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RESEARCH PAPER

Design and Atomization Properties for an Inside-Out Type Effervescent Atomizer

Frederik Jacob Petersen,[1,*] Ole Wørts,[1] Torben Schæfer,[1] and Paul E. Sojka[2]

[1]Department of Pharmaceutics, The Danish University of Pharmaceutical Science, Copenhagen, Denmark
[2]School of Mechanical Engineering, Purdue University, West Lafayette, Indiana, USA

ABSTRACT

Atomization of aqueous polymer solutions is a key step in the formulation of several pharmaceutical products. Droplet size control is essential in order to produce pharmaceutical products with the desired properties. The purpose of this paper is to investigate design issues for an inside-out type of effervescent atomizer used to spray water and aqueous solutions of polyvinylpyrrolidone (Kollidon® K-30) and hydroxypropyl methylcellulose (Pharmacoat® 603). The atomizer was operated at air-to-liquid mass ratios of 0.1, 0.3, and 0.5 and a feed pressure of 1172 kPa. Fluid viscosities ranged from 1 to 47 mPa.s. The influence of several atomizer design features was considered, including exit orifice length-to-diameter ratio, exit orifice diameter, the total area of the air injection holes, the distance between the air injection point and the exit orifice, the diameter of the mixing chamber, and the orientation of both air and liquid flows. Droplet size distributions were shown to vary significantly with the atomizer's exit orifice diameter, air injector design, and air injector distance to the exit orifice. In all cases air-to-liquid mass ratio played a key role in the mean droplet size. The design of the atomizer was shown to have the most pronounced effect on the mean droplet size at the lowest air-to-liquid mass ratios. Optimization of the atomizer design is very important in order to obtain small droplet sizes in pharmaceutical processes where the amount of air/gas should be minimized, e.g., closed-cycle spray drying and agglomeration processes.

Key Words: Effervescent atomization; Atomizer design; Exit orifice; Air injector; Droplet size.

*Correspondence: Frederik Jacob Petersen, M.Sc. Pharm., Department of Pharmaceutics, The Danish University of Pharmaceutical Science, Universitetsparken 2, DK-2100 Copenhagen, Denmark; Fax: +45-3-530-6031; E-mail: fjp@dfh.dk.

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INTRODUCTION

Atomization of aqueous polymer solutions is widely used in the pharmaceutical industry. It is an essential step in the granulation, coating, and spray-drying processes that control the physical properties of the pharmaceutical product. It has been shown that effervescent atomization has the potential to form acceptable pharmaceutical sprays.[1] The effervescent atomizer is an interesting alternative for the formation of sprays with small mean droplet sizes in pharmaceutical processes where the amount of air/gas should be minimized, e.g., in closed-cycle spray drying where the amount of nitrogen should be minimized due to the cost of the process.

Atomization is a difficult task to accomplish when the purpose is to produce sprays of a small mean droplet size and a narrow droplet size distribution. Such sprays are of special interest in the production of pharmaceuticals of fine particle sizes for pulmonary delivery[2] or in the preparation of coated particles having diameters less than 100 μm.[2,3]

Effervescent atomization is one possible solution. It is essentially an internal, two-fluid mixing technique, where the liquid is exposed to an atomizing gas before leaving the atomizer body. The gas and the liquid are separately injected into a mixing chamber, forming a two-phase gas-liquid flow exiting the atomizer exit orifice. Liquid break-up in effervescent atomization is initiated by aerodynamic shear forces generated by the injection of atomizing gas.[4] At low air-to-liquid mass ratio (ALMR), gas leaves the exit orifice as small bubbles surrounded by liquid.[5] As ALMR increases so does bubble coalescence. This leads to separate slugs of gas being formed and passed through the exit orifice. As a result, energy transfer is less efficient.[5] This phenomenon differs from other twin-fluid atomizers where high velocity gas is used to break up the liquid into droplets.[6] Effervescent atomization differs from conventional twin-fluid atomization in that energy and momentum transfer from gas to liquid no longer take place through boundary layers located at either side of the interface, instead being the result of intimate gas and liquid mixing.

Effervescent atomization offers advantages over conventional twin-fluid atomizers such as smaller droplet size at equal ALMRs, or equivalent droplet sizes at lower ALMRs than those used in conventional twin-fluid devices.[1,7] Both are beneficial in pharmaceutical processes such as closed-cycle spray drying and agglomeration.

It has previously been reported[5–13] that ALMR is the process variable having the largest influence on spray droplet size, with a higher ALMR leading to smaller droplets. The greatest impact of ALMR is found at values less than 0.15. The effect diminishes as ALMR increases beyond this value.

Liquid feed pressure has also been shown to play an important role in effervescent atomizer performance. An increase in liquid feed pressure leads to a decrease in droplet size. Furthermore, liquid feed pressure has its greatest influence on droplet size when the ALMR is lower than 0.15 because of superior energy transfer from the gas to the liquid.[13]

It has been reported that spray droplet size is related to the physical properties of sprayed liquids, to the atomizer design,[6,8,9] and to the atomizer operating conditions. In general an increase in viscosity enhances the ability of the fluid to resist the dynamic force associated with the atomizing gas leading to larger droplets. Effervescent atomizers have been shown to be able to atomize different types of polymer solutions with viscosities up to 228 mPa.s.[1]

While considerable work has been done to study the influence of operating conditions, physical properties, and atomizer geometry on effervescent atomizer performance, those studies have either focused on nonNewtonian fluids while neglecting the influence of atomizer internal design[1,5,14] or have considered the impact of internal geometry on spray performance but have been limited to Newtonian liquids.[4,6–9,15] Therefore, the influence of exit orifice length-to-diameter ratio, air injector design, and air injector area has yet to be considered when spraying pharmaceutical fluid systems.

With effervescent atomization, the internal flow structure is far more complex than with pneumatic atomizers because although the gas is injected into the liquid in a manner designed to create a bubbly flow, the flow pattern within the mixing chamber could be in a slug, annular, or even a dispersed flow regime. Different flow patterns inside the atomizer dictate different mechanisms for atomization processes and also strongly affect spray characteristics.[12] One study has shown that one of the most important parameters affecting atomization performance is the ratio of the exit orifice area to the area of the air injection holes,[9] while another claims no effect of air injector area on mean droplet size.[6] It has also been shown that the optimum size of the mixing chamber diameter is dependent on the atomizer design and operating conditions,[9] and that the length/diameter ratio of the final exit orifice is of great importance to atomizer performance. A reduction of length/diameter ratio is supposed to improve atomization quality, but due to technical reasons ratios below 0.5 are not recommended.[9,15] For these reasons there is disagreement in the literature on how aerator geometry influences effervescent atomizer performance.

Table 1. Viscosity and surface tension of solutions sprayed.

Liquid	Dynamic viscosity (mPa.s)	Surface tension (mN/m)
Water	1	71
Kollidon K-30 10%	6	58
Pharmacoat 603 10%	46	44

The purpose of this paper is to characterize the performance of effervescent atomizers by examining the effects of exit orifice diameter, exit orifice length, air injector area, air injector design, and ALMR on spray droplet size distribution for an inside-out design unit spraying liquids typical of pharmaceutical applications. Water and aqueous solutions of polyvinylpyrrolidone and hydroxypropyl methylcellulose were used.

MATERIALS AND METHODS

Polymer Solutions

Water and aqueous solutions of different polymers were sprayed. They include 10% (w/w) concentrations of polyvinylpyrrolidone (PVP) (Kollidon® K-30, BASF, Ludwigshafen, Germany) and hydroxypropyl methylcellulose (HPMC) (Pharmacoat® 603, Shin-Etsu Chemicals, Niigata, Japan).

Viscosity and Surface Tension Measurements

Shear viscosities of water and polymer solutions were measured using a Haake viscometer (Haake RV 20, Thermo Haake, Karlsruhe, Germany). All measurements were performed at 20°C using a NV II rotor configuration. The measurements were carried out at the highest rev/min that gave rise to a deflection on the scale. Results indicated no dependence of viscosity on rotor rotation speed, so reporting viscosity as a single value at a single rev/min value is justified.

Surface tensions of water and the polymer solutions were measured using a Krüss tensiometer (model K-10ST, Krüss GmbH, Hamburg, Germany). All measurements were performed at 20°C.

The mean viscosities and surface tensions of three measurements of water and the polymer solutions are shown in Table 1.

Effervescent Atomizer

The liquids were atomized using air and an effervescent inside-out atomizer as described by Petersen et al.[1] The atomizer exit orifice, mixing chamber, and air injector were replaceable. Exit orifices of 0.5, 0.75, and 1.0 mm in diameter with a corresponding length of 3.75 mm were available as well as two additional exit orifices of 0.75 mm diameter with lengths of 0.75 and 1.88 mm. Replaceable mixing chamber tubes had diameters of 3.0, 3.5, and 4.6 mm. Different air injectors having either smooth or helically shaped surfaces with air exit hole(s) according to Table 2 were also considered.

The atomizer liquid and gas control system employed in this study is identical to the one previously used by Petersen et al.[1]

Determination of Spray Droplet Size

The spray droplet size data are reported in terms of the Sauter mean diameter (D_{32}). D_{32} is the diameter of the droplets whose ratio of volume-to-surface area corresponds to that of the entire spray.[16] The droplet size distributions were measured using a Malvern 2600

Table 2. Air injector design.

Injector no.	Surface	Number of injector holes	Position of injector hole(s)—angle to flow direction (°)	Area of injector hole(s) (mm^2)
1	Smooth	2	0/45	0.14
2	Smooth	2	0/45	0.25
3	Smooth	2	0/45	0.39
4	Smooth	1	0	0.13
5	Smooth	1	45	0.13
6	Smooth	2	45/45	0.25
7	Helical	1	0	0.13
8	Helical	1	45	0.13
9	Helical	2	45/45	0.25

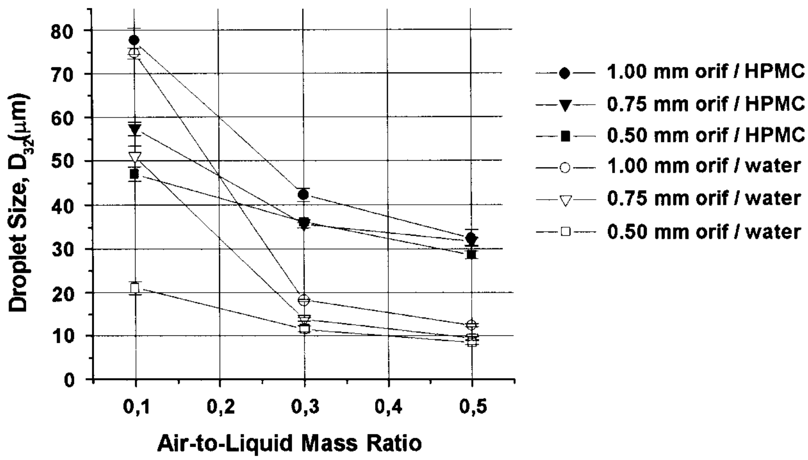

Figure 1. Effect of exit orifice diameter: Droplet size (D_{32}) vs. air-to-liquid mass ratio (ALMR) for water and 10% w/w Pharmacoat 603 (HPMC). Exit orifice diameter: 0.5, 0.75, or 1.0 mm. Exit orifice length: 3.75 mm. Liquid flow rate: 35 g/min.

C particle sizer (Malvern Instruments, Worcester, United Kingdom), using the principles of Fraunhofer diffraction pattern analysis. The atomizer exit orifice opening was positioned along a vertical line at a distance of 15 cm above the path of the laser beam and with the back edge of the spray at a horizontal position from the lens as calculated by Petersen et al.[1] A 100-mm focal length lens with a diameter of 46 mm was used in this study. Spray data were measured on the centerline of the spray and processed using the Malvern model independent mode. All measurements were done at 20°C and estimated on behalf of 1000 sweeps of the Malvern detector. Three replicas of the measurements were made for each setting. The average droplet diameter for the set of three replicas and the corresponding standard deviation are reported in Figs. 1–5.

RESULTS AND DISCUSSION

Exit Orifice Length-to-Diameter Ratio

The relationship between exit orifice length-to-diameter ratio and droplet size was investigated at a fixed exit orifice diameter of 0.75 mm, with length/diameter ratios of 1, 2.5, or 5 corresponding to exit orifice lengths of 0.75, 1.88, or 3.75 mm. The results showed no effect of length/diameter ratio on D_{32} at an ALMR of 0.1 and only a minor decrease in D_{32} at

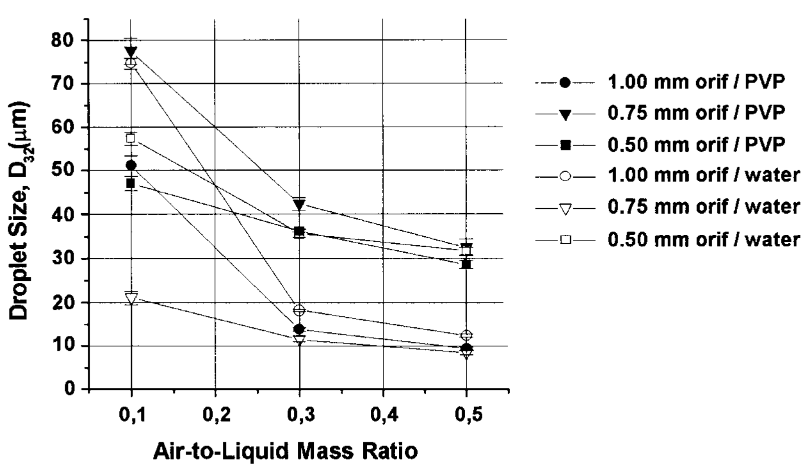

Figure 2. Effect of exit orifice diameters: Droplet size (D_{32}) vs. air-to-liquid mass ratio (ALMR) for water and 10% w/w Kollidon K-30 (PVP). Exit orifice diameter: 0.5, 0.75, or 1.0 mm. Exit orifice length: 3.75 mm. Liquid flow rate: 35 g/min.

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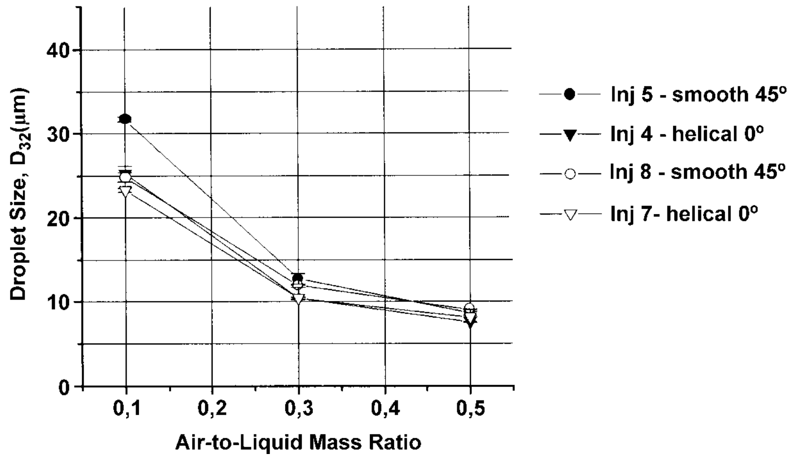

Figure 3. The influence of air/liquid injection geometry: Droplet size (D_{32}) vs. air-to-liquid mass ratio (ALMR) for water for atomizers having four different air injector designs. Exit orifice diameter: 0.75 mm. Exit orifice length: 3.75 mm. Number of injector holes: 1. Liquid flow rate: 35 g/min. Injector numbers refer to Table 2.

ALMRs of 0.3 and 0.5 when changing the length/diameter ratio from 5 to 1 for water as well as for polymeric solutions of PVP and HPMC. Results obtained with the present effervescent atomizer are less pronounced than previously published and therefore do not confirm the general claims of previous studies.[6]

Exit Orifice Diameter

D_{32} values for water sprays presented in Fig. 1 are consistently below corresponding values for HPMC. This behavior is expected and is consistent with previously published atomization studies of polymer solutions.[5] Figure 1 further shows that D_{32} usually decreases with a decrease in exit orifice diameter. This effect is most pronounced at ALMR values of 0.1. It is considerably less noticeable at an ALMR of 0.3, and nearly disappears as ALMR reaches 0.5. The likely explanation is the increased importance of secondary atomization, where the initial liquid droplets are broken up into smaller droplets. The products of secondary atomization are far less dependent than primary atomization on the characteristic size of the atomizer exit orifice.

The difference between water and HPMC droplet size values becomes more pronounced at higher ALMRs. Based on the surface tension data this behavior was unexpected since secondary atomization is likely to become more important as ALMR exceeds 0.1, and the lower surface tension exhibited by the HPMC (Table 1) suggests that HPMC would form smaller mean droplet size sprays.[16] There are three possible explanations: 1) dynamic surface tension effects are important and the static surface tension values reported in Table 1 do not reflect these effects; 2) Ohnesorge number values for HPMC ($\sim$0.3 for the 1-mm diameter exit orifice) are so much greater than the corresponding ones for water ($\sim$0.004) that viscous effects become important during secondary atomization. The Ohnesorge number is the ratio of the square root of the Weber number to the Reynolds number, a ratio of aerodynamic forces/surface tension forces divided by a ratio of aerodynamic forces/viscous forces;[16] and 3) HPMC elasticity is resisting the aerodynamic shear force that causes secondary atomization.

Droplet size values for the sprays of water and PVP as presented in Fig. 2 also show that droplet size usually increases with an increase in exit orifice diameter. Similar to Fig. 1, this effect is most pronounced at ALMR values of 0.1, is less noticeable at an ALMR of 0.3, and even smaller as ALMR reaches 0.5. Note, however, that there is still some variation in droplet size with exit orifice diameter at even the highest ALMR considered here. The likely explanation is again the increased importance of secondary atomization. In contrast to the HPMC data the PVP results suggest that the primary atomization process here plays a larger role throughout the range of ALMRs considered. This explanation is supported by comparing Fig. 1 data with Fig. 2 data where the influence of viscosity on secondary atomization is estimated by the ratio of the viscous to surface tension forces. Ohnesorge number values for HPMC ($\sim$0.3 for the 1-mm diameter exit office) are much greater than the corresponding ones for PVP ($\sim$0.027). The higher Ohnesorge numbers for HPMC are consistent with the larger droplets produced with HPMC, especially

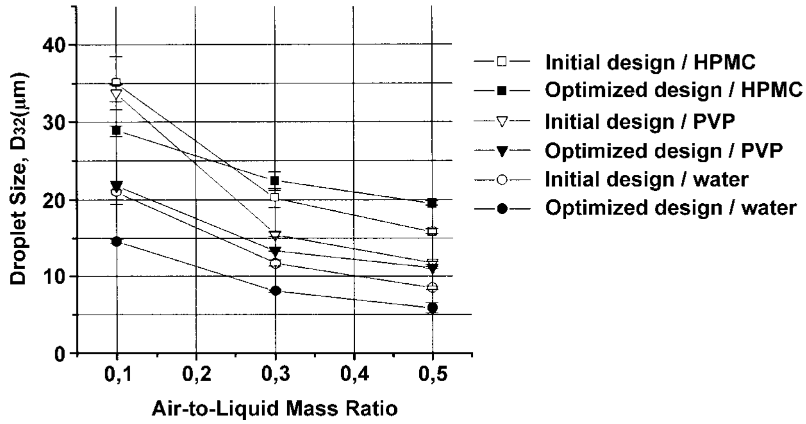

Figure 4. Performance of initial (I) and optimized (O) atomizer design on droplet size (D_{32}) vs. air-to-liquid mass ratio (ALMR). Exit orifice (I) 0.6 mm, (O) 0.5mm; exit orifice length-to-diameter ratio (I) 2.5, (O) 2.5; air-injector distance to orifice-exit (I) 5 mm, (O) 3.5 mm; air-injector (I) two outlets of 0.25 mm^2 (0° parallel to the bulk flow) and 0.14 mm^2 (45° to the bulk flow), (O) a helical-shaped air injector with one outlet of 0.13 mm^2 (0° parallel to the bulk flow); mixing chamber (I) 4.6 mm, (O) 3.0 mm. Liquids: Water, 10% Kollidon K-30 and 10% Pharmacoat 603. Liquid flow rate: approximately 35 g/min.

since viscous effects are known to be important for Ohnesorge numbers of 0.1 and greater, and supporting the claim that viscous effects are more important during secondary atomization for HPMC than for PVP.

Area of Air Injector Holes

The results showed no change in water spray droplet size with a change in air injector hole area, regardless of the value of ALMR or exit orifice diameter. Equivalent behavior was observed for all polymer fluids sprayed. This observation indicates that the internal two-phase flow regime is the same in all cases. The practical impact of this finding is that large diameter aerator holes can be employed to reduce clogging without fear of degradation in atomizer performance, i.e., an increase in D_{32}.

Air Injector Design

The influence of air/liquid injection geometry was tested using air injector designs according to Table 2. Two design features were considered, the angle between the gas injection and bulk liquid flow

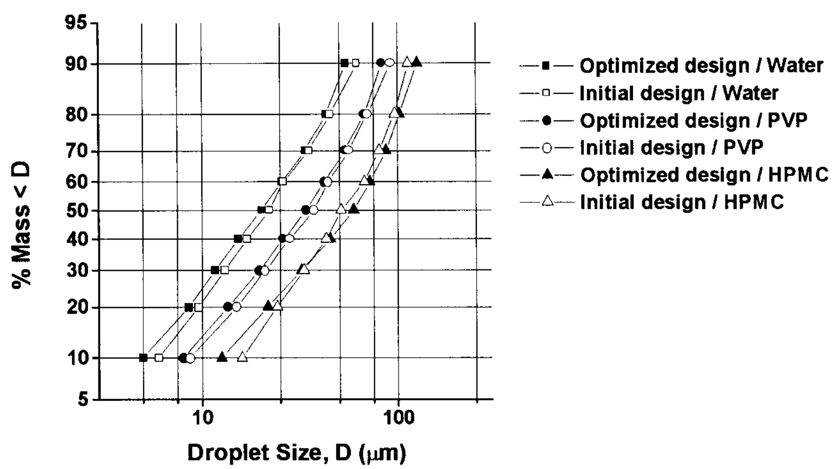

Figure 5. Droplet size distributions of sprays in log-probability scale from atomizers of the Initial (I) and optimized (O) atomizer design. Exit orifice (I) 0.6 mm, (O) 0.5 mm; exit orifice length-to-diameter ratio (I) 2.5, (O) 2.5; air-injector distance to orifice-exit (I) 5 mm, (O) 3.5 mm; air-injector (I) two outlets of 0.25 mm^2 (0° parallel to the bulk flow) and 0.14 mm^2 (45° to the bulk flow), (O) a helical-shaped air injector with one outlet of 0.13 mm^2 (0° parallel to the bulk flow); mixing chamber (I) 4.6 mm, (O) 3.0 mm. Air-to-liquid mass ratio: 0.3; liquids: Water, 10% Kollidon K-30 and 10% Pharmacoat 603; liquid flow rate: approximately 35 g/min.

Table 3. Influence of air injector design and distance between air injector and exit orifice on droplet size.

	Distance between air injector and exit orifice/D_{32}					
Distance/mm	8.5	7.0	5.0	3.5	2.5	1.5
$D_{32} \pm$ STDEV/μm	11.5±0.3	11.1±0.4	11.0±0.6	10.2±0.0	10.4±0.1	10.6±0.3

Data for a water spray at 35 g/min, air-to-liquid mass ratio of 0.3.

directions and the motion direction of the bulk liquid flow. The former was either zero (gas injection parallel to the bulk flow) or 45 degrees. The latter was changed from axial to swirling by inserting a helical air injector. Figure 3 shows that droplet size decreases with an increase in ALMR and that air injector design does influence droplet size. In particular, smaller droplet sizes are obtained with a parallel air outlet into the mixing chamber. Furthermore, the data demonstrate that a helical injector design (swirling liquid flow) leads to smaller droplet sizes at ALMRs of 0.1 when compared with a smooth-sided injector design (axial liquid flow). At ALMRs of 0.3 and higher no effects of liquid flow direction are observed, probably because the increased airflow leads to a more turbulent flow within the mixing chamber, which diminishes the effect of the liquid swirl.

The results shown in Fig. 3 indicate that a centred air flow in a swirled liquid flow leads to the most homogeneous air-liquid distribution in the mixing chamber. This homogeneous mixture of air and liquid leads to a smaller mean droplet size at all ALMRs tested, with its largest effect at the lowest ALMRs. Similar results were obtained for polymer solutions of PVP and HPMC.

Air Injector and Exit Orifice Distance

In order to determine the optimal distance between the air injector and the exit orifice, mean droplet size of a water spray was measured for different atomizer designs with air injector—exit orifice distances of 1.5, 2.5, 3.5, 5.0, 7.0, and 8.5 mm. Distances smaller than 1.5 mm lead to blocking of the flow through the exit orifice. The results showed that the smallest droplet sizes were obtained at a distance of 3.5 mm between the air injector and the exit orifice, Table 3. The effect of distance on droplet size, however, is seen to be rather small.

Diameter of Mixing Chamber

A reduction in the diameter of the mixing chamber combined with a swirling liquid flow was expected to improve atomization quality due to better mixing of water and air in the mixing chamber. Results were obtained for water with mixing chamber diameters of 3.0, 3.5, and 4.6 mm at a fixed exit orifice diameter of 0.75 mm. They showed no clear effect of mixing chamber diameter on droplet size. This leads to the conclusion that mean droplet size is independent of mixing chamber diameter within the range of diameters investigated in this study.

Performance of Atomizer

Figure 4 shows mean droplet sizes from sprays of water, 10% Kollidon K-30, and 10% Pharmacoat 606 for the standard atomizer design by Petersen et al.[1] and the optimized atomizer design. The optimized atomizer design leads to smaller droplet sizes at ALMRs of 0.1. Figure 4 illustrates the importance of an optimized atomizer design when trying to attain small droplet sizes at low ALMRs. For ALMRs above 0.3 only a minor (or no) effect is seen on the droplet size within the atomizer designs investigated in this study.

Figure 5 shows the droplet distribution from sprays of water, 10% Kollidon K-30, and 10% Pharmacoat 603 for the standard atomizer design by Petersen et al.[1] and the optimized atomizer design. Both atomizer designs lead to droplet size distributions in reasonable agreement with the log-normal distribution for liquids of low viscosity. As seen form Fig. 5, higher viscosity causes derivation from the log-normal distribution. The droplet size distributions of effervescent atomizers are equally uniform to the droplet size distributions of conventional pneumatic atomizers.

CONCLUSION

The results indicate that it is very likely that secondary atomization is the dominant mechanism of droplet formation. This is supported by the reduced influences of geometric factors as ALMR goes up, and by the reduced, but not absent, influences of fluid physical properties as ALMR increases.

In order to design an effervescent atomizer that produces small droplets is it important to consider the following design elements of the atomizer: A smaller atomizer exit orifice diameter yields smaller droplets. A minimum exit orifice diameter should be chosen based on the maximum mass flow rate desired and on plugging considerations. The injector's outlet holes should provide axial gas injection (parallel to the atomizer axis) into the mixing chamber, and the liquid flow should be swirling. It is also shown that there is a minor influence of air injector to exit orifice distance on mean droplet size. No clear effect on atomizer performance is seen for neither the length/diameter ratio of the exit orifice, the area of the air injecting holes, nor the diameter of the mixing chamber within the ranges of dimensions tested in this study.

The results presented in this work cover parameters to take into account when designing an effervescent atomizer for use in a spray process such as closed-cycle spray drying, where the air or gas mass flow should be minimized due to the process and product properties.

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