Chemical Engineering Science 131 (2015) 162–171

Contents lists available at ScienceDirect

Chemical Engineering Science

journal homepage: www.elsevier.com/locate/ces

High-pressure double stage homogenization processes: Influences of plant setup on oil droplet size

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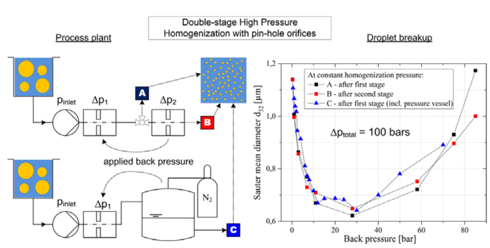
HIGHLIGHTS

- Influence of back pressure on high pressure emulsion formation is investigated.
- Second stage delivers back pressure changes cavitation pattern.
- In double stage processes droplets are broken up entirely in the first stage.
- Prediction of droplet sizes is only possible for constant back pressures.

GRAPHICAL ABSTRACT



ARTICLE INFO

Article history:
Received 17 December 2014
Received in revised form
17 March 2015
Accepted 31 March 2015
Available online 13 April 2015

Keywords:
High-pressure homogenization
Double stage
Hydrodynamic cavitation
Counter pressure
Oil drop size

ABSTRACT

High pressure homogenization is widely used to produce small droplets in liquid products. The use of two homogenization units (e.g. orifices) in series thereby is generally accepted as increasing homogenization efficiency, even when the mechanism is still not fully understood yet. It is shown that for a single stage orifice operated at a given overhead pressure, the drop size decreases with increasing backpressure first until a minimum is reached and then increases due to the lower total pressure drop due to the lower flow velocity and driving force. Simultaneously observation showed that backpressure suppresses cavitation. This paper focuses on the question whether the droplets break up at the first or second orifice. A process plant is designed enabling us to investigate droplet breakup in *o/w*-emulsions at each orifice separately. In subsequent experiments the second orifice was substituted by a pressure vessel to eliminate the influence of the second orifice entirely. Our research reveals that droplets are broken up only in the first orifice. The second orifice delivers back-pressure being responsible for a change in the cavitation pattern found after the first orifice. The smallest oil droplet sizes are reached at a back-pressure of ~30% of the inlet pressure, a value being in consensus to former experimental studies. A discussion of the droplet breakup with regard to the mean specific energy input E_v at the first orifice is given. It depicts that a change in flow pattern, especially the appearance of vapor after the first orifice has a huge impact on energy transfer efficiency. A prediction of resulting droplet sizes as result of pressure drop in the first stage is only possible for constant *Th* numbers.

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1. Introduction

High-pressure homogenization is widely used in continuous processing of emulsions to obtain highly valuable products, such as for foods, pharmaceuticals, fuels, and paintings. In general, the objective of this energy intensive process is the formation of an emulsion with a specific droplet size distribution in the micron to sub-micron scale. Pressures up to several 1000 bar are used to force the liquid through homogenization units with small cross-section areas, such as valves or orifices. As a result of the contraction the fluid velocity severely increases and induces elongation and shear stresses in laminar and turbulent flow conditions, which may

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http://dx.doi.org/10.1016/j.ces.2015.03.055
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disrupt the dispersed droplets. Additionally, hydrodynamic cavitation can occur in and after the constriction due to the increase in local fluid velocity and thereby a decrease of local pressure reaching the vapor pressure of the fluid (Arndt and George, 1979; Franc and Michel, 2004). The decrease in droplet size can be correlated to the applied energy density (Karbstein, 1994), with decreasing values for increasing mean specific energy input, multiple homogenization passes (Phipps, 1985) or the use of a faster absorbing emulsifier (Donsì et al., 2012). With regard to an energy efficient production, a so called double stage process including two high pressure units in series is preferentially installed in industrial homogenizers. The second high-pressure unit applies a 'back-pressure' p_{bp} towards the first one, while the inlet pressure p_{inlet} remains the same, compared to a 'single stage' process. As a result of increasing p_{bp} the visual appearance of cavitation decreases gradually. McKillop et al. (1955) were able to show for a high pressurized flow though a venturi nozzle with applied back-pressure first a decrease of visible cavitation occurrence until cavitation was totally suppressed at high back-pressures. Consequently, the vapor content in and downstream of the constriction decreases as well. Actual measurements in a rectangular micro-channel with a modified μ-PIV method show impressively how sensitive cavitation induced vapor content responds on applied p_{bp} (Gothsch et al., 2014). Without an applied p_{bp} cavitation can form coherent vapor pockets filling the complete cross-sectional area of the outlet channel, before forming a diffuse collapse area (Yan and Thorpe, 1990; Rooze et al., 2012). By applying and increasing the p_{bp} at constant p_{inlet} the coherent vapor pockets disappear and the cavitation switches to single bubble cavitation, forming a diffuse collapse area (Håkansson et al., 2010; Rooze et al., 2012).

Studies on the effect of a back-pressure inducing second orifice on emulsification efficiency concentrate on its effect on the droplet sizes obtained after the second stage. At constant inlet pressure p_{inlet} an increase in emulsification efficiency is obtained with increasing p_{bp}. This is represented by a reduction of droplet sizes compared to a single stage process at the same p_{inlet}. A minimum droplet size is obtained at back-pressure to inlet pressure ratios of 20–30% (Mohr, 1987; Freudig, 2004; Finke et al., 2014). Several researchers explained the increased efficiency in droplet breakup with an intensification of droplet break-up effective forces (McKillop et al., 1955; Kurzhals, 1977; Treiber, 1979; Karbstein, 1994; Freudig et al., 2003; Finke et al., 2014). Kolb et al. (2001) hypothesize that re-coalescence incidences of recently formed droplets are reduced. Secondary droplet breakup or breakup of coagulates of small droplets formed after the first stage is discussed as possible mechanisms found in the second stage (Köhler and Schuchmann, 2012). The latter is thought to be the main reason for double stage homogenization in dairy processes where casein bridging of small milk fat globules is often found after the first stage (Kessler, 2006; Köhler et al., 2008). Freudig et al. (2003) argue that cavitation intensity and thereby power density yield is increased. Besides inertia and viscous forces in turbulent flows, they argue that hydrodynamic cavitation supports droplet break-up in high pressure homogenizers. Shock waves and micro-jets induced by collapsing cavities are regarded as droplet disruptive effects in cavitating flows. Thereby the location of the cavitation collapse as well as the intensity of induced shock waves and micro-jets (cavitation intensity) are crucial (Kurzhals, 1977). As Innings et al. (2011) show from tests with cavitation-erosion it is known, that the collapse of the cavitation shifts further upstream with increasing back-pressure. Based on theoretical local pressure calculations, Freudig (2004) presumes that droplet breakup in double stage systems most likely occurs after the first orifice. An experimental proof of this assumption has not been given so far. As a deeper understanding of droplet breakup in double stage processes will help in an improved process design of homogenization plants, our study intends to investigate experimentally the effect of double stage processing and back-pressure on droplet breakup in the first homogenization step.

We thus want to consolidate new findings on the effect of back-pressure on hydrodynamic cavitation found after constrictions in venturi nozzles or micro-channels to the findings reported for the effect on the second stage in high pressure homogenization. This will improve our understanding and answer the open question if a second stage only affects droplet breakup in the first one (as proposed by Freudig, 2004). For this study we introduce an experimental setup by which an emulsion sampling is set between two high pressure orifices, without distracting the process of emulsion formation. This offers the possibility to investigate the influence of the first and second orifice on oil droplet breakup in double stage processes separately. For a deeper understanding of the multiphase flow in the orifices the emulsification results are accompanied by shadow graphic images of the cavitation patterns in the outlet channel after each orifice. A further variation of the process plant was performed to eliminate the influence of the second constriction entirely. Therefore the second orifice is substituted by a high volume pressure vessel installed at the end of the outlet channel to apply back-pressure. Emulsification results and flow calculations of this setup were compared to those achieved in the original two-stage setup. We finally discuss the results with regard to the mean specific energy E_v introduced by Karbstein (1994).

2. Theoretical considerations of characteristic parameters

To compare flow characteristics and emulsification efficiency for altering geometry or process parameters, several established approaches are used in this study. Since the mass flow rate $\dot{M}$ can be accurately measured, the mean velocity $\overline{u}_{orifice}$ inside the cylindrical coaxial orifice with the diameter $d_{orifice}$ and the resulting Reynolds number Re can be calculated. Based on the continuity equation for a flow line through an orifice, the mean velocity $\overline{u}_{orifice}$ is calculated as

$$\overline{u}_{orifice} = \frac{\dot{M}}{\pi \cdot \left(\frac{d_{orifice}}{2}\right)^2 \cdot \rho_l}. \quad (1)$$

Here ρ_l represents the density of the liquid at experimental conditions. Losses by friction are neglected in the present case. With $\overline{u}_{\text{orifice}}$ and known dynamic viscosity of the liquid η_l, the Reynolds number for the orifice can be calculated as

$$Re = \frac{\rho_l \cdot d_{orifice} \cdot \overline{u}_{orifice}}{\eta_l}. \quad (2)$$

To consider changes in friction loss due the constriction's geometry as well as detachment of flow and change in flow conditions, the coefficient of discharge C_D is used. It characterizes the relationship between the ideal pressure loss Δp_{ideal} and the actual measured pressure loss Δp. It can be described as (Ghassemieh et al., 2006)

$$C_D = \frac{\sqrt{\Delta p_{ideal}}}{\sqrt{\Delta p}} = \frac{\dot{M}}{A_{orifice} \cdot \sqrt{2 \cdot \rho_l \cdot \Delta p}} = \frac{\rho_l \cdot \overline{u}_{orifice}}{\sqrt{2 \cdot \rho_l \cdot \Delta p}}. \quad (3)$$

Plotted against $\sqrt{Re}$, the C_D value can be used to estimate a change in flow conditions from laminar to turbulent flow (Johansen, 1930). The plot against $\sqrt{Re}$ is preferred to provide as open scale as possible especially at low Re. Kelemen et al. (2014) found developed turbulent flow at $C_D \approx 0.75$ and $\sqrt{Re} > 60$ for high pressure homogenizers equipped with a single orifice similar to the ones used in this setup. Considering the effect of cavitation, Ramamurthi and Nandakumar (1999) determined decreasing C_D

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