

Effect of Atomizer Geometry and Rheological Properties on Effervescent Atomization of Aqueous Polyvinylpyrrolidone Solutions

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Abstract

The investigated effervescent atomizer is a special type of internal mixing pneumatic atomizer with a significantly reduced atomization gas consumption compared to conventional external mixing pneumatic atomizers. First the concentration influence of aqueous polyvinylpyrrolidone (PVP) solutions of concentrations from 15 to 45 % on the spray drop size distribution was investigated. As a significant increase in Sauter mean diameter and distribution width was only observed for concentrations above 40 % a change in breakup mechanism is assumed. This assumption is supported by the fact that also the improved model of Lund failed in the prediction of drop size at these concentrations. An essential challenge is the large width of the spray drop size distribution which is assumed to be connected to the spray pulsation. Therefore, the influence of nozzle orifice elongation and gas injection hole diameter reduction were investigated. Both depict no significant influence on Sauter mean diameter itself but reduced the standard deviation of Sauter mean diameter and the standard deviation of the distribution width enhancing thus the reproducibility of the spray characteristics. Also, reduction of the gas injection hole diameter reduced slightly the width of the distribution itself. The pulsation frequency of the spray was measured by high frequency laser diffraction spectroscopy. Nozzle orifice elongation and gas injection hole diameter reduction increased both the pulsation frequency as well as an increasing air-liquid mass ratio. The actual impact of the pulsation on the drop size distribution was not investigated in this work but will be future subject.

Introduction

The atomization of high viscous liquids like polyvinylpyrrolidone (PVP) solutions into a fine dispersed spray is a challenging task. It can be achieved with pneumatic atomizers but only with high atomization gas consumption. The effervescent atomizer is a special type of internal mixing pneumatic atomizer being distinct in the formation of a two phase flow prior to exiting the nozzle orifice [1]. The atomization gas consumption of effervescent atomizers is significantly reduced compared to conventional external mixing pneumatic atomizers [2,3]. This is favorable in processes with large throughputs and small profits margins like in spray drying of food products.

In this work, the effervescent atomization of aqueous PVP solutions and the effect of atomizer geometry and operating parameters on spray drop size distributions and spray pulsation were investigated. The width of the distribution is as important as the mean drop size because it can cause incomplete drying or thermal degradation of the particles during spray drying. Therefore, the focus of the investigation is on mean drop size and width of the drop size distribution.

Theory

A scheme describing the internal effervescent atomizer geometry is shown in figure 1 left. The atomization gas is injected in the mixing chamber and a two phase flow is formed. At ideal operating conditions, a bubbly flow regime is present in the mixing chamber. Depending on the nozzle geometry, fluid properties and operating parameters bubbly, slug or annular flow is present in the nozzle orifice due to different velocities and pressures. Main operating parameter is the air liquid mass ratio ALR according to equation 1. The thin liquid sheath surrounding the gas phase is then atomized by the expansion of the atomization gas at the orifice outlet [1].

$$ALR = \dot{m}_g / \dot{m}_l \quad (1)$$

Prior investigations [3,4] revealed pulsation of the spray depending on the atomization conditions and assumed a negative effect on the width of the drop size distribution. Jedelsky et al. [5] showed in atomization experiments with oil a reduction of the turbulence in the spray by elongation of the nozzle orifice length. Chin

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and Lefebvre [6] stated that the slight increase of spray Sauter mean diameter observed with increasing nozzle orifice length is due to friction loss.

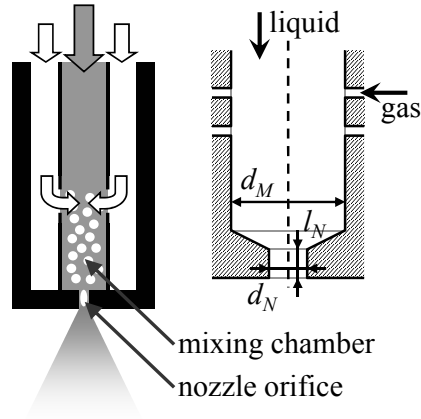


Figure 1. Sketch of effervescent atomizer. Gas is injected into the mixing chamber filled with liquid and forms a two phase flow which exits the atomizer through the nozzle orifice (left). Detail of the near orifice geometry with nozzle orifice diameter d_N and length l_N and mixing chamber diameter d_M (right).

The prediction of spray drop sizes in dependence of the liquid properties is of interest as they may vary in industrial applications due to changes in raw material. For prediction of spray drop sizes from effervescent atomization, Lund [7] proposed a two step calculation model. First, the thickness of the liquid filament sheath is calculated based on void fraction α and slip-ratio sr and the assumption of an annular flow regime in the nozzle orifice. A geometric calculation is used for calculation of the filament sheath breakup into ligaments. The second step of the calculation is based on a stability analysis of ligament fragmentation. The optimal wave length for ligament break-up is calculated with consideration of the interfacial tension and rheological properties. A more detailed description is given in a prior publication of the authors [3].

The model of Lund neglects the fluid properties like density and viscosity in the first step of the calculation. He proposed to use the iterative calculation method of Ishii [8] for calculation of void fraction α and slip ratio sr . Butterworth [9] summarized several methods for determination of the void fraction amongst them the correlations of Baroczy and Thom and the model of Turner & Wallis. Those three account for density ρ and viscosity η of gas and liquid as shown in equation 2, 3 and 4 with quality x .

$$\frac{1-\alpha}{\alpha} = \left(\frac{1-x}{x}\right)^{0.74} \cdot \left(\frac{\rho_g}{\rho_l}\right)^{0.65} \cdot \left(\frac{\eta_l}{\eta_g}\right)^{0.13} \quad (2)$$

$$\frac{1-\alpha}{\alpha} = \left(\frac{1-x}{x}\right)^{0.89} \cdot \left(\frac{\rho_g}{\rho_l}\right)^{0.89} \cdot \left(\frac{\eta_l}{\eta_g}\right)^{0.18} \quad (3)$$

$$\frac{1-\alpha}{\alpha} = \left(\frac{1-x}{x}\right)^{0.72} \cdot \left(\frac{\rho_g}{\rho_l}\right)^{0.4} \cdot \left(\frac{\eta_l}{\eta_g}\right)^{0.08} \quad (4)$$

The deviation of the calculated from the experimental measured Sauter mean diameter is calculated according to equation 5 and mean values are calculated for every concentration.

$$\text{prediction deviation} = \frac{x_{1,2;\text{calculated}} - x_{1,2;\text{experimental}}}{x_{1,2;\text{experimental}}} \quad (5)$$

Materials and Methods

PVP solutions were prepared using Luvitec K30 (BASF SE) and demineralized water. The properties of PVP solutions depend largely on concentration and polymerization degree, the latter commonly characterized by

the K value [10]. The water content of the PVP powder was measured regularly and considered for preparation of the solutions.

Concerning atomization, surface tension and rheological properties are amongst the most important liquid properties. Surface tension was measured with a Wilhelmy plate (DataPhysics DCAT 21). The rheological properties of the PVP solutions were determined by rotational and capillary rheometry at 25 °C. The rotational rheometer (Anton Paar Physica MCR 301) was equipped with a cone-plate geometry (CP-50-1/TG) or a coaxial cylinder (CC27) geometry depending on the viscosity of the solutions. The measurement was performed shear rate controlled from 0.1 to 1000 1/s. Since higher shear rates than 1000 1/s are expected in the atomization process, measurements with capillary rheometer were conducted in addition. The capillary rheometer (Rosand Rh2000) was equipped with a cylindrical capillary with a diameter of 0.315 mm and a length of 149.75 mm. The viscosity measurement was conducted at stabilized pressure. As both shear and extensional stress occurs in atomization, also extensional rheometry (HAAKE CaBER 1) was used for measurement of the rheological properties with an initial gap height of 3.0 mm. All measurements of rheological properties and surface tension were performed at least in triplicate. For calculation of liquid mass flow, density was determined with a measuring cylinder and a balance at least from two different experiments. Refractive index was measured with a refractometer (Carl-Zeiss) in triplicate.

A modular atomizer design [3] was used with four different nozzle orifice length l_N of 1.5 (D15), 3.0 (D30), 4.5 (D45) and 6.0 mm (D60) respectively. Nozzle orifice and mixing chamber diameter was kept constant at 1.5 and 6.0 mm, respectively. A scheme of the atomizer geometry is shown in figure 1 right. The gas injection holes were arranged asymmetrically in two or five rows and the total cross sectional area was kept constant. Either two rows of holes with a diameter of 0.5 mm and a total number of 9 or five rows of holes with a diameter of 0.3 mm and a total number of 25 were the latter is referred to as fine in the following text.

The atomizer was installed in a test rig above a vent to prevent recirculation of the spray into the measurement zone. The liquid was pumped pulsation free with a gear or eccentric screw pump and the volume flow was measured by a gear volume flow indicator (VSE VSI 04/16). The gas flow was controlled by a mass flow controller (Bronkhorst High-Tech EL-FLOW) based on the heat capacity of the gas. For spray characterization, a laser diffraction spectroscopy (Malvern Spraytec) equipped with a 750 mm range lens was used under consideration of the respective refractive indexes according to Mie theory. The temporal resolution was set to 1 Hz in continuous mode and data acquisition was at least 20 s for each data point. The laser was located 25 cm below the nozzle orifice and passes through the center line of the spray. The distance was chosen to ensure complete atomization of the solutions. Due to a strong influence of beam steering effects, the scattering data of the two detectors nearest to the laser axis were not considered for the analysis as proposed by Mescher and Walzel [12].

The Sauter mean diameter $x_{1,2}$ was used as characteristic mean value of the spray drop size distribution. The area weighted standard deviation s_2 was selected as a measure of the distribution width as shown in equation 6. The standard deviation of Sauter mean diameter and standard deviation s_2 is calculated from three separate experiments except D15 at 15 % PVP which has no standard deviation.

$$s_2^2 = \int_{x_{\min}}^{x_{\max}} (x - \bar{x}_{1,2})^2 \cdot q_2(x) dx \quad (6)$$

For measurement of the pulsation frequency two measurement methods were used. Comparable to the measurement of the number of revolutions of a turbine, a stroboscope (Rheintacho Pocket LED) was used. By adjustment of the flash frequency and the phase shift, the stroboscope was set to the same frequency as the pulsation of the spray. For the second method the laser diffraction spectroscopy was used to detect the variation of characteristic values depending on the pulsation by increasing the temporal resolution in rapid mode. Transmission, $x_{10,3}$, $x_{50,3}$, $x_{90,3}$ and $x_{1,2}$ were selected from the acquired data with a duration of 25 s. The pulsation frequency was determined by fitting a sine function (OriginLab Origin 8G). To our knowledge, the latter method has not been used for measurement of pulsation frequency of effervescent atomizers until now. Loskand et al. [13] proposed recently acoustical and high speed video data acquisition and application of Fourier transformation for determination of pulsation frequency but these methods were not applied in this work.

Results and Discussion

The results of solution properties (surface tension, density, refractive index) in dependence of PVP concentration are displayed in table 1. The static surface tension shows no dependency on PVP concentration whereas the density and refractive index increase with increasing PVP concentration.

Table 1. Solution properties as a function of PVP concentration.
Italic formatted values are derived from exponential (density) or linear (refractive index) fit.

	15 %	20 %	30 %	40 %	45 %	50 %
surface tension / mN/m	-	64.4 ± 1.0	65.2 ± 0.2	64.6 ± 0.2	-	63.0 ± 0.1
density / kg/m ³	<i>1048.1</i>	1049.2 ± 1.1	1061.2 ± 6.3	1083.0 ± 2.6	1102.4 ± 9.2	1139.3 ± 12.5
refractive index / -	<i>1.36</i>	1.37 ± 0.0	1.39 ± 0.0	1.41 ± 0.0	1.42 ± 0.0	-

Viscosity curves from shear rheological measurements are displayed in figure 2 left. Only every second point from rotational measurements is shown in the graph to improve visualization. The further points are in good agreement to those shown. Data from rotational and capillary measurement agree well. At low shear rates a Newtonian behavior is observed which changes into a shear thinning behavior at high shear rates. This change occurs with increasing solution concentration at lower shear rates. The slight viscosity increase observed above 10^2 1/s for a concentration of 15 % is likely due to Taylor instabilities. The results from the extensional measurement are shown in figure 2 right. Only every second measurement point is shown in the graph to improve visualization. The further points are in good agreement to those shown. They depict roughly a Newtonian behavior in the investigated shear rate range. The dashed lines are the theoretical viscosity values expected for comparison of shear and extensional viscosity in case of ideal Newtonian behavior according to Trouton [10]. With increasing concentrations, the extensional measured values and from shear measurement derived theoretical values are in good agreement. Only at the lowest investigated concentration of 20 %, a significant deviation is observed. Overall, it can be stated that the solutions depict a Newtonian behavior in shear and extensional stress at shear or strain rates below 10^3 to 10^5 1/s depending on the PVP concentration. Above these shear rates, a shear thinning is observed.

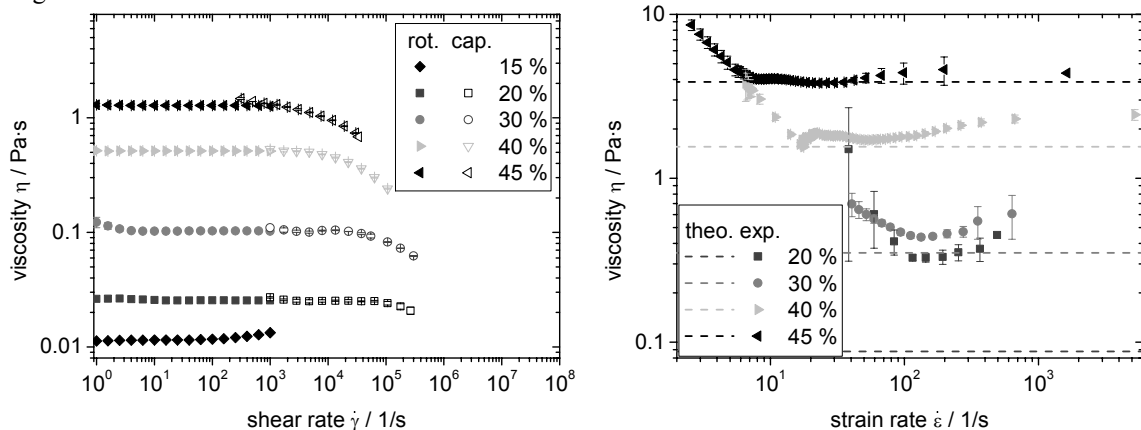


Figure 2. Viscosity curves as a function of shear rate and PVP concentration.
Rotational and capillary measurements (left) and extensional measurements (right).

Figure 3 left presents the Sauter mean diameter and standard deviation s_2 as a function of ALR for different PVP concentrations. Both, Sauter mean diameter and standard deviation s_2 decrease with increasing ALR as expected. Only minor increase of the Sauter mean diameter and standard deviation s_2 is observed up to 40 % PVP. This invariance to different liquid properties was already observed in prior work [3].

The pulsation of the spray is assumed to have a negative effect on the width of the drop size distribution. Two possibilities to reduce this are investigated in the further work: the influence of smaller gas injection hole diameter at same total cross sectional area and the elongation of the nozzle orifice. A more homogenous two phase flow is expected with the reduction of the gas injection hole diameter. The influence of gas injection geometry on spray drop size distribution at constant PVP concentration as a function of ALR is displayed in figure 3 right. No major change of the Sauter mean diameter is observed but a slight decrease of the standard deviation s_2 and its standard deviation for atomizer geometry D15 fine. The latter may be attributed to a better reproducibility of the spray measurement due to a more uniform two phase flow insight the atomizer and therefore a reduced pulsation.

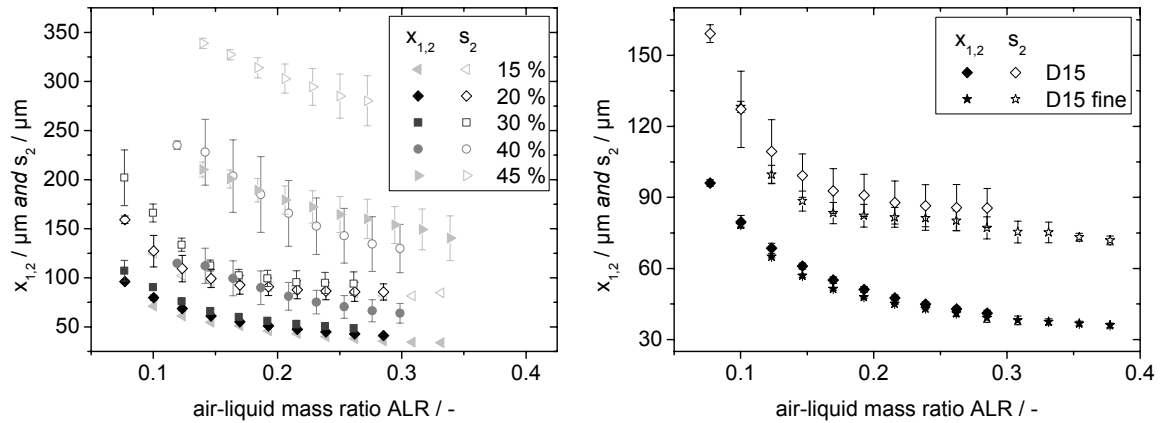


Figure 3. Influence of PVP concentration on spray drop size distribution as a function of ALR with atomizer geometry D15 (left). Influence of gas injection geometry on spray drop size distribution at constant PVP concentration as a function of ALR (right).

By elongation of the nozzle orifice a reduced turbulence is expected [5] and therefore a reduced standard deviation s_2 . Figure 4 presents the Sauter mean diameter (left) and standard deviation s_2 (right) as a function of ALR at different PVP concentrations. The Sauter mean diameter increases slightly with increasing nozzle orifice length at 20 and 30 % PVP as described before [6]. The standard deviation s_2 increases slightly with increasing nozzle orifice length. A significant decrease in the standard deviation of the Sauter mean diameter and the standard deviation s_2 is observed for increased nozzle orifice length at PVP concentrations of 40 and 45 % (not shown here). This decrease in the standard deviation is a positive effect on the reproducibility of the experimental data but does not improve the width of the drop size distribution overall. The better reproducibility may be attributed to a more favorable spray pulsation.

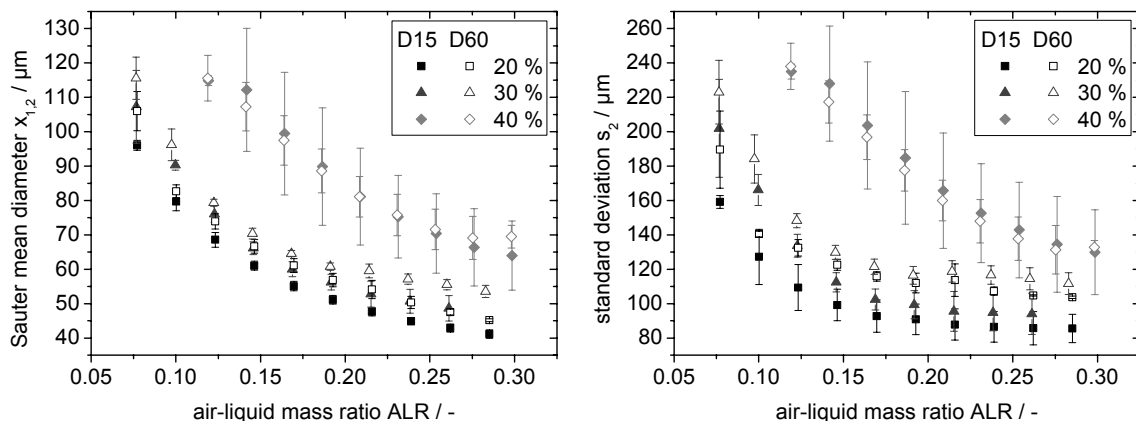


Figure 4. Characteristic values of spray drop size distributions as a function of ALR , PVP concentration and nozzle orifice geometry. Sauter mean diameter $x_{1,2}$ (left) and standard deviation of drop size distribution s_2 (right).

Therefore the target was to determine the influence of solution properties, atomizer geometry and ALR on the pulsation frequency as a measure of the pulsation. An example of the time dependent behavior of $x_{10,3}$ and $x_{90,3}$ of a spray from atomizer geometry D15 at an ALR of 0.139 measured at 250 Hz with the rapid mode laser diffraction spectroscopy is shown in figure 5. To obviate influence of the measurement frequency on the detected pulsation frequency, measurements with 50 Hz, 250 Hz and 1 kHz were performed as shown in table 2. As no significant change in pulsation frequency was observed for the selected frequencies, 250 Hz was chosen for the further measurements which were conducted in triplicate. This ensured adequate temporal resolution even at high pulsation frequencies. For validation, frequency measurements with stroboscope were performed. Due to the slight irregular pulsation, this method is not practical for this purpose but the measured frequency ranges by stroboscope are in good agreement with the frequencies measured by laser diffraction spectroscopy.

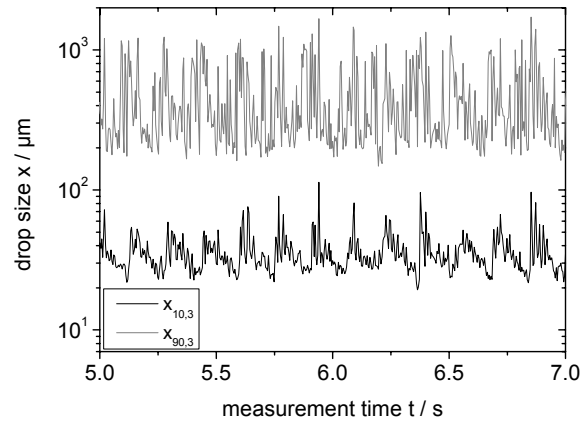


Figure 5. Example of the time dependent behavior of $x_{10,3}$ and $x_{90,3}$ of a spray from atomizer geometry D15 at an ALR of 0.139 and 20 % PVP measured at 250 Hz.

Table 2. Influence of measurement frequency on measured pulsation frequencies for two atomizer geometries at constant PVP concentration of 20 %. Standard deviations calculated from fit data of five characteristic values transmission $x_{10,3}$, $x_{50,3}$, $x_{90,3}$ and $x_{1,2}$ are below 1.2 %.

ALR	D15 50 Hz	D15 250 Hz	D15 1 kHz	D60 50 Hz	D60 250 Hz	D60 1 kHz
0.231	6.97 Hz	7.04 Hz	6.78 Hz	8.85 Hz	8.64 Hz	8.73 Hz

The influence of the nozzle orifice length l_N and gas injection hole diameter was investigated in a PVP concentration range from 15 to 40 % but it was not possible to fit reliably a pulsation frequency at concentrations above 20 %. Therefore only the results for 15 and 20 % are displayed in table 3. Almost all data depict a pulsation frequency increase with increasing ALR and decreasing PVP concentration. The influence of the nozzle orifice length is shown in figure 6 left. They depict a continuous frequency increase with increasing ALR . Overall, a maximum in frequency can be observed for D30 and D45. However, the results of D45 have to be considered carefully due to the high standard deviations. The influence of the gas injection geometry is shown in figure 6 right. The overall trend of a frequency increase with increasing ALR is confirmed and an increase of 1 Hz can be seen for D15 fine compared to D15.

Table 3. Measured pulsation frequencies at 250 Hz as a function of ALR , PVP concentration and atomizer geometry.

ALR	D15 15 %	D15 20 %	D15 fine 20 %	D30 20 %	D45 20 %	D60 15 %	D60 20 %
0.139	7.19 ± 0.09 Hz	6.22 ± 0.27 Hz	7.38 ± 0.03 Hz	8.70 ± 0.15 Hz	9.14 ± 0.01 Hz	8.55 ± 0.03 Hz	7.20 ± 0.18 Hz
0.231	9.44 ± 0.14 Hz	7.58 ± 0.47 Hz	8.51 ± 0.35 Hz	9.06 ± 1.19 Hz	11.15 ± 1.63 Hz	9.80 ± 0.04 Hz	8.84 ± 0.17 Hz
0.324	9.34 ± 0.03 Hz	8.82 ± 0.20 Hz	9.57 ± 0.23 Hz	10.33 ± 0.02 Hz	8.89 ± 3.56 Hz	8.80 ± 0.03 Hz	9.88 ± 0.16 Hz

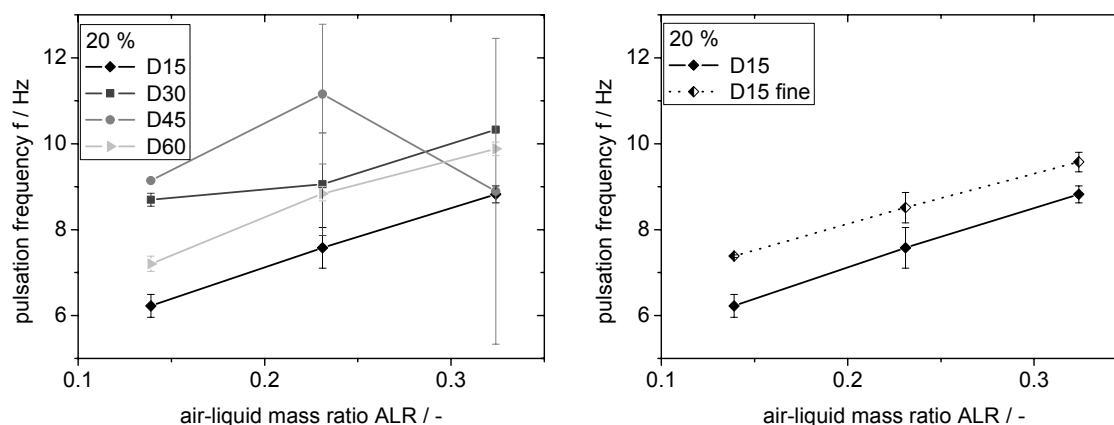


Figure 6. Influence of the nozzle orifice length (left) and the gas injection geometry (right) on pulsation frequency at constant PVP concentration as a function of ALR .

Comparing the slight decrease in standard deviation s_2 and its standard deviation (figure 3 right), the slight decrease of the standard deviation with increasing nozzle orifice length (figure 4) and the simultaneous increase in pulsation frequency (figure 6), it can be concluded that an increase in pulsation frequency results in a decrease of standard deviation of the standard deviation s_2 and thus a better reproducibility of the results. So far, this was only validated for D15 and D60 and not for the maximal frequency with D30 and D45. In combination with the fine injection geometry, even a slight decrease of the standard deviation s_2 is achievable.

Further on, the experimental data shown before were used to improve the model of Lund for the prediction of spray drop sizes. For this purpose, the void fraction was also calculated in dependence of density and viscosity according to the correlations of Baroczy and Thom and the model of Turner & Wallis. The calculation step of the slip ratio was adopted from Ishii. An exemplary data set as function of ALR is shown in figure 7 left. It can be seen that all methods reveal the tendency of decreasing Sauter mean diameter with increasing ALR but the correlations of Baroczy and Thom fit better than the others. Figure 7 right shows the overall mean prediction deviation from experimental data as a function of PVP concentration. It can be observed that up to 40 % PVP the correlations of Baroczy and Thom fit the experimental data the best. Above 45 %, all methods depict a significant increase of deviation. Overall, it can be concluded that the consideration of the fluid properties improve the prediction results significantly.

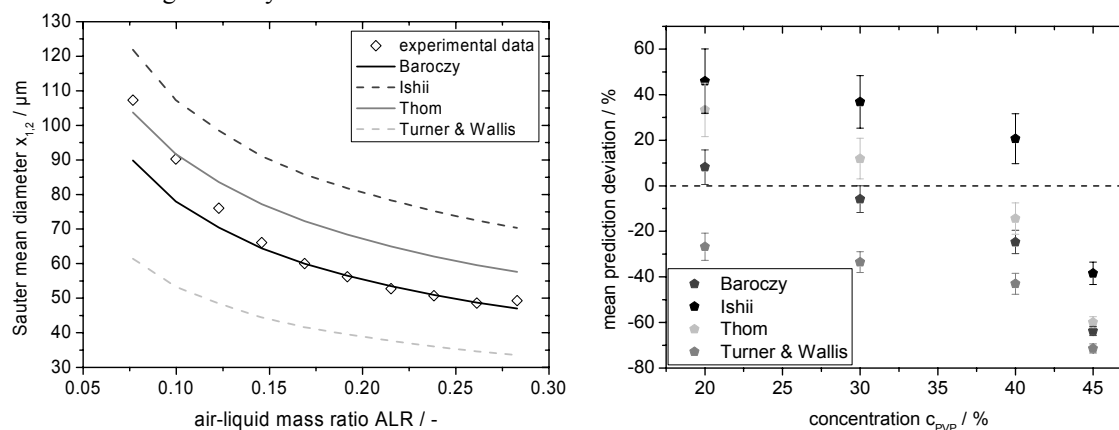


Figure 7. Experimental data of D15 and 30 % PVP and corresponding calculated values as a function of the ALR (left). Overall mean prediction deviation from experimental data as a function of PVP concentration (right).

By comparison of the significant increase of Sauter mean diameter and standard deviation s_2 at 45 % shown in figure 3 left and the failure of the improved prediction model at 45 % depicted in figure 7 right, it can be presumed that above 40 % PVP a change in the flow regime occurs due to the higher viscosity which has a major effect on the breakup mechanism in the atomization. As mentioned above, the shear rates present in the atomizer are estimated to be above 10^3 1/s. Therefore, the change of Newtonian to shear thinning behavior as shown in figure 2 left could be of relevance for this change, too. The surface tension is not expected to be of relevance due to its invariance to PVP concentration.

Summary and Outlook

The effervescent atomization of aqueous PVP solutions of up to 45 % was investigated. At concentrations above 40 %, a change of the breakup mechanism is assumed due to the significant increase of Sauter mean diameter and standard deviation s_2 of the spray drop size distribution. This assumption is supported by the fact that also the improved model of Lund failed in the prediction of drop size at these concentrations. All PVP solutions showed Newtonian behavior up to a distinct shear rate. For higher shear rates shear thinning behavior was observed. The shear rate depends on the PVP concentration and increased from 10^3 1/s at 45 % up to 10^5 1/s at 20 %.

The change of nozzle orifice length has no significant influence on either Sauter mean diameter $x_{1,2}$ or standard deviation s_2 . Thus, no positive effect concerning the width of drop size distribution is found. Only an improved reproducibility (decrease in standard deviation of $x_{1,2}$ and s_2) of experimental results at high PVP concentrations is observed for the longest nozzle orifice. The finer gas injection geometry results also in a small decrease of the standard deviation s_2 . This needs to be investigated further. Nozzle orifice elongation and gas injection hole diameter reduction increased both the pulsation frequency as well as an increasing air-liquid mass ratio. The actual impact of the pulsation on the spray drop size distribution will be focus of future investigations. Therefore, the acquired laser diffraction data from rapid mode measurements will be used.

The prediction model of Lund is improved by consideration of the fluid properties. Still, the calculation of the ligament diameter neglects these properties and further work is therefore needed to improve the model.

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Nomenclature

ALR	air-liquid mass ratio / -
c_{PVP}	concentration / %
d_M	mixing chamber diameter / mm
d_N	nozzle orifice diameter / mm
l_N	nozzle orifice length / mm
\dot{m}	mass flow / kg/h
PVP	polyvinylpyrrolidone
q_2	area weighted probability density function / $1/\mu\text{m}$
s_2	standard deviation of q_2 / μm
sr	slip ratio / -
x	quality / -
x	diameter at interval middle / μm
$x_{1,2}$	Sauter mean diameter / μm
$x_{10,3}$	diameter at 10 % of volumetric cumulative drop size distribution / μm
$x_{50,3}$	volumetric mean diameter / μm
$x_{90,3}$	diameter at 90 % of volumetric cumulative drop size distribution / μm
α	void fraction / -
$\dot{\epsilon}$	strain rate / 1/s
η	dynamic viscosity / Pa·s
$\dot{\gamma}$	shear rate / 1/s
ρ	density / kg/m^3
σ	surface tension / N/m

Subscripts

l	liquid
g	gas

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