

Optimisation of Orifice-Type High Pressure Emulsification Valves

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ABSTRACT

High pressure emulsification is a common unit operation in the technical production of emulsions in the sub-micron range. It is widely spread in food industry, particularly for the production of dairy products, fruit juices and concentrates. The droplet disruption efficiency is significantly influenced by the flow conditions which in turn depend on the geometry of the homogenising valve. In the 1990's, the Food Process Engineering division of University of Karlsruhe developed a simple but very efficient high pressure homogenising valve which was improved during the past years.

The research was focused on the one hand on the understanding of the mechanisms of droplet disruption and stabilisation in homogenisation valves. On the other hand the scale-up rules were developed. The understanding of droplet disruption and stabilisation mechanisms was improved by the use of computational fluid dynamics (Fluent[®]). Cavitation areas as well as turbulent kinetic energies were calculated, different flow regimes were localised. In parallel, an experimental method was developed that allowed to investigate droplet disruption and coalescence, independently from each other, within the homogenisation zone. Based on this understanding, the valve geometry can be optimised regarding the specific properties of the emulsion phases. Products can thus be designed to target key parameters of the particle size distribution as e.g. small mean or maximum droplet diameters. Further on, by varying flow regimes and current combinations, the stabilisation of the droplets can be enhanced and energy input can be decreased significantly.

Introduction

High pressure emulsification is a common unit operation in the technical production of emulsions in the sub-micron range. In food industry it is widely spread, in particular for the processing of dairy products, fruit juices and concentrates. The droplet disruption efficiency is significantly influenced by the flow conditions which in turn depend on the geometry of the homogenising valve. In the 1990's, the Food Process Engineering division of Karlsruhe University developed a geometrical simple but very efficient high pressure homogenising orifice valve which has been improved during the past years [Freudig 2004, Stang 1998, Tesch 2002].

In most homogenising devices droplets are not only deformed and disrupted by one single mechanism. Usually, different mechanisms affect the droplets simultaneously. In a simple orifice valve droplets are deformed in a laminar elongational flow partially superimposed by shear flow in front of the borehole, whereas behind the borehole forces in turbulent flow predominate. Furthermore, cavitation phenomena may occur.

The research within the last years was focused on both: Optimise the disruption and the stabilisation of the droplets as well as the endurance of a valve. For this, the elongation on the inlet of the valve, cavitation around the valve and the turbulence intensity behind

the valve was targeted modified. This research resulted in the development of the impingement jet valve, several deflection valves and special micro-structured systems described herein.

Droplet disruption in the valve technology

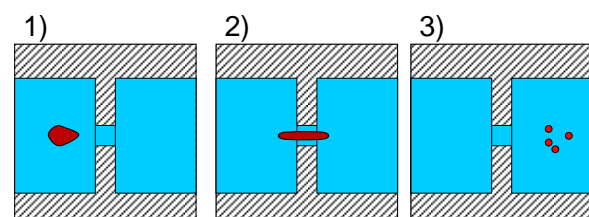


Figure 1: Disruption process using the valve technology – principle scheme: 1) Elongation in front of the valve 2) Deformed droplet inside the valve 3) Disruption due to disturbances in the flow behind the valve e. g. due to turbulence.

A laminar flow is found in front of the valve in which the flow lines are compressed towards the valve inlet (see Figure 1). In the inlet flow droplets are therefore elongated to filaments. The deformation of the droplets depends on the elongation rates, on the residence time in the elongational field and on the viscosity ratio λ between the dispersed and the continuous phases. The droplet's disruption in an elongational flow is for

long exposure times almost independent of the viscosity ratio [Bentley et al. 1986, Grace 1982, Kaufmann 2002]. However, due to the short residence time of a few milliseconds in high pressure homogeniser valves, the droplets are elongated in the inlet flow, but almost no droplets are disrupted [Budde et al. 2002, Kolb 2001, Tesch et al. 2001]. For droplet disruption in laminar flows the maximal droplet diameter depends on the energy density according to Schuchmann [Schuchmann 2005, Stang et al. 2001].

$$d_{\max, \text{lam}} \propto E_v^{-b} \quad b = 1 \quad (1)$$

While the droplets pass through the valve, external forces stabilising the filaments do not exist. When leaving the zones of elongational shear, droplets start to relax to the energetically optimised spherical shape. The lower the viscosity ratio and the higher the surface tension is, the faster the deformed droplets relax to spheres. The more elongated droplets are entering flow areas with high turbulence intensity, the easier they are disrupted and the more efficient droplet disruption is.

Behind the valve outlet the flow regime in the free jet becomes turbulent. Behind this transition point the turbulence intensity as well as the local power density rise, disrupting droplets. In turbulent flow, inertial forces are mainly responsible for droplet disruption. According to Kolmogoroff's theory for isotropic turbulence, eddies of different dimensions exist in a turbulent flow [Kolmogoroff 1958]. In case of eddies with a similar size as the droplets, the droplets can be broken up by pressure oscillations due to the variation of velocities of eddies. In this case the maximal droplet diameter depends on the power density after Walstra [Walstra 1983].

$$d_{\max, \text{turb}} \propto P_v^{-b} \propto E_v^{-b_1} \cdot z^{2/5} \quad (2)$$

Here b and b_1 are constants that may be empirically determined to $b = 0.4$ and $b_1 = 0.6$ for $\eta_d < 10$ mPa·s [Arai et al. 1977]. The parameter z is the characteristic length in which disruption occurs. In case of the superposition of both disruption mechanisms (1) and (2), intermediate values of the exponent are possible. The efficiency of droplet disruption also depends not only on the elongation rate and on the turbulence intensity but also on the residence time between deformation (valve inlet) and the rise of flow oscillations (e. g. maximum of the turbulence behind the valve outlet).

By changing the valve geometry, higher elongation rates, higher power densities and shorter transition times between flow patterns can be achieved. This resulted in the development of the impingement yet valves as well as several deflection valves.

If two free jets, leaving two oblique boreholes, collide, the flow will become turbulent immediately. In this type of valve (see Figure 2A), the position of the transition point depends on the pitch α . An increasing pitch and thus decreasing distance between the transition point and the valve results into smaller droplets at comparable homogenising pressure [Aguilar et al.

2004]. Obviously, forces in a turbulent flow play an important role in droplet disruption. Modifying the distribution of turbulence is an efficient tool to change emulsification efficiency.

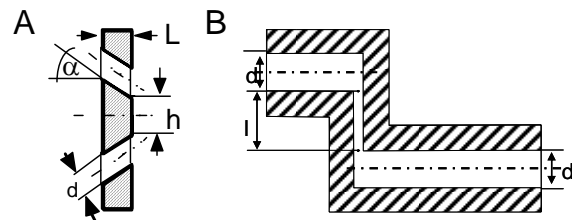


Figure 2: A: impingement jet valve B: Example of a deflection valve [Aguilar et al. 2006]

In Figure 2B one example of a deflection valve is depicted. Because of the enforced deflection of the flow within the valve, high values of the power density can be obtained. This is important for systems with very short relaxation times of the droplets (high interfacial tension, small viscosity ratio λ).

In both valve types, increased disruption efficiency is counterdicted by a stagnation point. This leads to an increased static pressure and thus decreased flow rate. This local back-pressure has an influence on the cavitation behaviour, as well, and therefore on the homogenisation results, also. Applying a small back-pressure behind a homogenising valve can also improve the result of homogenisation. Back-pressure displaces the position and increases the intensity of the compression wave in the outlet flow so that the droplets are disrupted more efficiently [Treiber 1979]. However, up to now cavitation cannot be fully described and simulated in a satisfying way. Hence, the influence of the cavitation on the disruption process is still not sufficiently accessible.

Mechanisms of droplet stabilisation in a valve

Emulsions as considered in this article are not stable without addition of emulsifier molecules.

In high pressure homogenisation, droplet elongation is huge and residence time is too short for emulsifier molecules to adsorb at the new interfaces. As shown in recent work, the emulsifier has therefore no major influence on the disruption process itself [Kempa et al. 2006]. Droplet deformation and disruption occur in this type of machines in characteristic times of few milliseconds. The characteristic time for the diffusion of the emulsifier molecules from bulk face through the boundary layer and the adsorption on the surface is by far larger than the disruption time in these type of valves. Hence, disruption results are independent of the emulsifier type, providing the fine droplets are stabilised fast enough after their generation [Kempa et al. 2006, Walstra 1983].

After the disruption of the droplets the newly created interfaces, however, have to be stabilised to prevent aggregation and coalescence. The conventional way to

stabilise the fine droplets is to use an emulsifier. In most cases it is possible to mix the emulsifier in the premix hence the emulsifier molecules only have to diffuse through the boundary layer and reorient to adsorb at the interfaces. That is the case of low molecular emulsifiers in turbulent flow. For high molecular emulsifiers, like proteins, diffusion adsorption and interfacial molecular conformation change times can be much higher than the residence time of the droplets in the turbulent flow. Coalescence and aggregation rates thus are significantly increased.

Coalescence and aggregation rates, however, may be minimised by hydrodynamic effects. Using special geometries, a stabilisation zone can be created. In such a stabilisation zone, the collision time between two droplets is less than the minimum time required for coalescence (e. g. the drainage time). As a result, the droplet size remains constant. Another possibility to reduce coalescence and/or aggregation is to dilute the homogenisation stream directly after the valve.

The last case is especially interesting if the emulsifier can not be added in the premix. This idea resulted in the development of a new combined homogenisation-micro-mixing valve. In Figure 3 this new combi-valve type is shown. It allows mixing a second component current directly into the homogenisation main stream. By injecting a second component current it is possible to realize both, dilute the main stream and thus to reduce the collision frequency, and to feed emulsifier in the homogenisation zone where it is required. However feeding into the disruption zone changes local flow conditions and thus has consequences on the disruption process itself. The distribution of turbulent intensity κ varies due to the formation of a new stagnation point. The turbulent kinetic energy was simulated by computational fluid dynamics (CFD) and is a measure for the intensity of turbulence. For a homogenisation pressure of 100 bar mixing times of a few milliseconds are realized using a T-mixer directly behind the homogenisation valve outlet. Based on these CFD simulation results, the distance between homogenisation valve itself and the T-micro-mixer was optimised for both, minimised negative impact on disruption and shortest mixing times.

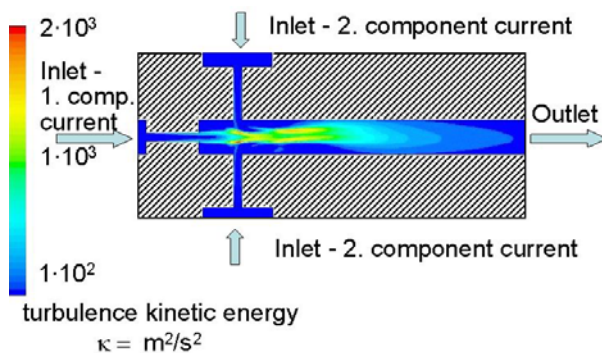


Figure 3: Turbulence kinetic energy distribution in the novel micro-structured combi-valve [Aguilar et al. 2006].

In the dairy industry, for example, milk is commonly homogenised by partial homogenisation [Kessler 1996]. In this process the milk is separated in cream (42 vol.-% fat) and skim milk (0.3 vol.-% fat). Afterwards the cream is re-mixed with skim milk to a fat content of 17 vol.-%, homogenised by 100 to 250 bar and finally mixed to the final consumer product concentration of e. g. 3.5 vol.-%. Thus, energy input is significantly decreased as less volume has to be homogenised at high pressure. However, up to now, no solution has been found to homogenise milk with fat contents higher than 17 vol.-%. Above this concentration coalescence and agglomeration rates are thus increased that droplet diameters significantly increase resulting in undesired product characteristics especially lowered shelf life.

Using the novel homogenisation combi-valve shown in Figure 3, it is possible to homogenise cream up to 42 vol.-% by mixing skim milk via the second component current [Aguilar et al. 2006].

Scale up of the new homogenising valves

To scale up of the new homogenising valves the Food Process Engineering division of the University of Karlsruhe investigated three different approaches during the past years (see Figure 4).

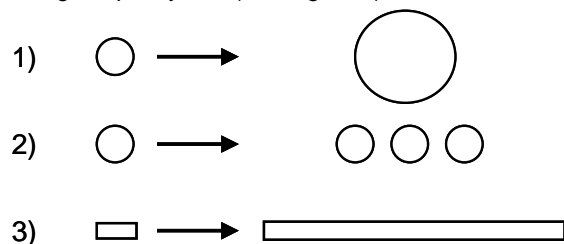


Figure 4: Three different possibilities to scale up a homogenising valve [Aguilar et al. 06].

In the first case, it is possible to scale up the valve by enlarging its diameter. This option is strongly limited as above a critical diameter (often $0.8 \mu\text{m}$) the elongational rates are too low in order to sufficiently deform the droplets of the premix emulsion and therefore the homogenisation results worsen.

The second case, the so-called numbering up, is a common way in technical scale up. The critical parameter in numbering up is the distance between the different valves. Below a critical distance interactions between the free streams lead to a deterioration of the flow streams behind the valve and thus worsen homogenisation results. Experiments have shown that for low viscous emulsions ($<10 \text{ mPa s}$) the critical distance is about six times bigger than the valve diameter [Aguilar et al. 06].

In the last case, the width of the valve is enlarged by building a slit. Experiments show that for sufficient large slits the homogenisation results depend mainly on the shortest dimension of the cross section area [Schultz 2003]. A scale-up is thus possible. One dimension of the cross section area, e. g. the width,

may be increased, provided that the shortest dimension, i. e. the height, is held constant.

Life time of a homogenising valve

The endurance of the valve depends mainly on the intensity and location of cavitation always found. There are different areas where cavitation may appear, e. g., in locations of high velocity gradients perpendicular to the flow lines. This situation is given at the inlet and outlet of the valve. In both areas a shaped edge exists. Using small geometrical modifications, as rounding inlet and outlet edges of the valve channel, cavitation regions are minimised resulting in a significant improvement of the valve life-time.

Conclusions

In most homogenising valves droplets are not only deformed and disrupted by a single mechanism. Generally, several forces act on the droplets simultaneously. By changing valve geometry, a specific mechanism can be reinforced. The inertial forces in turbulent or cavitation flows are the most important mechanisms of droplet break-up in high-pressure homogenisation of low viscosity emulsions, provided that a critical value of the elongation rate is realized. The higher the viscosity of the emulsion the more important the droplet deformation in the elongational flow becomes. For emulsions with a high viscosity ratio λ , the standard valve is advantageous. For emulsions with a lower viscosity ratio λ , the angular impingement yet valves and the deflection valve provide better results.

During the past years the Food Process Engineering division of the University of Karlsruhe developed and optimised the orifice valve technology regarding the characteristics of the material systems. Valves adapted to product characteristics are then produced by the Institute for Micro-Process Engineering. Today, with the valve technology it is possible to homogenise emulsions down to droplet sizes below 100 nm depending on the surface tension, the viscosity ratio, the viscosity of the emulsion. However, droplet stabilization after disruption is crucial to stabilize this result.

For large volumes the partial homogenisation has enormous economic advantages as the homogenised volume is significantly reduced. Using a novel combi-valve combining homogenisation valve with a micro-structured mixing system, it is possible to reach the same particle sizes by partial homogenisation as for full stream homogenisation even in slow stabilizing emulsifiers have to used. This is of special interest for food industry as e.g. in milk homogenisation.

Acknowledgement

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