Department of Pharmaceutical Technology, Charles University in Prague, Faculty of Pharmacy in Hradec Králové, Czech Republic

The influence of orifice height on flow rate of powder excipients

Z. Zatloukal, Z. Šklubalová

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Zdenka Šklubalová, Ph.D., Charles University in Prague, Faculty of Pharmacy, Heyrovského 1203, 500 05 Hradec Králové, Czech Republic
zdenka.sklubalova@faf.cuni.cz

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The influence of the orifice height of a cylindrical, flat-bottomed hopper on the mass flow rate of the free-flowable size fractions of sodium chloride and boric acid was investigated. It was observed that a zone of sudden acceleration of the mass flow under gravity occurred when a critical orifice height had been achieved. Based on the results, an orifice diameter equal to 12 mm with a height of between 8–16 mm is recommended for the faster flow of sodium chloride while an orifice diameter equal to 8 mm with a height of less than 8 mm is appropriate for the slower flow of boric acid. In summary, the orifice height should be taken into consideration as an important parameter of a cylindrical test hopper in order to obtain a reproducible and comparable mass flow as the single-point characteristic of powder flowability.

1. Introduction

There are two basic aspects in powder handling, powder flowability and powder compressibility. Powder flow behavior is multifaceted and, therefore, it can never be characterized with only one method (Zhou and Qiu 2010). Monitoring of the powder flow rate through a hopper orifice has been proposed as the best measure of flowability of free-flowable powders; a cylindrical hopper with a flat bottom is recommended. To obtain an uniform, well reproducible mass flow rate, hopper geometry must follow general guidelines (Nedderman et al. 1982; Xie and Puri 2006): The inner diameter of the circular hopper D is greater than 2.5 times the circular orifice diameter D_0; the diameter of the opening D_0 is greater than 6 times the diameter of the particle d. For orifices that are less than six particle diameters, the flow is intermittent and irreproducible. Brown and Richards (1960) also recommended that the difference (D-D_0) should be greater than 30 times the particle diameter d. In accordance with the principle of the hour-glass theory, the flow rate is independent of the height of a powder column h if the head is greater than twice the diameter of the hopper orifice D_0. If it is less, the flow rate increases (Tighe and Sperl 2007).

In this experimental work, the influence of the orifice geometry on the mass flow rate of four size fractions of sodium chloride and boric acid through a cylindrical, flat-bottomed hopper was investigated. The optimization of the orifice height to achieve the best reproducibility in a single-point powder flowability test was the aim of the study.

2. Investigations, results and discussion

Determination of flow rate through an orifice is useful with free-flowing materials. The flow rate is generally measured as a mass flow rate of powder flowing from a container. Because the size and shape of the hopper used and its orifice are critical, the hopper geometry must be well-defined (Nedderman et al. 1982; Xie and Puri 2006). In this work, four size fractions ranging from 250–630 μm of two model free-flowable pharmaceutical powder excipients were studied. The geometry of the testing hopper followed the general recommendations $D/2.5 > D_0 > 6\ d$ mentioned above where D, D_0, and d are as described.

Continuous measurement of the mass flow rates Q (g/s) using an electronic balance showed the steady-state flow of powders from the model hopper in a range of 10–90% of the powder bed. At the beginning, the funnel-like flow is inappropriate to characterize the powder flow behavior (Prescott and Barnum 2000) as well as the sliding over the remaining powder in a hopper at the end of a discharging process. On the other hand, the mass of the remaining powder in a cylindrical hopper could be used to estimate the drained angle as the specific flow characteristic as previously referred to by Šklubalová and Zatloukal (2009).

The mass flow rates Q of sodium chloride for a given orifice height H and a given orifice diameter D_0 are summarized in Table 1. Data are completed with the values of relative deviations (%) in brackets which were calculated as follows: the arithmetic mean of the mass flow rate was computed for each orifice diameter regardless of the orifice height ($N = 4$). Then, the percentage relative deviations of Q from the average for each orifice height $H = 2$, 4, 8, and 16 mm were expressed. The sum of the deviations is equal to zero. The plus or minus sign of each value indicates the faster and/or slower actual mass flow rate in comparison to the average one. In the lower part, Table 1 is completed with the average flow rates calculated for the sodium chloride regardless of the size fractions. The principal objective of this data presentation is to evaluate the effect of the orifice height on the powder flow rate. In Table 1, thus, the pairs of the orifice heights within which the acceleration of the mass flow rate was greater than 5% are shown in bold italic script. Similarly, the results for boric acid are summarized in Table 2.

The profile of powder discharge in a cylindrical hopper is the combination of funnel flow and mass flow. Although the effect of the orifice height is generally assumed to be non-significant,

Table 1: Influence of the orifice geometry on the mass flow rate of the size fractions of sodium chloride

Size fraction (μm)	Orifice height (mm)	Mass flow rate (g/s) through the orifice diameter		
		8 mm	10 mm	12 mm
250–315	2	11.5 (−3.2%)	20.8 (−3.4%)	34.1 (−5.3%)
	4	11.5 (−3.2%)	20.2 (−6.2%)	35.8 (−0.6%)
	8	12.4 (4.4%)	22.5 (4.5%)	37.0 (2.8%)
	16	12.1 (1.9%)	22.6 (5.0%)	37.1 (3.1%)
315–400	2	10.9 (−4.2%)	20.3 (−3.3%)	34.1 (−3.7%)
	4	10.9 (−4.2%)	19.7 (−6.2%)	34.9 (−1.5%)
	8	11.9 (4.6%)	22.0 (4.8%)	35.8 (1.1%)
	16	11.8 (3.7%)	22.0 (4.8%)	36.9 (4.2%)
400–500	2	10.5 (−3.7%)	19.5 (−3.1%)	32.7 (−4.7%)
	4	10.5 (−3.7%)	18.9 (−6.1%)	34.2 (−0.4%)
	8	11.4 (4.6%)	21.1 (4.8%)	34.9 (1.7%)
	16	11.2 (2.8%)	21.0 (4.3%)	35.5 (3.4%)
500–630	2	9.97 (−3.6%)	18.5 (−3.1%)	30.5 (−6.8%)
	4	9.90 (−4.3%)	17.8 (−6.8%)	32.7 (0%)
	8	10.9 (5.4%)	20.1 (5.2%)	33.8 (3.3%)
	16	10.6 (2.5%)	20.0 (4.7%)	33.9 (3.6%)
Average	2	10.72 (−3.6%)	19.78 (−3.2%)	***32.85(−5.1%)***
	4	***10.70 (−3.8%)***	***19.15 (−6.3%)***	***34.40 (−0.6%)***
	8	***11.65 (4.7%)***	***21.43 (4.8%)***	35.38 (2.2%)
	16	11.43 (2.7%)	21.40 (4.7%)	35.85 (3.6%)

indeed, it plays an important role in the direction of the discharging particles. The centripetal movement of the powder at the powder head is directed to mass flow under gravity upon approaching a hopper outlet. The centrifugal tendency of powder particles for an orifice of lower height can be corrected by an increase in the orifice height. This is associated with the acceleration of the flow (Datta et al. 2008). At a certain orifice height, however, an unexpected acceleration of flow could lead to serious flow failure. By a further increase in the outlet height the wall friction becomes dominant following with a reduction of particle velocity. Longer orifices, therefore, would not generally be recommended.

Reliable detection of the flow failure zone might be difficult but it is important regarding the correct testing of flowability. The arrangement of results in Tables 1 and 2 allows the examination of how the mass flow rate is affected due to changes in the orifice height. A non-linear relationship can be observed. With the orifice diameter $D_0 = 12$ mm, a zone of significant flow acceleration was noted for the orifice heights between 2–4 mm for both studied powders. Similarity was concluded for $D_0 = 10$ mm when H increased from 4 to 8 mm. With the orifice diameter $D_0 = 8$ mm, a zone of the accelerated flow rate was detected for a range of H 4–8 mm for sodium chloride while for a range of $8 < H < 16$ mm for boric acid. The difference between the risk zones noted for

Table 2: Influence of the orifice geometry on the mass flow rate of the size fractions of boric acid

Size fraction (μm)	Orifice height (mm)	Mass flow rate (g/s) through the orifice diameter		
		8 mm	10 mm	12 mm
250–315	2	6.14 (−1.7%)	11.6 (−1.3%)	18.4 (−5.8%)
	4	6.11 (−2.2%)	11.0 (−6.4%)	19.5 (−0.1%)
	8	6.14 (−1.7%)	12.3 (4.7%)	19.8 (1.4%)
	16	6.59 (5.5%)	12.1 (3.0%)	20.4 (4.5%)
315–400	2	5.95 (−1.2%)	11.5 (−1.1%)	18.2 (−5.0%)
	4	5.93 (−1.6%)	10.8 (−7.1%)	19.1 (−0.3%)
	8	5.86 (−2.7%)	12.1 (4.1%)	19.5 (1.8%)
	16	6.36 (5.6%)	12.1 (4.1%)	19.8 (3.4%)
400–500	2	5.57 (−1.5%)	10.9 (−0.9%)	17.7 (−4.7%)
	4	5.55 (−1.8%)	10.2 (−7.3%)	18.3 (−1.5%)
	8	5.51 (−2.5%)	11.5 (4.5%)	18.9 (1.8%)
	16	5.98 (5.8%)	11.4 (3.6%)	19.4 (4.4%)
500–630	2	5.05 (−1.5%)	9.94 (−2.5%)	16.3 (−5.4%)
	4	5.01 (−2.3%)	9.42 (−7.6%)	17.1 (0.7%)
	8	4.96 (−3.3%)	10.8 (6.0%)	17.5 (1.6%)
	16	5.49 (7.1%)	10.6 (4.0%)	18.0 (4.5%)
Average	2	5.68 (−1.5%)	10.99 (−1.4%)	***17.65 (−5.2%)***
	4	5.65 (−2.0%)	***10.36 (−7.0%)***	***18.50 (−0.6%)***
	8	***5.62 (−2.5%)***	***11.68 (4.8%)***	18.93 (1.7%)
	16	***6.11 (6.0%)***	11.55 (3.6%)	19.40 (4.2%)

both studied powders resulted from the differences in the actual mass flow rates. The isometric particles of sodium chloride of higher density flow faster than anisometric particles of boric acid of lower density. Moreover, particles of boric acid show slight electrostatic charge when discharged due to particle friction. The mass flow of sodium chloride can thus be accelerated more easily with a shorter orifice height while a longer orifice height is required for boric acid.

Based on the results, the following conclusions can be drawn from our experimental arrangement, with subsequent recommendations for different orifice diameters D_0. To optimize flowability testing, an orifice diameter of $D_0 = 12$ mm and an optimum H in a range of 8–16 mm can be recommended for sodium chloride with the flow rates near the maximum of quadratic polynomial function. An orifice diameter of $D_0 = 8$ mm with an optimum orifice height $H < 8$ mm would be recommended for boric acid with the flow rates near the minimum of quadratic polynomial function.

In conclusion, the significant influence of orifice height on powder mass flow rate has been detected experimentally. When a certain critical height is achieved, the flow suddenly accelerates, but a further increase in the orifice height does not produce an increase in the flow rate in any linear way. To obtain a reproducible flow, the zone of risk flow acceleration should be avoided. For better comparison of the experimental results, therefore, information regarding the orifice height used should always be referred to in pharmaceutical technology.

3. Experimental

3.1. Materials

Two different powder excipients of pharmaceutical quality were used: sodium chloride (true density of 2.17 g/cm^3) and boric acid (true density of 1.48 g/cm^3). Size fractions of 250–315, 315–400, 400–500, and 500–630 μm were obtained using a vibrating screen Pulverisette® 0 (Alfred Fritsch, Laborgerätebau, Idar Oberstein, Germany).

3.2. Methods

The measurements were performed at a controlled ambient temperature of 25 ± 1 °C and relative air humidity of 48 ± 3%. The testing apparatus consisted of a model stainless steel, cylindrical hopper, having an inner diameter D of 40 mm, and a stainless steel flat bottom with a concentric circular orifice. The bottom was fixed to the cylinder by a bronze screw-thread. Twelve experimental combinations were assembled using changeable bottom with an orifice diameter D_0 of 8, 10, and 12 mm, each with a height diameter $H = 2$, 4, 8, and 16 mm, respectively.

The uniform mass discharge rate Q (g/s) was measured such that the orifice was opened and the time to free steady-state discharge of a minimal amount of 100 g of powder from the hopper was measured. Each size fraction was characterized with an average of ten measurements with a relative standard deviation of less than 0.5%.

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