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The influence of liquid spray rate and atomizing pressure on the size of spray droplets and spheroids

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Abstract

The effects of liquid spray rate and atomizing pressure on the size of spray droplets and spheroids were investigated in this study. The range of droplet sizes tended to narrow and relatively smaller droplets were formed at higher atomizing pressures. When liquid spray rate was increased, the droplets were larger and had a wider size range. Although alterations in liquid spray rate could affect spray droplet size, greater changes in droplet size were achieved by varying atomizing pressure. In these investigations, changes in spray droplet size and atomizing pressure did not appear to greatly affect mean spheroid size. However, the presence of an atomizing pressure was necessary for the even distribution of the moistening liquid to reduce localized overwetting during spheronization by rotary processing. The amount of oversized particles may thus be decreased and a more uniformly sized yield may be achieved with the use of relatively higher atomizing pressures. With a fixed volume of moistening liquid sufficient for spheronization, larger spheroids were produced with increasing liquid spray rates. The increased wetting per unit time resulted in the formation of larger nuclei and enhanced the growth rate of spheroids.

Keywords: Liquid spray rate; Atomizing pressure; Spray droplet size; Spheroid size; Spheroid

1. Introduction

The formation of spheroids by agglomeration-spheronization consists basically of three stages: liquid addition, massing and drying. These stages are carried out in a single continuous run. From our previous work, it was found that the amount of moisture actually present in the powder mass during the formative stage established spheroid structure and determined spheroid size (Wan et al., 1994). This amount of moisture was dependent on the volume of moistening liquid delivered to the powder mixture and its rate of delivery. Many studies have shown that the amount of moistening liquid was an important parameter for controlling spheroid size (Miyake et al., 1973; Bains et al., 1991; Elbers et al., 1992; Pinto et al., 1992). The effect of liquid spray rate on spheroid size has not been as widely investigated as most of the spheronization studies were on the extrusion-spheronization procedure. However, the influence of flow rate on granule size in fluidized bed granulators and high shear mixers has been examined by several workers (Schaefer and Worts, 1978a; Holm et al., 1983, 1984; Niskanen et al.,

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1990; Watano et al., 1994). Their studies often showed contradictory results. In some of these cases, granule size increased with higher spray rates while a decrease or only a slight change in granule size was observed in other situations. The conflicting conclusions may be attributed to the different ways in which the studies were carried out. For example, spray droplet size may be kept constant while increasing liquid flow rate or the moisture content of granules was fixed when spray rate and droplet size were increased. Besides that, the influence of liquid loss through evaporation and air exchange could also affect the actual moisture content of the product. For our present study, a fixed volume of moistening liquid, which was found to be adequate for spheroid formation, was delivered to the powder mixture while atomizing pressure and spray rate were varied. Variations in the droplet size of the moistening liquid may be induced by altering spray rate and atomizing pressure. As such, their effects on spray droplet size were determined before proceeding to an investigation on the influences of atomizing pressure, liquid spray rate and spray droplet size on the size and size distribution of spheroids produced in a rotoprocessor.

2. Materials and methods

2.1. Materials

Lactose monohydrate (Pharmatose 200M, De Melkindustrie Veghel, The Netherlands) and microcrystalline cellulose (MCC; Avicel PH-101, Asahi Chemical, Japan) were used as supplied. Distilled water was used as the moistening liquid.

2.2. Methods

2.2.1. Determination of spray droplet size

The droplet size of the moistening liquid was measured using a laser diffraction droplet and particle sizer (Malvern Instruments, Malvern 2600, UK). The two-fluid one-head spray nozzle had a liquid entrant diameter of 1 mm and the air cap position was set at 1. The nozzle was positioned 10 cm away from the lens (focal length of 100 mm) and 10 cm away from the path of the laser beam which passed through the centre of the lens. When a measurement was to be made, an open spray was directed perpendicularly across the path of the laser beam. Three replicates were carried out for a particular atomizing pressure and spray rate. This procedure was repeated with varying atomizing pressures and liquid flow rates. The measurements of the droplet diameters at the 10, 50 and 90 percentiles on cumulative percent oversize plots (by volume) were plotted in a three-dimensional graphic form.

2.2.2. Preparation of spheroids

MCC and lactose (1:3) were pre-mixed in a laboratory double cone mixer (J. Engelsmann AG, JEL, Germany) for 30 min. 500 g of the powder mixture was transferred to a rotoprocessor (Niro Aeromatic, MP-1, Switzerland). The 30 cm diameter frictional plate had a cross-hatch design of square studs with rounded edges (diameter 9 mm, height 2 mm) arranged in a 10 mm square grid pattern at alternate intersections. The upper wall of the spheronizer chamber was lined with polytetrafluoroethylene (PTFE) tape to reduce material deposition on the wall. 200 ml of moistening liquid was used. The gap air pressure was set at 0.8 bar and the inlet temperature at 30°C. A peristaltic pump (Watson Marlow 503S1, UK) was used to deliver the moistening liquid to the powder mixture. The spray rate of the moistening liquid was varied between 14 and 38 ml/min. The atomizing pressures used ranged from 0 to 1.2 bar. The speed of the frictional plate was set at 700 rpm (terminal velocity = 659.7 m/min) during the liquid addition phase. After liquid addition, the rotational speed was reduced to 480 rpm (terminal velocity = 452.4 m/min). The frictional plate was rotated for 20 min before drying was carried out. The inlet temperature was set at 60°C and the wall of the inner chamber was lifted for the fluidization and drying of spheroids.

2.2.3. Size analysis of lumps and spheroids

The total yield of dried spheroids were sieved through sieves of 3.35, 2.80 and 2.00 mm aperture sizes. The amount retained on these sieves represented the amount of agglomerates of diameter greater than 2.00 mm. For this study, these agglomerates of greater than 2 mm in size were defined as lumps.

The lengths of 50 of the largest oversized particles were measured using a micrometer screw gauge (Mitutoyo 7305, Japan) to determine their average length.

The total yield of spheroids were divided into randomly sampled portions. A nest of sieves (Endecotts test sieves, UK) was used to separate a weighed amount of spheroids into various size fractions. Mass median diameter was the spheroid diameter at the 50 percentile mark on a cumulative percent oversize plot. The spheroid diameters at the 10 and 90 percentile marks on the size distribution curves were also noted. These results were presented as three-dimensional plots.

2.2.4. Moisture content determination

Spheronization runs were conducted with varying liquid spray rates at a fixed atomizing pressure of 0.8 bar. Spheroids collected after the liquid addition stage were weighed before drying was carried out at 115°C for 4 h in an oven. After drying, the samples were cooled to room temperature in a vacuum chamber. The weights of the dried samples were noted. The difference in the initial and final weights of a sample represented the amount of moisture lost. The moisture content is the quotient, expressed as a percentage, of the amount of moisture lost and the weight of the dried sample. It was necessary to correct the moisture content attributed to the moistening liquid by subtracting the basal moisture content. The powder mixture used for spheronization was found to contain 1.34% w/w moisture.

3. Results and discussion

3.1. Effect of atomizing pressure and liquid spray rate on the droplet size of the moistening liquid

The droplet size of the moistening liquid may be altered by varying atomizing pressure and/or liquid spray rate. Thus, it was possible that spray droplet size may indirectly affect granule size as observed in some studies on fluidized bed granulations where increasing atomizing pressure often resulted in decreasing spray droplet size as well as granule size (Schaefer and Worts, 1978b,c; Merkku and Yliruusi, 1993; Merkku et al., 1993). The estimation of spray droplet size has been discussed previously by Schaefer and Worts (1977) in their fluidized bed granulation studies. They mentioned that few investigations on the droplet size of binder solutions had been conducted due to the lack of accurate and efficient methods for droplet size analysis. Recently, the advent of laser diffractometry presented a relatively simple, efficient and less time-consuming method for measuring droplet size. An open spray was used in our measurements as attempts to decrease obscuration values by shielding often altered the spray pattern. A three-dimensional plot was used to depict the relationship between atomizing pressure and liquid spray rate on the spray droplet size (Fig. 1). At a constant spray rate, the median diameter of the spray droplets decreased when the atomizing pressure was increased. This observation was linked to an increase in the air-to-liquid mass ratio with greater atomizing pressures (Schaefer and Worts, 1978c). A greater air-to-liquid mass ratio increased the efficiency of the spray nozzle for dispersing the moistening liquid. When the liquid spray rate was increased whilst maintaining a constant atomizing pressure, larger spray droplets were obtained. The range of droplet sizes widened at higher spray rates and at lower atomizing pressures as indicated by the larger differences in droplet sizes at the 10 and 90 percentile marks. There was a greater change in droplet size with increasing spray rates at a low atomizing pressure. The differences in droplet sizes became less obvious at high atomizing pressures. From these results, it was noted that a greater change in droplet size may be obtained by varying atomizing pressure at a constant spray rate rather than by changing spray rates while maintaining a fixed atomizing pressure.

3.2. Effect of atomizing pressure and liquid spray rate on spheroid size and size distribution

During the liquid addition phase, the absence of an atomizing pressure resulted in overwetting near the nozzle. The uneven distribution of water gave rise to stagnation and build-up of moist powder mass. This hampered the formation and rolling motion of spheroids. Besides that, lumps may be formed from pieces which may break off from the overwetted mass. Consequently, the end-products would have a wide size distribution. An atomizing pressure was thus essential for dispersing the moistening liquid into fine droplets to improve liquid distribution (Holm et al., 1983, 1984). This would reduce the occurrence of overwetting especially around the nozzle area.

From this study, it was observed that there was a suitable range of atomizing pressures for successful spheroid production. The pressure must be strong enough to disperse the moistening liquid but not so strong as to cause a substantial loss of dry powder from the spheronizer chamber at the start of the liquid addition phase. For the particular load used in these experiments, the atomizing pressure was not increased beyond 1.2 bar. This was because even at 1.2 bar, some of the powder mixture tended to be blown out of the chamber during the initial stage of liquid addition before the powder was adequately moistened. With higher air pressures, there may be a significant loss of powder resulting in a reduction of the spheronization load and the end-products may be less predictable.

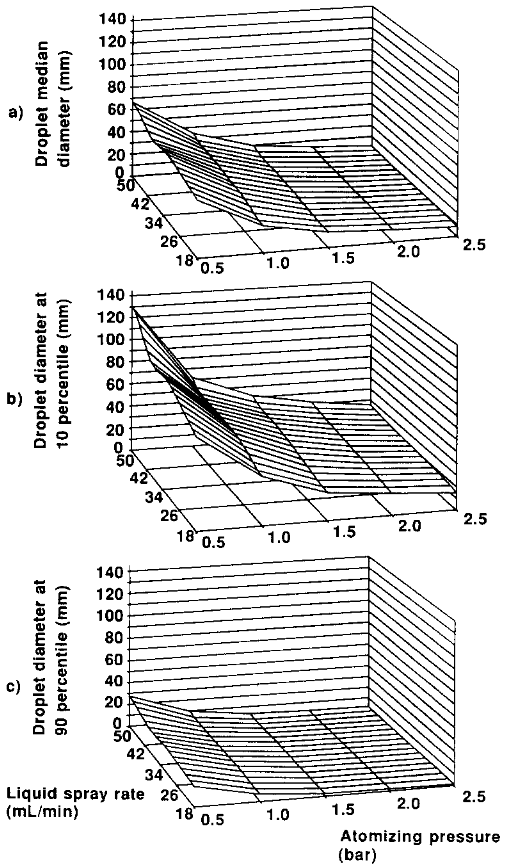

Fig. 1. Effect of atomizing pressure and liquid spray rate on the droplet size of the moistening liquid.

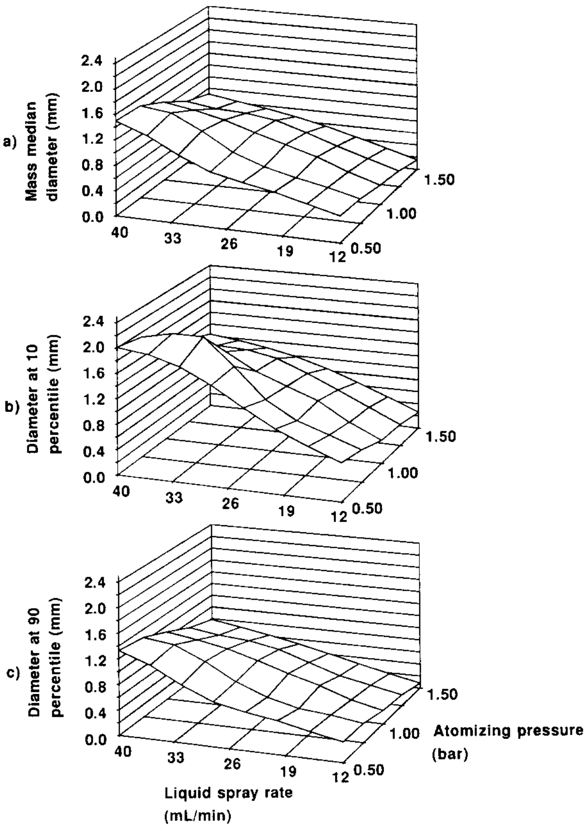

Fig. 2. Influence of atomizing pressure and liquid spray rate on spheroid size and size distribution.

A three-dimensional plot may be used to aid the analysis of the influences of atomizing pressure and liquid spray rate on the size of spheroids (Fig. 2). The spheroid mass median diameter seemed to show only slight variations in size with alterations in atomizing pressure. However, it was

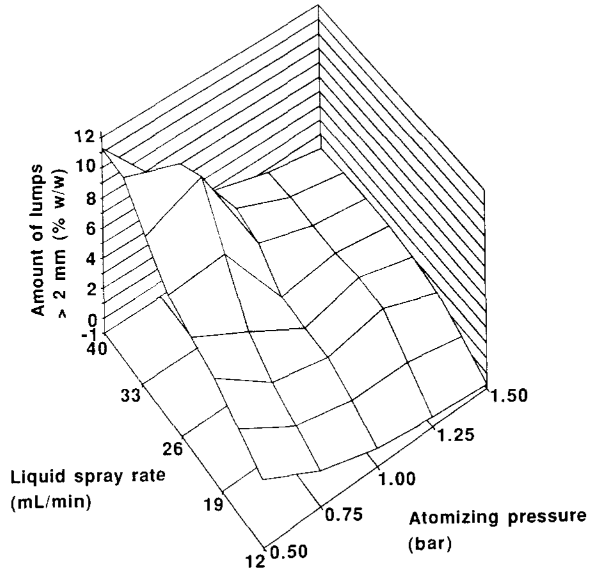

Fig. 3. Effect of atomizing pressure and liquid spray rate on the amount of agglomerates greater than 2 mm in size.

observed that oversized particles were more predominant in the absence of an atomizing pressure and at a very low air pressure. The amounts of these lumps seemed to diminish with higher atomizing pressures (Fig. 3). This was attributed to the greater air pressures associated with high atomizing pressure values which helped to improve the distribution of the moistening liquid and the dispersion and circulation of the powder mass. The decrease in the amount of lumps resulted in the formation of spheroids which were more uniform in size.

In fluidized bed granulations, granule growth generally proceeded via a nucleative process. Upon wetting, the primary powder particles formed nuclei held together by liquid bonds. The formation of these nuclei may be influenced by the size of the spray droplets. Bigger droplets were able to form larger nuclei as they could bind more primary particles together (Schaefer and Worts, 1978c). However, the size of the spray droplets did not seem to have a great effect on spheroid size. This was probably due to the different experimental conditions and growth mechanisms involved. The centrifugal forces set up by the rotating frictional plate assisted in the circulation of the powder mixture and in the coalescence, agglomeration and densification processes during agglomeration-spheronization. These forces were less intense than those generated in high shear mixers but were probably stronger than the forces experienced by the suspended particles in fluidized bed granulators. The rotational speed of the frictional plate also played a role in controlling spheroid size as the strength of the centrifugal forces may be altered by varying the rotational speed. Therefore, the contributions which spray droplet size may have had on the size of spheroids could have been overshadowed by the prevalent centrifugal forces.

As spray rate was increased, spheroid size was observed to increase (r^2 values = 0.93 to 0.99) for the range of spray rates studied whilst the atomizing pressure and the amount of moistening liquid delivered were kept constant. Since changes in droplet size caused by varying atomizing pressure did not greatly affect spheroid size, spheroid growth with higher spray rates was not entirely due to the effect of increasing droplet size. As the powder mass was wetted, liquid bonds began to form between the primary powder particles. Whilst rotating on the frictional plate, the moistened primary particles formed nuclei. The size of the nuclei would depend on the amount of wetting and the cohesiveness and strength of the powder mass. Generally, greater cohesiveness and strength were required for forming larger nuclei. With higher spray rates, the increase in the amount of moistening liquid delivered per unit time led to the induction of more liquid bonds between the powder particles. The cohesiveness of the powder mass was augmented and larger nuclei may be formed. Besides that, the greater wetting also enhanced granule surface plasticity. This heightened the chances for coalescence and agglomeration to occur and increased the growth rate of spheroids. Therefore, bigger particles were observed with greater spray rates after the liquid addition stage (Fig. 4). The time for liquid addition was also shortened when the moistening liquid was delivered to the powder mixture at a faster rate. Accordingly, the loss of moisture due to evaporation may be reduced with higher spray rates. With a fixed volume of water, faster spray rates resulted in spheroids retaining larger amounts of moisture after liquid addition (Fig. 5). As such, higher granule moisture contents after liquid addition apparently also contributed to the production of larger spheroids.

After liquid addition, some of the moistened mass tended to form a stationary layer on the lower wall of the spheronizer chamber which was not lined with PTFE tape. This was observed when the process was stopped immediately after liquid addition for the collection of spheroids for moisture content determination. Such caking could give rise to the formation of some oversized particles. The average length of 50 of the largest particles in a batch ranged from 5 to 9 cm. From these results, there did not seem to be a general trend in their average length with changes in liquid spray rate and atomizing pressure. However, from a three-dimensional plot of the amount of agglomerates with diameters greater than 2 mm vs both atomizing pressure and liquid spray rate, it was noted that the largest quantity of such lumps were obtained with high spray rates at low atomizing pressures (Fig. 3). Visual observation indicated that relatively small spheroids with some oversized particles were obtained at low spray rates. These lumps may be attributed to pieces detached from the caked mass. When a fixed volume of water was delivered, an augmentation in the spray rate led to an increase in the extent of coalescence and agglomeration. The amount of large particles increased in line with an increase in spray rate. However, these lumps became less obvious as the overall spheroid size was likewise increased. Larger granular or flat pieces also gave way to relatively rounder agglomerates at high liquid flow rates as the roundening effect was enhanced by an augmentation in granule surface plasticity.

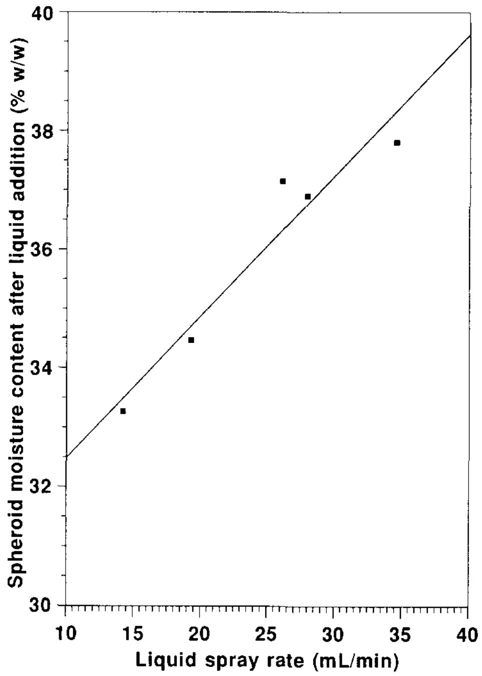

Fig. 5. Effect of liquid spray rate on the moisture content of spheroids immediately after liquid addition.

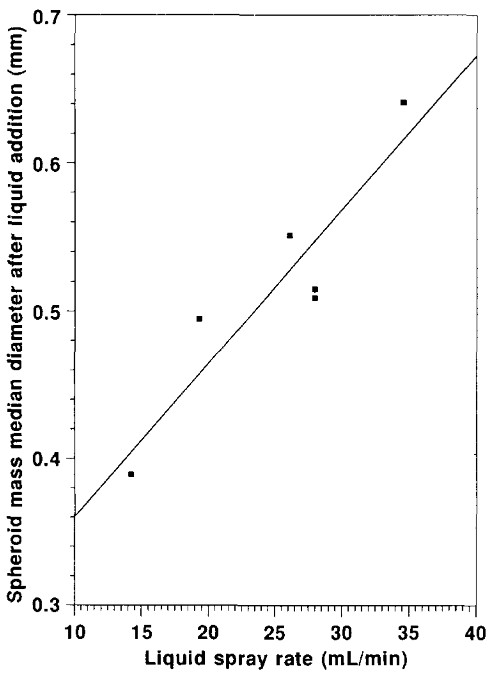

Fig. 4. Mass median diameters of spheroids obtained immediately after liquid addition with varying liquid spray rates.

From these observations, it was inferred that, with a suitable amount of moistening liquid for spheronization, the delivery rate of the moistening liquid was an important variable for determining the size of spheroids produced in a rotoprocessor. The main role of an atomizing pressure was essentially that of providing an even distribution of the moistening liquid to the powder mix. With improved liquid distribution, localized overwetting was reduced. For that reason, while there was only slight changes in the median spheroid size, the amount of lumps may be decreased by using a higher atomizing pressure. Although, the droplet size of the moistening liquid may be altered by varying atomizing pressure and spray rate, spray droplet size did not seem to have a great effect on the median size of spheroids.

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