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Communication

Design of a Microstructured System for Homogenization of Dairy Products with High Fat Content

High pressure homogenization of dairy products is today state of the art but limited by the fat content (max 17 vol.-%). This article describes the development of a novel simultaneous homogenization and mixing (SHM) valve which allows homogenization of dairy products with a fat content of up to 42 vol.-%. The challenging task of homogenizing dairy products with high fat content is to stabilize disrupted fat droplets especially against extensive aggregation. Aggregation and coalescence rates could be significantly reduced by a new microstructured valve allowing the emulsifier-containing phase to be injected directly into the zone of droplet disruption.

Keywords: Food industry, Homogenization, Microprocess engineering, Process intensification

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1 Introduction

High pressure homogenizers are widely used in technical emulsification, especially in the pharmaceutical, cosmetic, chemical, and food industries. Milk, dairy products, fruit juices and concentrates are examples of high pressure homogenized commodities. These products are often homogenized in order to reduce droplet size and thus improve product characteristics such as creaminess or texture and shelf life stability by retarded creaming or sedimentation. Pasteurized milk, for example, has a minimum shelf life of approximately six days. To prevent the formation of a cream layer during this time, the mean diameter of the fat droplets in pasteurized milk has to be well below 1 µm. The volume based averaged fat droplet diameter $x_{3,2}$ in raw milk being in the range of 4 µm is usually reduced to 0.6–0.7 µm [1]. Conventional processing comprises homogenization in one or two stages at pressure levels between 100 and 200 bar and temperatures between 50 and 70 °C [2]. During the homogenization process milk fat droplets are disrupted by shear and inertial forces as the raw milk is pumped through homogenization valves of the flat valve type. Technical innovations of the last 20 years have concentrated on improving the homogenization valve increasing elongation prior to turbulence and cavitation zones [3–5].

To adjust the fat content to the value required for specific product qualities, raw milk is separated prior to homogenization into a low-fat (0.03–0.3 vol.-% fat, 'skim milk') and a fat-enriched phase (30–42 vol.-% fat, 'cream') by a separator. Cream must then be diluted with skim milk to a fat content of 15–17 vol.-% and homogenized [6]. The target fat concentration of, for example, 3.5 vol.-% in full cream milk is achieved by a second mixing step in which the homogenized fat-enriched phase is diluted again with the low-fat skim milk phase. This stepwise process is called partial homogenization (see Fig. 1, left-hand side).

The two-step remixing process interrupted by high pressure homogenization is necessary as, to date, cream with a fat content higher than 17 vol.-% cannot be homogenized with satisfactory results. After breakup, fat globules tend to coalesce as new interfaces are generated, but are not sufficiently stabilized. Coalescence of fat droplets occurs until adsorbing dairy proteins stabilize the droplets. Stabilization kinetics, however, is slow [7]. Coalescence thus can only be prevented at low fat content. [8]. During stabilization of fat globules in cream, a secondary droplet membrane is built up by adsorbing casein micelles and submicelles as well as lacto albumins and lacto globulins [9, 10]. As adsorbed casein micelles strongly interact and form bridges, aggregates of fat droplets are formed. These droplet aggregates can be partially destroyed in a second homogenizing stage [11], as realized in conventional technical processes. However, with increasing fat globule concentration, coalescence and aggregation rates also increase [12, 13]. This is the reason for homogenization pressure being increased by about 20 % during homogenization of cream with a fat con-

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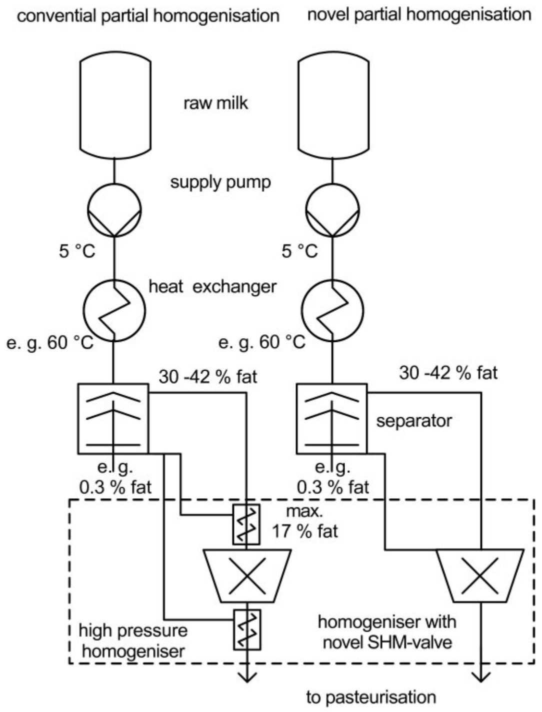

Figure 1. Flow chart of conventional (left-hand side) and novel (right-hand side) partial homogenization process.

tent of 15–17 vol.-%, compared to homogenization of milk with a fat content of 3.5 vol.-%. However, even for increased homogenizing pressures the target product quality cannot be achieved any more for a fat content above 17 vol.-%. Thus, partial homogenization of milk is limited to date.

Partial homogenization of cream with a higher fat content, however, is of major economic interest, being the key to further reduce processing costs in the dairy industry. A novel partial homogenization valve was therefore designed as a one-step processing unit. It allows cream coming from the technical separation process with a fat content of up to 42 vol.-% to be homogenized and mixed directly with skim milk to the final fat concentration in the same processing unit without loss of product quality. This results, on the one hand, in a reduced product volume to be pressurized, thus reducing investment and energy costs, and, on the other hand, in a process line simplified by eliminating two mixing units, reducing investment and cleaning costs.

2 Materials and Methods

Solving the problems of homogenization of cream with a high fat content requires reducing the coalescence and agglomerate build up rates in the moment of fat globule disruption. Droplet stabilizing molecules have to be injected directly into the zone of disruption, and the droplet collision rate has to be reduced significantly in the moment of disruption and stabilization. Technically, this is realized by injecting skim milk directly into the droplet disruption zone of a high-pressure homogenizer valve. For this, a novel microstructured homogenization unit was developed combining a simple homogenizing orifice valve and a micromixer. Allowing homogenization and mixing in a single process unit, this unit is called simultaneous homogenizing and mixing (SHM) valve.

Two main effects can thus be utilized. First, skim milk is rich in whey proteins, they help to stabilize the new fat globule interfaces at the moment when they are created. Secondly, injecting skim milk dilutes the product, thus increasing the distance between fat globules and reducing their collision rate. Both coalescence and aggregate build up rates can be reduced and require a high mixing quality.

However, injecting a high volume of skim milk directly into the disruption zone of fat globules will also change local flow conditions. Transition of laminar flow into turbulent as well as local turbulent kinetic energy dissipation, both being responsible for fat globule disruption, will also be affected.

Therefore, a CFD based simulation of local flow velocities and turbulent kinetic energy dissipation rates was used in order to investigate the effect of the technical design of the micromixing zone in the homogenizing valve on local flow conditions and mixing quality.

The commercial software Fluent® was used. It is based on the finite volume method (FVM) dividing the simulated volume into cells of equal surface size. The turbulence of the fluid was simulated using the RNG-k-ε model. All simulations were steady state and one phase simulations regarding only local flow conditions of the continuous phase causing stresses on fat globule surfaces. Droplet deformation and breakup simulations are not the subject of this investigation.

Fast and intensive mixing is of great importance as it ensures fast transport of surface active molecules to the new surfaces of the fat globules and high dilution rates on the microscale of the droplets. To investigate the influence of the SHM valve design on the mixing quality, the variance of the concentrations of emulsifier molecules was calculated (see Eq. (1)). The lower the variance, the higher the mixing quality. The variance was calculated for all flow lines, set into relation to the expected mixture equilibrium at the end of the mixing zone, and weighted with a speed factor, taking into account the inhomogeneous distribution of the flow rate per cell at the valve exit. The impact of the boundary regions (with lower flow velocities and flow rates) on the mixing quality (variance) is thus reduced.

$$\sigma = \frac{\sqrt{\sum_1^n \left[(x_i - \bar{x})^2 \; \frac{\dot{v}_i}{\bar{\dot{v}}_i} \right]}}{n(n-1)} \tag{1}$$

where,

x_i	concentration of emulsifier molecules in cell i
$\bar{x}$	theoretical concentration of emulsifier molecules at the end of the mixing zone
$\dot{v}_i$	flow rate in cell i related to the surface area of cell i
$\bar{\dot{v}}_i$	average value of the individual flow rates
n	number of cells

First simulation results showed that a T-shaped micromixer element (see Fig. 2) allows you to both maintain high local turbulent energy dissipations rates and to reduce mixing times. The distribution of elongational shear stresses and turbulent

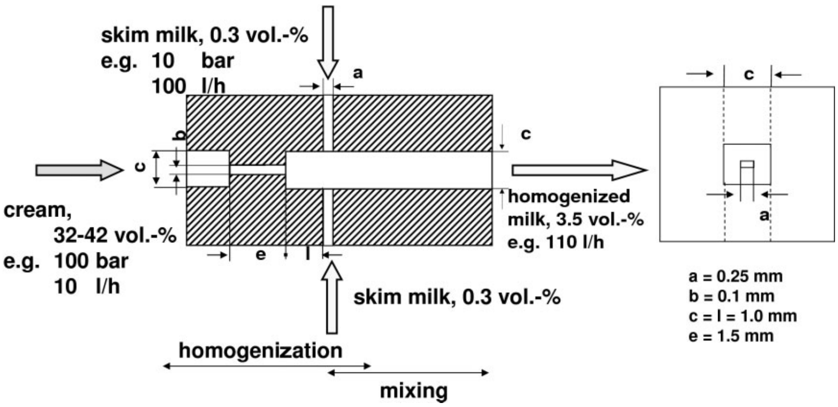

Figure 2. Principle design of the combined homogenization valve and T-shaped micromixer (SHM valve).

kinetic energies as a measure for droplet elongating and disrupting forces, as well as the mixing rate were calculated. The results were used as a basis for the apparatus design. The homogenization orifice in the SHM valve consists of a central channel with the cross-sectional area dimensions of $a = 0.25$ mm and $b = 0.1$ mm and the T-shaped micromixer of two lateral inductors with cross-sectional area dimensions of $a = 0.25$ mm and $c = 1.0$ mm, being located opposite each other. The distance between the outlet of the homogenization valve (central channel) and the outlets of the inline inductors ranged from $l = 0$ mm to $l = 7$ mm.

In the experiments, commercial non-homogenized, pasteurized cream with a fat content of 32 to 42 vol.-% (Alnatura, Germany) and pasteurized skim milk with a maximum fat content of 0.3 vol.-% (Mibell, Edeka, Germany) were used. In comparison, non-homogenized milk with a fat content of 3.5 % was homogenized in one stream passing the homogenization orifice without any side-stream mixing. This full cream milk was produced by mixing the cream with the skim milk to the required fat content. The homogenization temperature was set to 65 °C. Heating and cooling of the cream and the full cream milk (fat content 3.5 %) prior to and after homogenization were realized by micro heat exchangers enabling rapid temperature changes ($\Delta T/\Delta t > 50$ K/s). The residence time in the heat exchangers was less than three seconds. Both fractions were pumped by two parallel working membrane piston pumps (Lewa, Germany). The homogenizing pressure varied according to the volume flow rate which was controlled by a motor control unit changing the stroke length of the pistons. The experimental setup is shown in Fig. 3.

The fat-enriched phase (cream) was pumped through the homogenization valve (central channel) at pressures of up to 300 bar, corresponding to a volume flow rate of up to 14 L/h. Skim milk was injected through the lateral inductors of the SHM valve. A maximum feeding pressure of 30 bar and a maximum volume flow rate of approx. 112 L/h was realized in this test rig. By using a pressure ratio of 10 to 1 between the homogenization pressure of the fat-enriched phase (cream) and the feeding pressure of the skim milk, a mixing volume ratio of 1 to 8 was achieved.

The droplet size distributions of the product were measured using a laser diffraction spectrometer combined with PIDS technology (Beckman Coulter LS 230) ensuring reliable results in the target size range of 100 nm to 10 μm. Volume distributions were used to characterize the fat globule collective. Maximum droplet diameters of the volume distribution $x_{90,3}$ were depicted as a measure for the creaming stability: the higher the maximum droplet diameter, the lower the creaming stability or the shorter the shelf life of the product. To distinguish between aggregation and coalescence of fat globules, the samples were diluted either with water or with SDS solution (sodium dodecyl sulfate, trade mark Texapon K1296, Henkel KGaA, Düsseldorf) prior to particle size measurement. SDS dissolves the casein bridges between the fat globules and thus breaks fat globule aggregates. Differences in the maximum droplet diameters measured with and without SDS are a direct measure for the aggregation rate ξ [14].

$$\xi \equiv \frac{x_{90,3}(\text{measured without SDS})}{x_{90,3}(\text{measured with SDS})} \qquad (2)$$

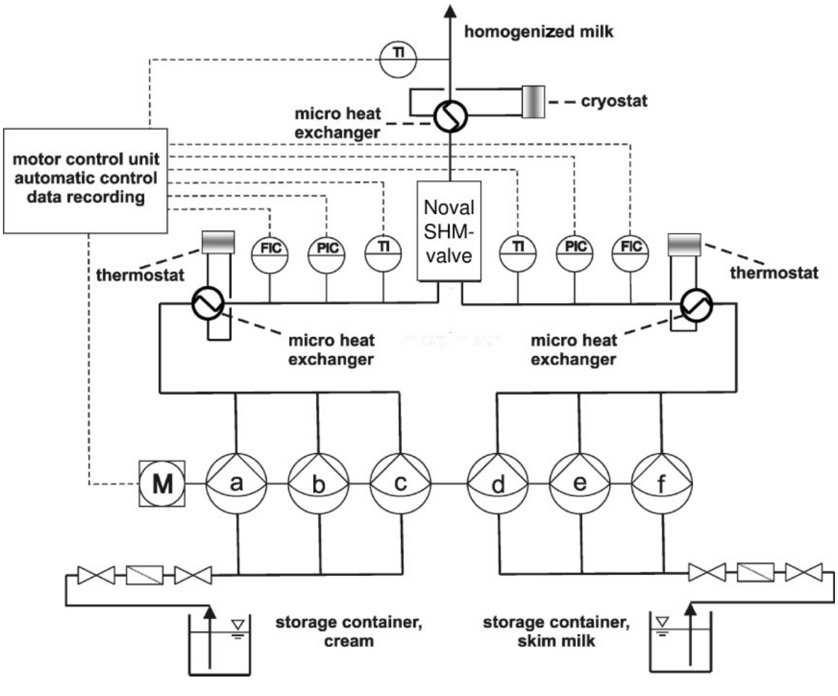

Figure 3. Experimental setup of test rig for partial homogenization using SHM valve.

3 Results

Fig. 4 plots the variance of the emulsifier concentration against the time passed after leaving the homogenization orifice outlet.

In the case of skim milk injection directly into the homogenization orifice outlet (at a distance of *l* = 0 mm), the variance of the emulsifier concentration is reduced very fast, standing for the highest mixing quality and the fastest kinetics compared to higher distances. If the point of injection is at a distance of *l* = 1 mm, the mixing process has a delay of around 20 µs. For *l* = 3 mm, a variance delay is found in the first 60 µs. Regarding the flow lines (not shown in this article) simulated by Fluent, backflow parallel to the stream of the orifice outlet is found to be responsible for this mixing dead time. After passing the mixing point (which corresponds to the position or time of lateral skim milk injection) the variance falls significantly, but remains at an elevated level compared to the other geometries. This again is explainable by the back flow created by lateral injection at this distance. Total concentration levels correspond to equilibrium values with regard to dead zones within the valve.

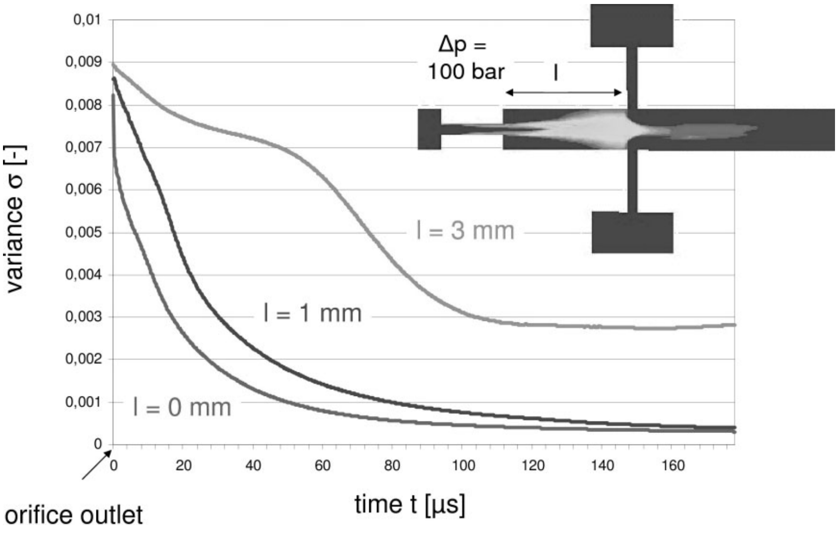

Figure 4. Variance (as indirect measure for mixing quality) as a function of time passed after leaving the homogenization orifice outlet for three SHM valve geometries (distance between lateral conductors and orifice outlet *l* = 0, 1 and 3 mm).

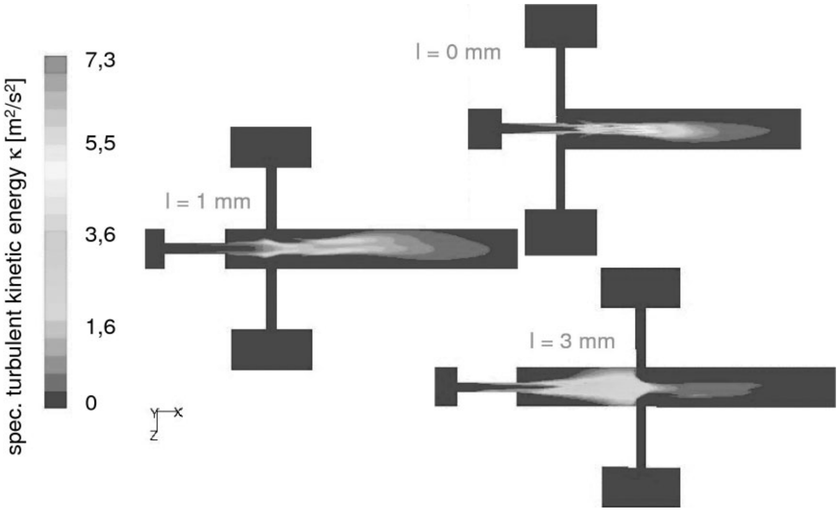

Figure 5. Distribution and intensity of specific turbulent kinetic energy κ in SHM valve. Parameter varied: distance between homogenization orifice outlet and lateral injection (*l* = 0, 1 and 3 mm).

The injection of high volumes of skim milk into the 'main' stream (corresponding to only 10 % of the volume) also affects the distribution and intensity of the specific turbulent kinetic energy κ in the orifice outlet. For a SHM valve geometry with lateral injection at a distance of *l* = 0 mm (directly at the orifice outlet), κ is concentrated in the flow center (see Fig. 5). However, injection at larger distances totally changes energy dissipation localities and intensities. For an injection at a distance of *l* = 1 mm, two areas of turbulent kinetic energy dissipation κ are found, one area in front and a second one behind the mixing inlet point. If the distance is larger (in Fig. 5: *l* = 3 mm), the turbulent energy dissipation area is compressed in the region directly after the valve outlet. Local intensities decrease with increasing distance (not shown).

In conclusion, the impact of the injection point locality on mixing quality and disruptive stresses is counteracting. While a distance of *l* = 0 mm will be optimal if the mixing process (i.e., stabilization) is the dominant factor, larger distances will be better in terms of an undisturbed disruption process. The compression of the flow resulting from the injection of high volumes can even be positive in terms of creating an internal counterpressure.

To prove the concept, a SHM valve with a mixing point at 3 mm behind the orifice outlet was built and the results compared with those achieved with the simple orifice valve (without lateral injection) used for full stream homogenization.

Fig. 6 shows the homogenization results for full stream homogenized milk (3.5 vol.-% fat) and cream (32 vol.-% fat) as well as the results of partial homogenization of cream (32 vol.-% fat) with skim milk (0.3 vol.-% fat) injection using the novel SHM valve. In full stream homogenization of milk (3.5 vol.-% fat) droplet diameters less than 4.0 µm can be achieved. With higher fat content (here 32 vol.-% fat) droplet diameters up to 40 µm are found due to coalescence and mainly aggregation of the disrupted droplets. The higher the homogenization pressure, the higher the coalescence and aggregation rates. Using the SHM valve to feed skim milk directly into the disruption and stabi-

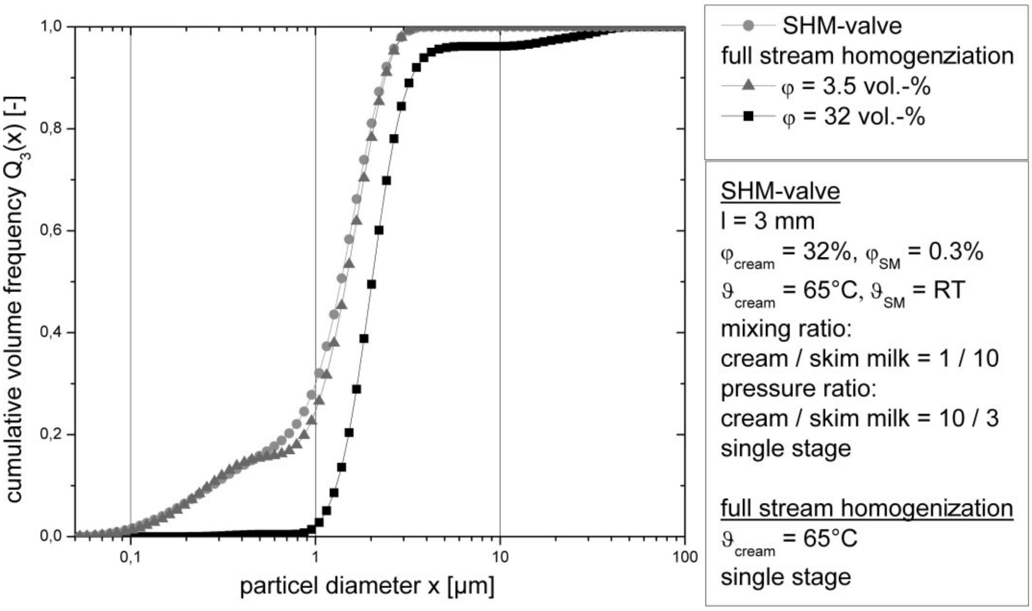

Figure 6. Homogenization results (cumulative volume distribution Q_3) for full stream homogenization of milk (volume fat content φ = 3.5 vol.-%) and cream (φ = 32 vol.-%) and for partial homogenization of cream (32 vol.-% fat) with skim milk (φ= 0.3 vol.-%) injection in novel SHM-valve on a homogenization pressure of Δp = 100 bar.

lization zone allows an effective disruption and stabilization of the droplets. Thus, homogenization results comparable to full stream homogenization of full cream milk can be achieved even for cream with 32 vol.-% fat.

As shown in Fig. 5, too large distances, in turn, will worsen the mixing quality and thus allow fat globule coalescence and aggregation. The positive effect of locally increased disruptive stresses by compressed flow areas will also be lost. An optimal distance in the region between 3 and 10 mm was predicted by CFD simulations. This optimum was found through experiments (see Fig. 7).

For this, SHM valves with injection points at distances of 0, 1, 3, 5 and 7 mm were constructed. Cream with 32 vol.-% milk fat was homogenized at 100 bar as 'main' stream, and skim milk with 0.3 vol.-% was injected laterally at different distances. For this product (full cream milk), best results were found for injection at a distance of 5 mm. Smaller distances resulted in a decreased fat globule disruption and higher distances in an increased globule aggregation and coalescence. Generally, the agglomeration rate ξ remains nearly constant over a distance of l = 0–7 mm by ξ = 1.

This means that the disruption process in the homogenizing orifice outlet is the dominant process. Stabilization by mixing surface active material and dilution is of minor influence as long as mixing within around 100 µs is ensured.

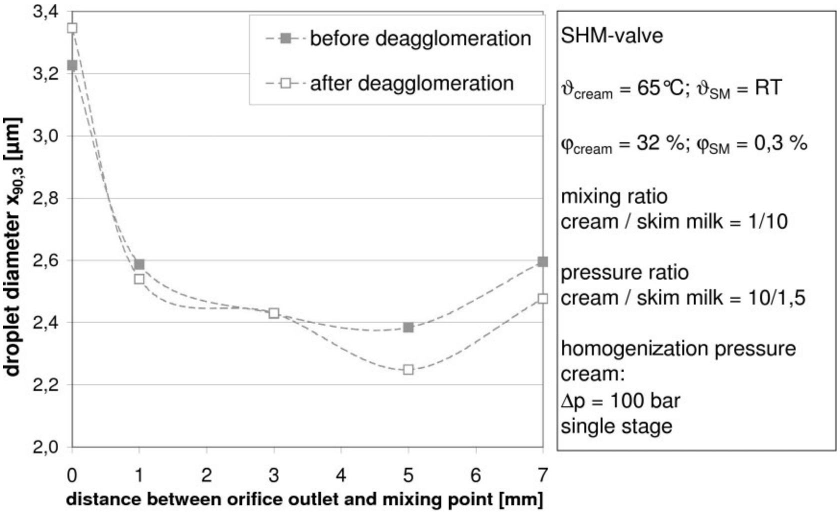

Figure 7. Homogenization results (maximum droplet diameter $x_{90,3}$ of volume distribution) for partial homogenization in novel SHM valve by changing the distance between valve outlet and mixing point.

4 Summary

High pressure homogenizers are widely used in technical emulsification, especially in the pharmaceutical, cosmetic, chemical, and food industries. Milk and cream, dairy products, fruit juices and concentrates are examples of high pressure homogenized commodities. Homogenization improves product quality and reduces creaming, thus improving shelf life stability. In industrial applications, cream with a milk fat content of up to 15–17 vol.-% is homogenized by conventional high pressure devices. The homogenized cream is then diluted with skim milk in a separate process step to adjust the fat concentration to the desired level (partial homogenization). Partial homogenization results in a decrease in the volume to be homogenized at high pressure and thus in a reduction in energy savings. However, to date, partial homogenization of cream is limited to a fat content of 15–17 vol.-%. Even when two-stage homogenization is applied, a build-up of milk fat aggregates and coalescence of fat droplets leads to an increase in fat globule diameter and consequently to a loss in product quality. The higher the fat concentration, the larger the fat globules and aggregates in the product, giving rise to reduced shelf life.

A novel microstructured system combining a simple homogenizing orifice with a T-shaped micromixer (SHM valve) was developed. This system allows skim milk to be injected directly into the zone of fat globule breakup, leading to an improved stabilization of the newly created droplets and their dilution. Thus, the fat content in the partial homogenization of cream can be increased to at least 32 vol.-% with no loss of product quality. Based on CFD simulations and experiments, an optimal distance between the outlet of the homogenization orifice and the injection point for the skim milk is found to be about 5 mm.

For milk homogenization, the novel SHM valve reduces the product volume to be homogenized at high pressure and the number of processing units in dairy homogenization processes. In comparison with a one-stage conventional homogenization, the new system combining homogenization and micromixing requires only 20 % of energy input compared to full stream, and only 60 % of energy input compared to conventional partial homogenization of milk. This results in considerable energy and cost savings in milk processing without any loss of product quality. In addition, two mixing units can be eliminated from the process line, resulting in less investment, cleaning and maintenance costs.

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