial extension. Because of the large initial droplet size in pass 2 caused by using in-situ

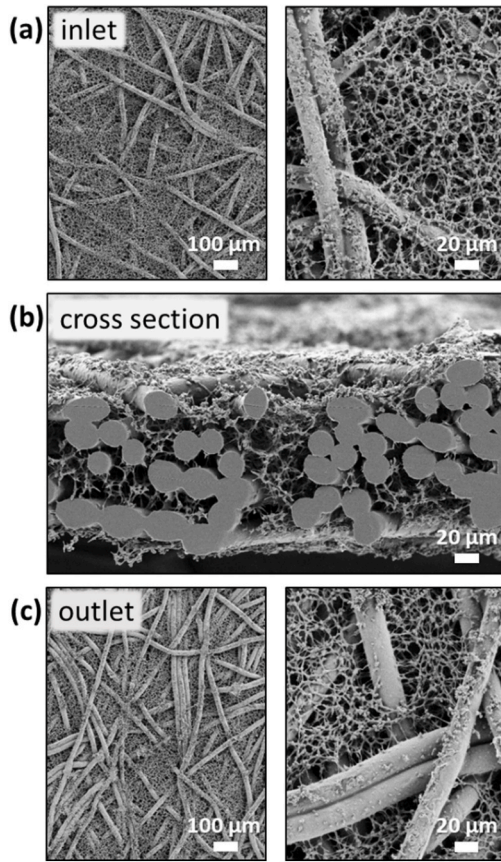


Fig. 2. Scanning electron microscopy (SEM) images of the Versapor composite membrane: (a) inlet surface at $\times 200$ (left) and $\times 1000$ (right); (b) cross-section at $\times 200$; and (c) outlet surface at $\times 200$ (left) and $\times 1000$ (right). The fine structure is the acrylic copolymer, and the coarse fibers are the nonwoven nylon support. The nylon fibers appear to be more densely packed on the outlet side than on the inlet side.

emulsification, droplets moving inside membrane pores are deformed into elongated cylinder shapes and prone to passive breakup in pore junctions. The membranes used in this study contained highly interconnected pores which were approximated as a series of pore junctions for applying the T-junction breakup model [20,27]. Droplets are stable in straight pore sections, but they can break into two smaller droplets at each junction depending on the capillary number Ca and the initial extension ε_0 of the droplets. The capillary number Ca indicates the relative effect of viscous drag forces in the continuous phase versus surface tension forces acting across an interface between two immiscible liquids and thus depends on the dynamic viscosity of the continuous phase η_c , the mean pore velocity ν (which can be approximated by the emulsion velocity) and the interfacial tension γ :

$$Ca = \frac{\eta_c \nu}{\gamma} \quad \text{Eq. (3)}$$

The mean pore velocity ν can be calculated as $\nu = J / \Phi$, where J is the volumetric transmembrane flux and Φ is the membrane porosity. The initial droplet extension ε_0 depends on the droplet length l_0 upstream of the junction and the pore width w_0 :

$$\varepsilon_0 = \frac{l_0}{\pi w_0} \quad \text{Eq. (4)}$$

For cylindrical pores, $w_0 = 2R_p$, where R_p is the pore radius. Based on Link et al.'s model, the droplets passing through the pores do not break for $Ca < Ca_{cr}$, while they break into two daughter droplets for $Ca > Ca_{cr}$, where Ca_{cr} is the critical capillary number, which depends on

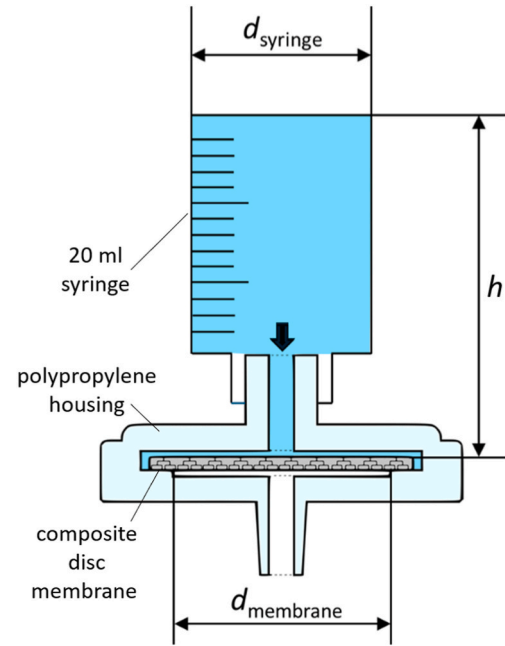


Fig. 3. Schematic diagram of the syringe filter membrane resistance measurement. The membrane pores of the composite membrane in the polypropylene housing are represented as parallel channels with bifurcations. Forward flow is defined as flow from the inlet side to the outlet side.

ε_0 . The higher the ε_0 value is, the smaller Ca_{cr} becomes. If the initial extension ε_0 is sufficiently high, the droplet will always break no matter how small the droplet velocity is. Link et al.'s model assumes that large droplets touch pore walls as they travel down the pore. In Hunter et al.'s model, large droplets traveling down the pore are separated from the pore wall by a lubrication layer of the continuous phase of thickness h^* [21]. The diameter of a droplet spherocylinder (cylinder with hemispherical ends) moving through the pore is equal to: $d_{\text{droplet in pore}} = 2(R_p - h^*)$. The h^* value depends on the capillary number Ca and the pore radius, R_p :

$$h^* = 2.05 R_p Ca^{2/3} \quad \text{Eq. (5)}$$

However, Eq. (5) is strictly valid for disruption of lipid vesicles. The capillary numbers Ca and the corresponding lubrication layer thicknesses h^* applied in this study are shown in Table 1.

2.2.4. Calculation of volumetric flux

The volumetric flux J was determined by recording 20 consecutive passes through the membrane. To do this, videos of the process were analyzed for the plunger velocity and the volumetric flow rates calculated. The volumetric flow rate $\dot{V}$ of the emulsion was calculated based on the time required for each retraction of the syringe plunger and the batch volume and J was then calculated as follows:

$$J = \frac{\dot{V}}{A} \quad \text{Eq. (6)}$$

With A being the cross-sectional area of the membrane. The mean J value in all experiments was calculated based on passes 1, 2, 19 and 20 and this value was compared with the mean J value calculated from all passes. For example, for the emulsion produced at 23 °C and 300 kPa using polysorbate 20, $\bar{J} = 71.76 \pm 5.70 \text{ L s}^{-1} \text{ m}^{-2}$ calculated from passes no. 1, 2, 19, and 20, as compared to $73.73 \pm 5.67 \text{ L s}^{-1} \text{ m}^{-2}$ if calculated from all 20 passes. A video showing the production of this emulsion is in the supplementary materials. Although the first pass is not described by the model of Link et al., the mean flux based on passes 1, 2, 19, and 20 was retained as a representative process value for the model calculations

Table 1

Capillary numbers of the continuous phase and lubrication-layer thickness calculated for the investigated emulsions using the model of Hunter and Frisken.

dispersed phase content φ	30 vol%				3.5 vol%
Emulsifier	polysorbate 20		polysorbate 80		polysorbate 20
temperature $T/^{\circ}\text{C}$	23 ± 3		60 ± 2		23 ± 3
capillary number $\text{Ca}/-$	0.037 ± 0.003		0.043 ± 0.003		0.031 ± 0.001
thickness of lubrication layer $h^*/\mu\text{m}$	1.131 ± 0.060		1.265 ± 0.062		1.017 ± 0.028

[20]. The comparison with the mean flux calculated from all 20 passes was used to check whether the selected-pass average represented the overall process.

2.3. Emulsion characterization

2.3.1. Droplet size

The droplet size distributions of emulsions were determined by a laser diffraction particle size analyzer in a flow measuring cell (Horiba LA-940, Retsch Technology, Haan, Germany) at the same day as they were produced with membrane emulsification. Emulsions were diluted prior to the analysis and measured three times at room temperature. A sample was taken after each pass to study the influence of individual passes on the droplet size, see Section 3.2.2. The mean droplet size was expressed using the volume median diameter, $d_{50,3}$ and the Sauter mean diameter, d_{Sauter} , while the width of particle size distribution was characterized using the span value calculated as: $\text{span} = (d_{90,3} - d_{10,3})/d_{50,3}$. ANOVA and t-tests were run at the significance level of 0.05 to assess whether different operating and formulation factors (temperature, surfactant, and dispersed phase content) caused differences in mean Sauter diameter based on nine particle size measurements. All statistical tests were performed using Excel's Data Analysis Tools "t-test: Two-Sample Assuming Equal Variances" (see Table 2) and were additionally evaluated using "ANOVA Single Factor" (see Table 3). The statistical results are listed in Tables 2 and 3.

2.3.2. Light microscopy

The emulsions were observed with a light microscope (Eclipse LV100ND, Nikon, Shinagawa, Japan). Pictures of oil droplets in the emulsions were taken using a camera lens with 50x magnification (TU Plan ELWD 50x/0.60B OFN25 WD11, Nikon, Shinagawa, Japan).

2.3.3. Rheological properties

The dynamic viscosity was measured using a double-gap measuring system (DG26.7SN47667, 3.8 mL per sample) on a Physica MCR 101 rheometer (Anton Paar, Graz, Austria). The viscosity was measured on the day the continuous phase and the emulsion were produced. 20 data

Table 2

Two-sample t-test results for differences in d_{Sauter} .

factor	comparison	df	t-value	p-value	outcome
emulsifier type	23 °C (polysorbate 20 vs. polysorbate 80)	16	−5.81	2.66×10^{-5}	significant*
	60 °C (polysorbate 20 vs. polysorbate 80)	16	−4.62	2.82×10^{-4}	significant*
temperature	polysorbate 20 (23 °C vs. 60 °C)	16	9.30	7.46×10^{-8}	significant*
	polysorbate 80 (23 °C vs. 60 °C)	16	9.55	5.20×10^{-8}	significant*
dispersed phase content	polysorbate 20 (3.5 % vs. 30 % at 23 °C)	16	14.61	1.13×10^{-10}	significant*

*p < 0.001.

Table 3

One-way ANOVA results for differences in d_{Sauter} .

factor	comparison	F	p-value	η^2	outcome
emulsifier type	23 °C (polysorbate 20 vs. polysorbate 80)	33.76	2.66×10^{-5}	0.678	significant*
	60 °C (polysorbate 20 vs. polysorbate 80)	21.37	2.82×10^{-4}	0.569	significant*
temperature	polysorbate 20 (23 °C vs. 60 °C)	86.50	7.46×10^{-8}	0.844	significant*
	polysorbate 80 (23 °C vs. 60 °C)	91.18	5.20×10^{-8}	0.851	significant*
dispersed phase content	polysorbate 20 (3.5 % vs. 30 % at 23 °C)	213.56	1.13×10^{-10}	0.930	significant*

*p < 0.001; η^2 = effect size (large effect: $\eta^2 > 0.14$); F(between, within) = (1, 16) for all comparisons.

points were recorded with the time difference between the data points of 15 s. Shear rate was increased from 1 s^{-1} to 1000 s^{-1} . The viscosity values were determined at 1000 s^{-1} . The measurements were carried out in triplicates.

2.3.4. Interfacial tension

The interfacial tension γ was measured using an optical contact angle measuring and contour analysis system (OCA15LJ, DataPhysics Instruments GmbH, Filderstadt, Germany). The specified volume of 5 μL medium chain triglyceride oil was held as a hanging droplet using a dispensing needle in a glass cuvette filled with the 2 wt% aqueous polysorbate solution. The glass cuvette was placed in a temperature-controlled chamber (TFC100Pro, DataPhysics Instruments GmbH, Filderstadt, Germany) in which the temperature was regulated using a thermostat (Julabo F12, Sigma Aldrich, St. Louis, USA). The drop contour was then recorded using the measuring device's camera and the interfacial tension was measured over time until equilibrium was established. The measurement was carried out three times and the measured values are given in Table 4.

Table 4

Interfacial tension between medium chain triglyceride (MCT) oil and aqueous 2 wt% polysorbate 20 and polysorbate 80 solution at 23 °C and 60 °C.

Temperature $T_{\text{cuvette}}/^{\circ}\text{C}$	interfacial tension $\gamma/\text{mN} \cdot \text{m}^{-1}$	
	polysorbate 20	polysorbate 80
23	5.52 ± 0.01	4.92 ± 0.01
60	2.37 ± 0.02	0.89 ± 0.01

3. Results and discussion

3.1. Influence of temperature and flow direction on membrane resistance

The syringe filter membrane resistance R_m for different temperatures and flow directions is given in Table 5. At 23 °C, the forward and backward resistance values overlapped within their standard deviations, whereas at 60 °C, the resistance was substantially higher in backward flow. With increasing temperature from 23 °C to 60 °C, the average overall resistance approximately doubled from $1.09 \cdot 10^8 \text{ m}^{-1}$ to $2.23 \cdot 10^8 \text{ m}^{-1}$.

The membrane resistance depended on flow direction and temperature. The SEM images suggest small differences in the arrangement of the nylon fibers between the inlet and outlet sides. These differences may create different flow paths in the two directions. However, the fiber arrangement alone cannot explain why a directional difference was observed at 60 °C but not at 23 °C. At higher temperature, structural changes in either the composite membrane or the polypropylene housing may have altered the effective flow paths. Since the membrane structure was not examined at 60 °C, this remains a possible explanation. In addition, the experiments at 60 °C were carried out 5 °C above the maximum operating temperature specified by the manufacturer. This may have affected the membrane structure and resistance. Overall, no clear directional difference was evident at 23 °C, whereas a pronounced direction-dependent resistance was observed at 60 °C. The mechanism responsible for this temperature-dependent behavior could not be identified conclusively. The corresponding effect of flow direction on emulsion flux is discussed in Section 3.2.1.

3.2. Influence of pass number

3.2.1. Influence of pass number on transmembrane volumetric flux

The variation of the transmembrane volumetric flux J with the pass number for the production of an emulsion using polysorbate 20 at 23 °C and 400 kPa, corresponding to a measured syringe pressure of $300 \pm 30 \text{ kPa}$, is shown in Fig. 4. The layered aqueous and oil phases in the first syringe were pushed together through the membrane into the empty second syringe, marking the first pass (flowing in the intended direction for the syringe filter, defined as forward use). The resulting coarse emulsion was then pushed back through the membrane into the first syringe, marking the second pass (defined as backward use). This alternating process was repeated for a total of 20 passes. The transmembrane flux increased slightly with pass number and was consistently lower in forward than in backward flow.

The increase in transmembrane flux with pass number is consistent with previous studies [30,31]. As the number of passes increases, flux improves because the droplets get smaller, and less energy is needed for droplet deformation and breakup [32]. Transmembrane pressure is mainly used to overcome flow resistances in the pores and interfacial tension forces, which counteract the deformation and disruption of large oil drops into smaller droplets. This behavior can be described by the following pressure balance:

$$P_{\text{syringe}} = \Delta P_{\text{friction}} + \Delta P_{\text{break}} \quad \text{Eq. (7)}$$

The pressure drop due to friction loss between the emulsion flow and pore walls ($\Delta P_{\text{friction}}$) is proportional to the transmembrane flux, while

Table 5

Membrane resistance R_m at temperatures 23 °C and 60 °C for forward and backward flow directions.

	membrane resistance $R_m/10^8 \text{ m}^{-1}$	
	at 23 °C	at 60 °C
forward flow	1.15 ± 0.15	1.82 ± 0.07
backward flow	1.02 ± 0.05	2.64 ± 0.24
average	1.09 ± 0.13	2.23 ± 0.48

the expenditure of pressure for droplet breakup (ΔP_{break}) is proportional to the increase in the interfacial area which is inversely proportional to the droplet diameter. From the second pass onwards, when a coarse emulsion was already present, the measured syringe pressure was 300

$\pm 30 \text{ kPa}$ and was treated as approximately constant for the following pressure-balance consideration:

$$P_{\text{syringe}} = \eta_{e,i} (R_m + R_{f,i}) J_i + C \phi \gamma \left(\frac{1}{d_{\text{sauter},i}} - \frac{1}{d_{\text{sauter},i-1}} \right) = \text{constant} \quad \text{Eq. (8)}$$

where C is a constant independent of the number of passes, R_m is the hydrodynamic resistance of the membrane, and $\eta_{e,i}$, $R_{f,i}$, J_i , and $d_{\text{sauter},i}$ are the emulsion viscosity, fouling resistance, transmembrane flux, and final mean Sauter diameter in the i th pass through the membrane. The fouling resistance occurs due to the accumulation of oil phase on the membrane surface (external fouling) and inside the membrane pores (internal fouling). Within the range described by Eq. (8), the maximum ΔP_{break} value is expected in the second pass, since then the greatest proportion of pressure energy is spent on droplet breakup leading to the greatest decrease in d_{sauter} . In the subsequent passes, the mean Sauter diameter shows a progressively smaller decrease, therefore, $\Delta P_{\text{break}} \rightarrow 0$ as $i \rightarrow \infty$, and $J_i \rightarrow J_{\text{max}}$. In Fig. 5, for $i > 17$, the mean droplet size reaches a constant value ($d_{\text{sauter},i} \approx d_{\text{sauter},i-1}$), so $\Delta P_{\text{break}} = 0$ and $P_{\text{syringe}} = \Delta P_{\text{friction}}$. Therefore, from the pass no. 17 onwards, the inlet pressure is predominantly used to overcome friction due to membrane and potential fouling resistances and the flux J_i reaches the maximum value for the given inlet pressure. The maximum flux, i.e. the limiting flux that can be reached after many passes, depends on the emulsion viscosity, hydrodynamic resistance of the membrane, and the fouling resistance. Emulsion viscosity mainly depends on temperature and the dispersed phase content, but the mean droplet size also has some impact, with smaller droplet sizes generally leading to higher viscosity. The transmembrane flux varied with the flow direction and was lower in forward than in backward flow at 23 °C. However, this difference cannot be explained by the membrane resistance, because the forward and backward resistance values overlapped within their standard deviations at 23 °C. Moreover, flow direction and pass number were coupled in the alternating sequence: each backward pass followed a forward pass, while the flux increased slightly with pass number. Therefore, the observed flux difference cannot be attributed clearly to membrane orientation. In addition, emulsion droplets may partly block narrow flow paths or accumulate at the membrane entrance. Since droplet accumulation and membrane fouling were not measured directly, this remains a possible explanation.

3.2.2. Influence of pass number on droplet size

The droplet sizes and microscopic images of emulsion droplets were assessed directly after production. Micrographs of droplets from selected passes, characteristic droplet diameters ($d_{10,3}$, $d_{50,3}$, and $d_{90,3}$) and span values for the emulsion produced at 23 °C and $p_{\text{syringe}} = 300 \text{ kPa}$ using polysorbate 20 are shown in Fig. 5.

Increasing the number of passes reduced all characteristic droplet diameters and the span value. For all characteristic diameters, the deviations of the data points from the corresponding trend line were smaller with increasing the pass number, providing additional evidence that the emulsion became more homogeneous. Therefore, the applied pressure (300 kPa) was insufficient to finely break up all coarse emulsion particles in a single pass. However, after 20 passes, highly uniform emulsion droplets were obtained, as shown in Fig. 5(a) and (b). This highlights the importance of repeated emulsification passes in achieving homogeneity, particularly when operating at lower driving pressures.

The final median droplet size $d_{50,3}$ of approximately $2.5 \mu\text{m}$ achieved using the membrane with a pore size of $10 \mu\text{m}$ is consistent with the literature, where the final droplet size after multiple extrusions was

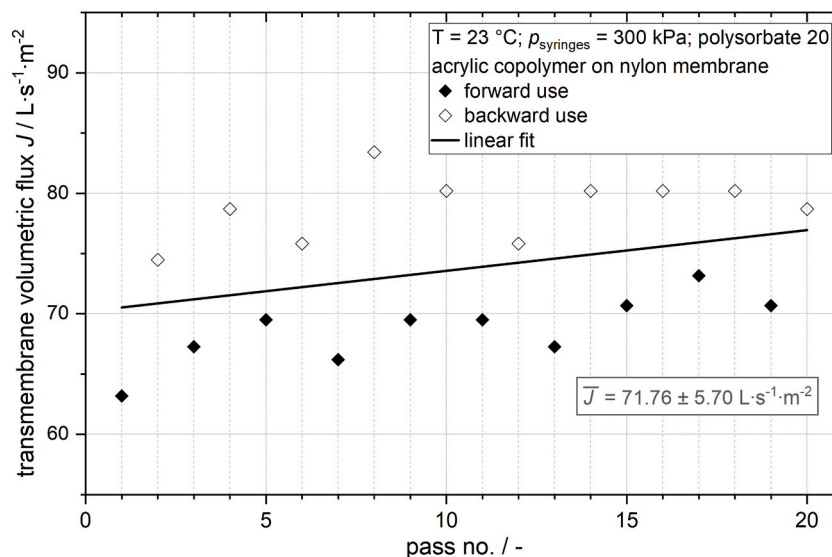


Fig. 4. The effect of the number of passes on the transmembrane volumetric flux $\bar{J}$ at 23 °C, piston pump pressure of 400 kPa, and ϕ of 30 vol% using a membrane with a pore size = 10 μm . The surfactant was polysorbate 20. Filled squares represent forward flow, empty squares represent backward flow, and the solid line represents the linear fit. The average flux of $71.76 \pm 5.70 \text{ L s}^{-1} \text{ m}^{-2}$ was calculated based on passes 1, 2, 19, and 20. Each data point represents the flux measured during one membrane pass in a single extrusion experiment.

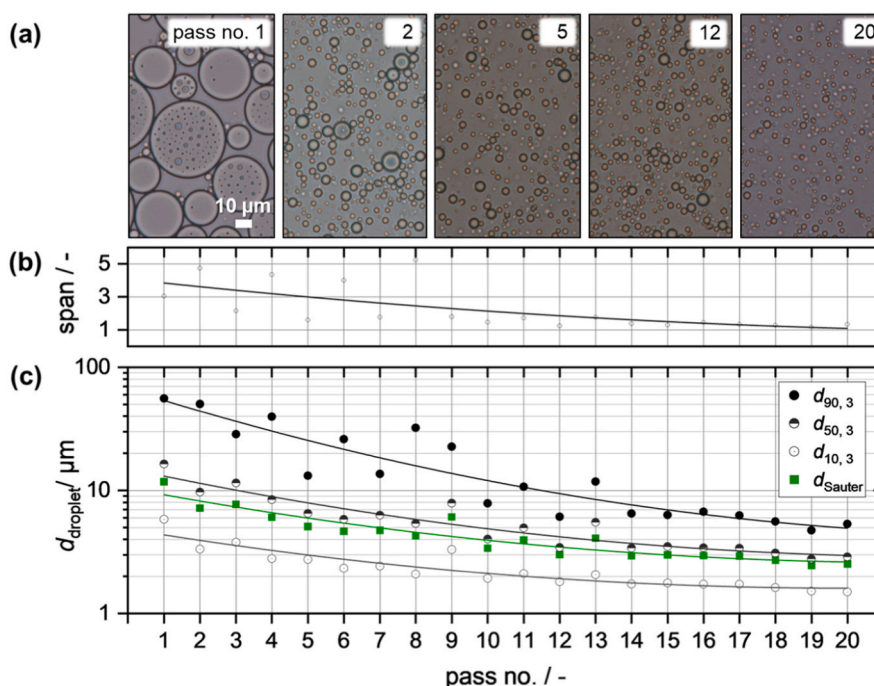


Fig. 5. Influence of the number of passes through the membrane (pore size = 10 μm) on emulsification process performed in the Temperature Adjustable Membrane Emulsification Device (TAMED) at 23 °C under a transmembrane pressure of $300 \pm 30 \text{ kPa}$ using polysorbate 20 surfactant: (a) micrographs of emulsion droplets collected after selected pass numbers; (b) the span value as a function of the pass number; (c) the characteristic droplet diameters as a function of the pass number. With increasing pass number, the droplet sizes decreased but after 17 passes the differences in droplet sizes became negligible. The droplet size distributions after the first, second, fifth, 12th and 20th pass can be seen in Fig. S2.

typically smaller than the pore size for the pore size of about 10 μm [21, 30,32–35]. For example, after repeatedly passing emulsions containing 5 % n-hexadecane through a metal sieve at 200 kPa, Nazir et al. (2011) achieved the final droplet size of 6 μm , which was less than half the pore width of the sieve (13.2 μm).

Also, a decrease in droplet size with increasing the number of passes has been widely observed. The most significant reduction in droplet size was found after the first pass, with subsequent passes leading to more

gradual decreases [21,30,32–35]. For instance, Hunter et al. reported a decrease in vesicle diameter from 173 nm to 110 nm by extruding a suspension of large vesicles through a polycarbonate membrane with a pore size of 100 nm at $\sim 1400 \text{ kPa}$ and 30 °C [21]. The most significant decrease in vesicle size from 173 nm to 130 nm was achieved in the first pass with a gradual decrease in size upon additional extrusions and plateauing after 10 passes. Also, Hwang et al. observed that the size of liposomes decreased with increasing the number of passes. Liposomes

with the final size between 0.1 and 1 μm were achieved by extruding primary liposomes 5 times through a SPG membrane with a pore size between 0.2 and 2 μm at 400 kPa [33].

The first pass forms a coarse emulsion from the layered oil and aqueous phases. The further decrease in droplet size from the second pass onwards can be explained by the work of Link et al. on droplet splitting in microfluidic T-junctions [20]. At the beginning of the second pass, the droplets are still larger than the mean pore size and the droplet extension (ε_0) given by Eq. (4) is high. If the droplet extension is sufficiently high, the droplet will break at the junction even at the smallest upstream velocity. It means that droplets will break even in the largest pores. With increasing the number of passes, the mean droplet size becomes smaller and smaller and the droplet breakup at the pore junctions is increasingly more difficult until finally no further decrease in droplet size is possible. Probably, in subsequent passes, droplets can break only in the smallest membrane pores, where the capillary number has the maximum value leading to the maximum lubrication layer thickness and the most unstable interface. As the number of passes increases in our experiments, the transmembrane flux rises, leading to even higher velocity within the pores and, consequently, an increasing capillary number Ca . After many passes through the membrane, the large proportion of droplets become significantly smaller than the pore width ($l_0 \ll w_0$) and their length in the pores approaches their diameter ($l_0 \approx d_{\text{droplet}}$). As a result, the initial elongation ($l_0/\pi w_0$) becomes much smaller than $1/\pi$, preventing droplet breakup even at high capillary numbers (smallest pores).

3.3. Influence of temperature and emulsifier on droplet size

The Sauter mean diameters of emulsions produced using different polysorbates at 23 °C and 60 °C are compared in Fig. 6 to investigate the influence of temperature on the droplet size. The one-way ANOVA and t-tests performed at the significance level of 0.05 showed significant differences in the Sauter mean diameters. Under the investigated operating conditions, polysorbate 20 led to smaller droplet sizes than polysorbate 80 at both temperatures. Because the nominal piston-pump pressures differed between the polysorbate 20 and polysorbate 80 experiments,

the observed differences cannot be attributed exclusively to surfactant type. Nevertheless, both surfactants produced smaller droplets at 60 °C than at 23 °C. All differences were statistically significant ($p < 0.05$). Both surfactants are non-ionic, but they differ in their solubility in water, hydrophilicity, and emulsification properties due to their different fatty acid components. Polysorbate 20 has an HLB (Hydrophilic-Lipophilic Balance) value of 16.7, while polysorbate 80 has an HLB value of 15. It is generally better to use a surfactant with a higher HLB (Hydrophilic-Lipophilic Balance) value for oil-in-water (O/W) emulsions. For both emulsifiers, a significant decrease in droplet size was observed with increasing temperature, which can be attributed to lower interfacial tension of both surfactants at higher temperature. As the temperature goes up, the intermolecular forces at the interface become weaker leading to a lower interfacial tension. At lower interfacial tension, droplets are more prone to break as they pass through the membrane. A good indicator of the likelihood of droplets to break is the capillary number of the continuous phase in a pore (Ca), given by Eq. (3). With higher Ca and ε_0 values, the droplet breakup is more likely to occur [20]. As the temperature rises from 23 °C to 60 °C, Ca increases from 0.037 to 0.043 for the polysorbate 20 emulsion and from 0.048 to 0.156 for the polysorbate 80 emulsion (Table 1). A higher capillary number leads to a greater thickness of the lubrication layer h^* based on Equation (5) [21]. This increase in h^* was observed for polysorbate 20 emulsions (from 1.13 μm to 1.27 μm) and to a higher extent for the polysorbate 80 emulsions (from 1.35 μm to 2.98 μm). A thicker lubrication layer results in a higher initial droplet extension ε_0 , meaning the droplets are more elongated prior to breakup and less stable due to higher surface area per unit volume; thus, they are more likely to break.

Unexpectedly for both surfactants, the volumetric flux was smaller at higher temperature even though the emulsion viscosity was much smaller at higher temperature (for polysorbate 20, $\eta_{e,60^\circ\text{C}} = 1.76 \text{ mPa s}$ and $\eta_{e,23^\circ\text{C}} = 3.1 \text{ mPa s}$). However, this aligns well with the membrane resistances measured in water, as these increase when the temperature rises from $1.09 \cdot 10^8 \text{ m}^{-1}$ to $2.23 \cdot 10^8 \text{ m}^{-1}$. In addition, the measured syringe pressure decreased from 300 ± 30 to 210 ± 30 kPa for the polysorbate 20 emulsions and from 400 ± 30 to 310 ± 10 kPa for the polysorbate 80 emulsions, even though the nominal pneumatic pressure

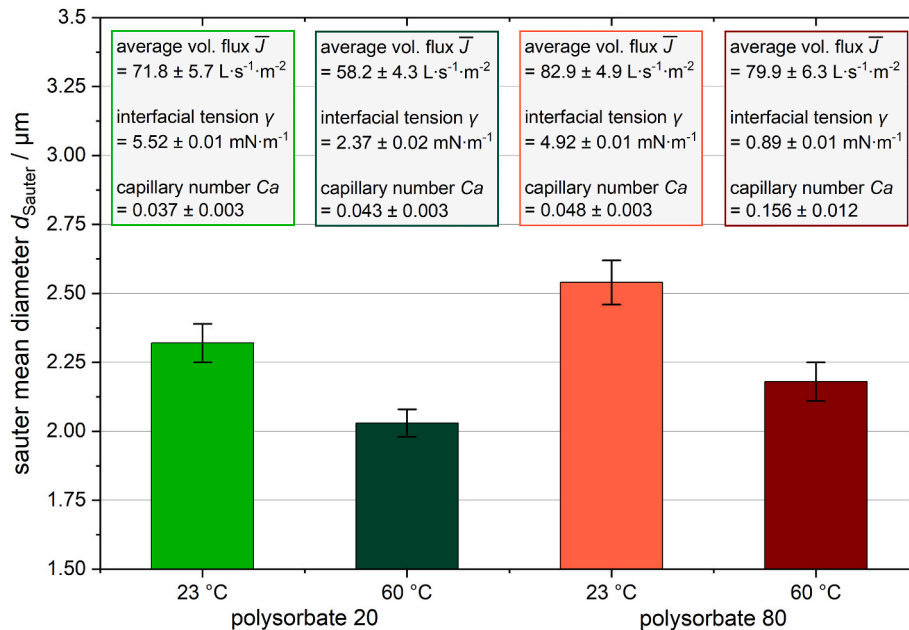


Fig. 6. Sauter mean diameters d_{Sauter} of emulsions produced by in-situ membrane emulsification after 20 passes using polysorbate 20 (green) and polysorbate 80 (red) at 23 °C (light green; light red) and 60 °C (dark green; dark red). The dispersed phase content was 30 vol%. The mean flux was smaller at 60 °C than at 23 °C. This corresponds with the lower measured syringe pressures and a higher membrane resistance at 60 °C. Error bars show the standard deviation calculated from all nine droplet size measurements.

was unchanged for each emulsifier. Although the lower emulsion viscosity at 60 °C would be expected to facilitate flow, this effect was outweighed by the higher membrane resistance and the lower measured syringe pressure. Importantly, smaller droplets were obtained at 60 °C despite the lower transmembrane flux. This indicates that the decrease in interfacial tension outweighed the lower continuous-phase viscosity and pore velocity, resulting in higher capillary numbers and enhanced droplet breakup despite the lower flux.

Joseph and Bunjes (2012) performed premix membrane emulsification experiments using a double syringe membrane extruder at room temperature and 65 °C to prepare nanoemulsions and solid lipid nanoparticles stabilized with different emulsifiers, which resulted in different interfacial tensions. However, the experiments were carried out manually, at relatively high pressures (1000–30,000 kPa) using polycarbonate membranes with a pore size of 50–200 nm which led to small submicron particle sizes of 100–200 nm. The authors could not observe a clear correlation between the processability of different formulations and the particle size distributions with the interfacial tension between oil and water phase. Despite the high interfacial tension of 15.7 mN m⁻¹ between MCT oil and aqueous solution of Poloxamer 188, very small droplets were produced using this surfactant. Polyglyceryl-10-laurate and sucrose laurate surfactants could not make small uniform droplets in all experiments, although they create very low interfacial tensions (3.0–4.7 mN m⁻¹). It can be explained by more pronounced interactions between these surfactants and membrane material. For making small uniform droplets, binding of surfactant molecules to the membrane surface must be minimized to avoid wetting of the membrane surface with the dispersed phase.

3.4. Influence of dispersed phase content on droplet size

Reducing the dispersed phase content of polysorbate 20 emulsions from 30 vol% to 3.5 vol% significantly increased the Sauter mean diameter ($p < 0.05$), see Fig. 7. The pressure in pneumatic piston pumps driving both syringes was set at 400 kPa but the pressure within the syringes was 300 kPa at $\varphi = 30$ vol% and 240 kPa at $\varphi = 3.5$ vol%. This was likely due to fewer oil droplets being forced through the membrane pores and the lower emulsion viscosity at $\varphi = 3.5$ vol%, resulting in lower pressure buildup upstream of the membrane. Thus, at 3.5 vol%, the larger droplets were associated with lower syringe pressure and lower transmembrane flux. Because of the smaller transmembrane pressure, the transmembrane flux was also smaller at $\varphi = 3.5$ vol% leading to the smaller liquid velocity in the pores. Since the interfacial tension remains the same, the capillary number was smaller for the 3.5 % emulsion leading to a larger mean Sauter diameter d_{Sauter} . The increase in droplet size when producing emulsions with a lower dispersed phase content was consistent with a decrease in the lubricating layer thickness h^* from 1.13 to 1.02 μm , suggesting less elongated, more stable droplets. In addition to interfacial instability driven by droplet deformations, droplet disruption is driven by shear stress on the pore walls given by:

$$\sigma_{w,p} = \frac{4\eta_e J \xi_p}{\Phi R_p} \quad \text{Eq. (9)}$$

where ξ_p is the pore tortuosity and Φ is the membrane porosity. Therefore, the wall shear stress in the pores is proportional to the product $\eta_e J$.

In the production of O/W emulsions by repeated premix emulsification using an SPG membrane with a pore size of 10.7 μm under the driving pressure of 20–150 kPa, the mean droplet size was independent of the dispersed phase content over a wide range of φ between 1 vol% and 60 vol% [32]. However, in that case, the transmembrane flux was higher in the experiments with smaller dispersed phase contents compensating for smaller viscosities of these emulsions. Here, both the transmembrane flux and the viscosity of the emulsion were lower in the

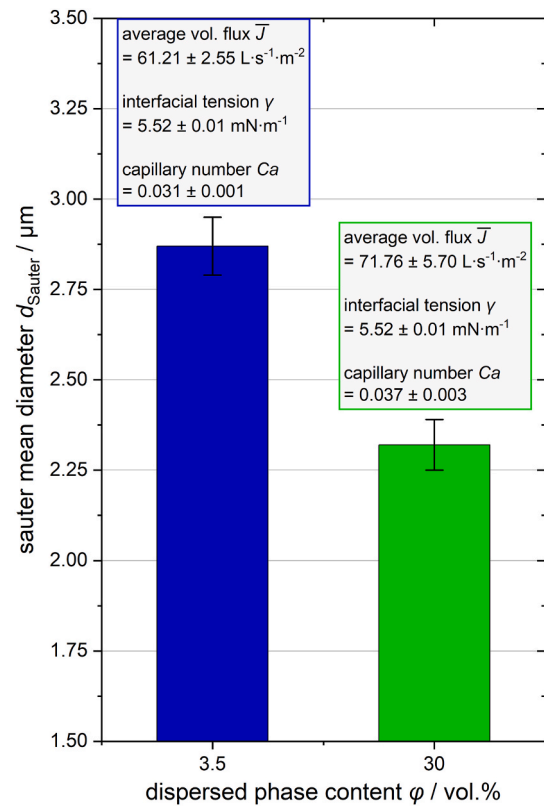


Fig. 7. Sauter mean diameters d_{Sauter} of polysorbate 20 emulsions produced by in-situ membrane emulsification after 20 passes at 23 °C. The dispersed phase content was $\varphi = 30$ vol% (green) and $\varphi = 3.5$ vol% (blue). Lower average flux at $\varphi = 3.5$ vol% was due to lower transmembrane pressure in this case, although the pneumatic system was set at the same nominal pressure of 400 kPa. Error bars show the standard deviation calculated from all nine droplet size measurements.

experiments with $\varphi = 3.5$ vol% leading to higher mean droplet sizes.

4. Conclusions

The Temperature Adjustable Membrane Emulsification Device (TAMED) was developed and evaluated as an automated and temperature-controlled system for laboratory-scale membrane emulsification. The main objective of this study, replacing manual syringe extrusion with a defined automated process, was achieved. TAMED allows the oil and aqueous phases to be added without prior premixing and then processed directly by an automated extrusion sequence. It therefore provides a basis for automated in-situ emulsification under controlled temperature conditions. Similar final droplet size distributions were obtained with and without prior premixing, showing that a separate premixing step was not required under the investigated conditions. Within the investigated range, droplet size was affected by surfactant type, temperature, dispersed phase content, applied pneumatic pressure, interfacial tension, phase viscosity, syringe pressure, and transmembrane flux. Increasing the number of passes reduced the Sauter mean diameter and narrowed the droplet size distribution. After 20 passes, Sauter mean diameters of approximately 2.0–2.9 μm and a narrow droplet size distribution with a span value of approximately 1 were obtained. Both surfactants produced significantly smaller droplets at 60 °C than at 23 °C, despite the lower transmembrane flux at 60 °C. This indicates that the reduction in interfacial tension outweighed the opposing effects of the lower continuous-phase viscosity and transmembrane flux. Under the investigated operating conditions, polysorbate 20 produced smaller droplets than polysorbate 80, while

reducing the dispersed phase content from 30 vol% to 3.5 vol% increased the final droplet size. The results were consistent with the proposed breakup mechanism, in which higher capillary numbers and thicker lubricating films promote droplet elongation and breakup. Direction-dependent behavior was condition-specific. At 23 °C, the transmembrane flux was higher during backward passes, although no clear directional difference in membrane resistance was evident. At 60 °C, the membrane resistance was substantially higher in backward flow.

The current setup is intended for laboratory-scale studies and formulation screening. A key limitation is that constant pneumatic pressure did not produce constant syringe pressure or transmembrane flux, because both depended on emulsion properties, temperature, and membrane resistance. Consequently, the effects of formulation changes and processing conditions could not always be clearly separated. Future versions should therefore directly control either transmembrane pressure or flux. This would allow more precise process control and better comparison between experiments. Further work should assess scale-up and compare TAMED with established processes such as high-pressure homogenization in terms of droplet size control, energy demand, and throughput.

CRediT authorship contribution statement

Philipp R. Schochat: Writing – original draft, Visualization, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Goran T. Vladislavljević:** Writing – review & editing, Supervision, Methodology, Conceptualization. **Ulrike S. van der Schaaf:** Writing – review & editing, Resources. **Nico Leister:** Writing – review & editing, Supervision, Resources, Funding acquisition.

Informed consent

Not applicable.

Ethical review

This study does not involve any human or animal testing.

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Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.jciso.2026.100203>.

Data availability

Data will be made available on request.

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