



Influence of viscosity ratio and initial oil drop size on the oil drop breakup during effervescent atomization



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ABSTRACT

The present study focused on the effervescent atomization of oil-in-water emulsions to evaluate the influence of viscosity ratio and initial oil drop size on the oil drop size after atomization. Constant atomization conditions were assured by spray drop size measurements. The viscosity ratio of the emulsion was varied between 0.24 and 27 by changing the oil viscosity. The initial oil drop size was adjusted to values from 0.67 to 21 μm . The disperse phase content was set to 1 wt-% to exclude coalescence phenomenon of the oil drops. The low disperse phase content was also chosen to ensure comparable process conditions especially the stresses acting on the oil drops in atomization. Oil drop breakup was visible for atomized emulsions with initial oil drop size above 2 μm , while oil drops in the emulsion with an initial oil Sauter mean diameter of 0.67 μm remained stable in effervescent atomization. In terms of viscosity ratio, oil drop breakup was found up to a viscosity ratio of 13.5. Comparing oil drop breakup results to those of capillary rheology measurements with defined shear rates we conclude that the shear rates acting on the oil drops in the effervescent atomizer were higher than $1.000.000 \text{ s}^{-1}$.

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1. Introduction

A challenging task in combustion, agriculture, pharmaceutical and food industry is the atomization and spray drying of emulsions. For this reason, numerous studies on emulsion stability in atomization are found in literature. Research motivated from agricultural and combustion applications shows, that the presence of a disperse phase inside the atomized liquid influences the spray drop size [1–17]. In these studies, the change of the disperse phase drop size was not considered. Atomizers used were mainly pressure or pneumatic atomizers, besides some specialized geometries.

In the pharmaceutical and the food industry, emulsions are atomized and dried most often for microencapsulation of volatile ingredients [18–37]. As typical for these applications most of the investigated emulsions were oil-in-water emulsions. So far, there are only few studies which have looked at the change of the disperse phase drop size during the atomization step e.g. by

measuring the oil drop size after reconstitution of the spray-dried powders. Some studies compared the oil drop size prior to atomization and after reconstitution [38–42]. Others did not include the oil drop size after reconstitution or neglected to mention the oil drop size prior to atomization [18,20,24,28–30,36,37].

For oil drop breakup in general, several influencing parameters can be found in the emulsification literature. A parameter of high relevance for drop deformation and breakup is the viscosity ratio, which is defined as the ratio of the viscosity of the disperse phase η_d to the viscosity of the continuous phase η_c [43]. If the disperse phase content is high, the viscosity of the continuous phase has to be substituted by the viscosity of the emulsion η_e (see Eq. (1)) [44,45]. In this study, the viscosity of the emulsion was also used for calculating the viscosity ratio, since a very low disperse phase content will not influence the viscosity measurement [46].

$$\lambda = \frac{\eta_d}{\eta_c} \text{ respectively } \lambda = \frac{\eta_d}{\eta_e} \quad (1)$$

The viscosity ratio is important for the transmission of the deforming stresses acting from the continuous phase to the disperse phase during the emulsification process. At very low viscosity ratios ($\lambda \ll 1$), the viscosity of continuous phase is very high compared to the disperse phase viscosity. Due to the high

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Nomenclature

ALR	Air-to-liquid ratio by mass /-
ACLR	Air-Core-Liquid-Ring Nozzle
d	Diameter /mm
l	Length /mm
$x_{1,2}$	Sauter mean diameter / μm
η	Viscosity /mPa s
λ	Viscosity ratio /-
τ	Stress /Pa
γ	Interfacial tension /N/m
p_c	Capillary pressure /Pa
t	Time /s
\dot{m}	Mass flow /kg/s

Subscripts

c	Continuous phase
d	Disperse phase
e	Emulsion
g	Gas
l	Liquid
m	Mixing chamber
o	Orifice
oil	Oil drop size
spray	Spray drop size
initial	Initial oil drop size prior to atomization
def	Deformation

viscosity, oil drop breakup is elevated. At low viscosity ratios ($\lambda < 1$), stresses are transmitted to the disperse phase effectively. For a viscosity ratio range of 0.1–1, the oil drop breakup is eased. [43] At high viscosity ratios ($\lambda > 1$), the oil viscosity is higher than the surrounding emulsion viscosity. In general, the distorting tension is counteracted by the capillary pressure of the deformed drop. The dimensionless value, which combines both terms for laminar flow conditions, is the Capillary number Ca [43] (see Eq. (2)).

$$Ca = \frac{\tau_{\text{def}} \cdot X}{2 \cdot \gamma} \quad (2)$$

These findings hold true for simple shear flow, single drops in a couette or a four roll apparatus and have been published by Grace 1982 [43]. The influence of elongational flow on single drops has also been investigated by Bentley and Leal [47]. In elongational flow, the shear stress T_{def} is defined with the elongational viscosity and strain rate. Drop breakup is found at lower Capillary numbers and is hardly influenced by the viscosity ratio.

For oil drop breakup, the droplet deformation time must be exceeded. The droplet deformation time is given in Eq. (3) and further described in [48]:

$$t_{\text{def}} = \frac{\eta_d}{\tau_{\text{def}} - p_c} \quad (3)$$

The critical Capillary number is used to identify the value at which the distorting forces are high enough to break up a drop with a certain diameter and viscosity. Given the critical Capillary number, droplet deformation time and the viscosity ratio, the occurrence of oil drop breakup during a process could hence be predicted.

It has been demonstrated that the stresses during atomization are sufficient to break up the drops of a disperse phase. Munoz-Ibanez et al. [49] have shown that for rotary and external mixing pneumatic atomizers, the oil drop breakup depends on the viscosity ratio (with studied values of $\lambda = 0.07, 0.2$ and 0.6) and

on the initial oil drop diameter prior to atomization (with studied values between 0.1 and $1 \mu\text{m}$). The authors estimated the shear rates for their atomizer by models proposed by Ghandi et al. [50] and García et al. [51]. They found that the oil drop breakup was in concordance to values calculated from critical Capillary numbers for the rotary atomizer. For the external mixing pneumatic atomizer, however, they claimed a distinct divergence from Grace's theory.

For effervescent atomization, Schröder et al. [52] showed a dependence of the oil drop breakup on the viscosity ratio. In this study, the disperse phase content was 20 wt-%, which is why coalescence phenomena could not be excluded. In addition, the viscosity ratio was varied by the emulsion viscosity which influenced the stresses acting on the oil drops during atomization as well as on the spray drop size.

The effervescent atomization, which is also the focus of this study, is a subtype of internal mixing pneumatic atomization. In the outside-in geometry, which is investigated in the present study, the atomization gas enters the atomizer and the liquid through aerator holes [53,54]. Due to the mixing zone being situated inside the atomizer, the gas mass flow and liquid mass flow cannot be adjusted independently. The resulting two-phase flow pattern depends on the ALR (Air-to-Liquid-Ratio by mass) and the viscosity of the atomized liquid [54]. Kleinhans et al. [55] and Stähle et al. [56] have shown that slug flow inside mixing chamber leads to a coarse and undefined spray pattern. However, adjusting the process parameters allows a more well-defined two-phase flow pattern, i.e. an annular flow. Here, the atomization gas forms an air core with a liquid ring around it. The annular flow is also part of a describing model for the prediction of spray drop sizes produced by effervescent atomization [57]. With that model, shear rates in the atomizer nozzle can be calculated and used for the calculation of the Capillary number. This model is based on geometrical assumptions without considering liquid or even emulsion properties as viscosity, surface or interfacial tension. Therefore, within this study we will compare resulting oil drop sizes after atomization with those from defined flow conditions with easy-to-calculate shear rates and deduce shear rates in the atomization experiments from this.

Therefore the target of this study was to determine the influence of viscosity ratio (varied by the oil viscosity) and oil drop size prior atomization (initial oil drop size) on the oil drop size after atomization at otherwise constant process parameters. To evaluate the occurring shear rates for the calculation of the Capillary numbers for the atomization, the atomization results will be compared to the oil drop sizes after capillary rheometer experiments.

2. Material and methods

2.1. Emulsion properties

To adjust the initial oil drop size independently, the emulsions were prepared in two steps. In the emulsification step, emulsifier 0.3 wt-% Polysorbat20 (Tween20, Carl Roth GmbH+Co. KG, Karlsruhe, Germany) was first mixed with demineralized water. Subsequently, 9 wt-% silicone oil (polydimethylsiloxane, Wacker Chemie AG, München, Germany) was dispersed into this continuous phase to gain 500 g emulsion. For initial oil Sauter mean diameter above $2 \mu\text{m}$ (see Table 1), a colloid mill from IKA (magic LAB, IKA-Werke GmbH & CO. KG, Staufen, Germany) with two different modules (colloid mill and gear rim dispersing mill) was used to homogenize the initial emulsions. Emulsions with an initial oil Sauter mean diameter of $< 1 \mu\text{m}$ were homogenized by a high pressure homogenizer M110-Y from Microfluidics, Westwood, USA at 300 bar. For these trials, the amount of emulsifier was doubled.

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