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The relationship between drained angle and flow rate of size fractions of powder excipients

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Received May 25, 2009, accepted June 12, 2009

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Pharmazie 64: 846–847 (2009)
doi: 10.1691/ph.2009.9170

The influence of powder size of chosen pharmaceutical powder excipients on drained angle as well as the correlation between drained angle and the mass flow rate of certain powder size fractions were investigated in this work. A method of the indirect estimation of the three-dimensional drained angle from the mass of the residual powder was used experimentally to study the influence of powder size fractions in range of 0.200–0.630 mm for sodium chloride, sodium citrate, potassium chloride, and potassium citrate. Failures of flow significantly increased the drained angles for powder size fraction of 0.200–0.250 mm. For the uniformly flowable powder size fraction of 0.400–0.500 mm, the faster the flow rate, the smaller drained angles were observed for excipients investigated. To estimate parameters of the flow equation, the measurement of material flow rates from the hopper of different orifice sizes is needed, while the estimation of drained angle is much easier needing only one hopper. Finally, the increase of the hopper wall angle of the standard conical hopper to 70° could be recommended to achieve uniform mass flow and to reduce the adverse effect of powder gliding along the hopper walls.

Testing of powder flow rate through an orifice of a cylindrical hopper employs the powder mass flow which subsequently changes into the funnel flow. In the hopper, the residual powder remains at the end of a discharging process, the weight of which correlates with the powder mass flow rate. Both the mass flow rate and the mass of the residual powder are significantly influenced by the hopper orifice diameter in contrast to the drained angle of the residual powder. The angle of repose, compressibility index and the Hausner ratio are conventionally used to characterize powder flow (USP 30/NF 29, <1174> Powder flow (harmonization)). According to the assessment of the angle of repose in range of 25–65°, the general scale is used in the description of powder flow properties from excellent to very poor (Carr 1965). The angle of repose is defined as the constant, three-dimensional angle to the horizontal surface, which, assuming a cone-like pile of material, is formed when the powder passes through a funnel-like container. Disadvantages of the angle of repose include segregation, consolidation or aeration of material as the cone forms following limited reproducibility of measurement (Rios 2006).

The drained angle expresses the angle of the residual bulk solid in the cylindrical hopper after its self-draining. The use of two-dimensional translucent boxy hopper separated by a dividing plate with a square opening in order to estimate the three-dimensional drained angle (Li et al. 2004) did not represent the suitable model hopper (Zhu and Yu 2005). The method in which the 3-D drained angle was estimated indirectly using the mass of the residual powder of known bulk density, remaining in a cylindrical flat-bottomed hopper after its self-discharging at gravity conditions, was proposed by Zatloukal and Šklubalová (2008):

$$\alpha = \arctan\left[\frac{24 \cdot \mathrm{M}}{\pi(2\mathrm{D}^2 - \mathrm{d}(\mathrm{D}+\mathrm{d}))(\mathrm{D}-\mathrm{d}) \cdot \mathrm{d_b}}\right] \qquad (1)$$

where α is drained angle (°), M is the mass of the residual powder at the end of the self-draining process (g), D is the inner diameter of the cylindrical hopper (cm), d is the diameter of the circular orifice in its bottom (cm), and d_b the powder bulk density (g/cm^3).

The influence of powder particle size on the drained angle was studied for the size fractions in range of 0.200–0.630 mm. In Fig. 1, the influence of the geometrical mean of size fractions (i.e. 0.224, 0.281, 0.355, 0.447, and 0.561 mm, respectively) on drained angle estimated using the cylindrical test hopper having the orifice diameter equal to 10 mm is illustrated for sodium chloride and sodium citrate. For smaller particles, the smaller drained angle was observed for both powder excipients. This is in accordance with the increase in the mass flow rate and the decrease in the mass of the residual powder remaining in the hopper after its self-draining. In contrast, at the maximum flow rate, detected for powder fraction of approximately 0.2 mm, the maximum drained angles were noted. In the zone of maximum flow rate, the incidence of flow failures is increased affecting the drained angle. From this, drained angle could be understood as a sensitive indicator of the flow failures. Wherever the serious flow disturbances occur, such as for example arching and/or core flow (Prescot and Barnum 2000), the drained angle cannot (might not) be estimated.

The pharmaceutical powder excipients of different properties, such as the true and/or bulk density, hygroscopic properties, electrostatic charge, flowability, etc., were studied in this work. The correlation between the drained angle and the mass flow rate for the optimum flow behaviour particle size fraction of 0.400–0.500 mm is illustrated in Fig. 2. The full line, characterized with the milder slope, joins data representing the uniform

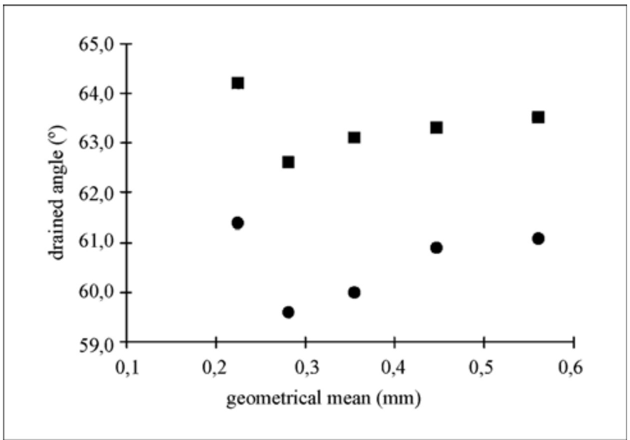

Fig. 1: Influence of geometrical mean of size fractions of sodium chloride (●) and sodium citrate (■) on drained angle

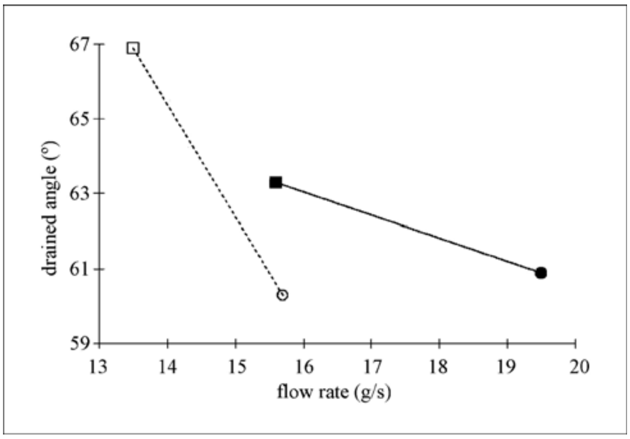

Fig. 2: Correlation between flow rate of particle size fraction 0.447 mm and drained angle (sodium chloride (●), sodium citrate (■), potassium chloride (○), and potassium citrate (□))

flow of sodium chloride and sodium citrate. On the other hand, the dashed line with the sharper slope demonstrates the flow of worse uniformity for potassium chloride and potassium citrate. The isometric but edgy particles of potassium chloride showed the just audible noise during the discharging from the hopper, while the anisometric, slightly hygroscopic particles of potassium citrate, showed slight flow failures having tendency to a mild funnel (core) flow due to the friction. In such situations, sometimes, the promotion of flow due to the mild tap was necessary. As a result, the mass flow rate of potassium citrate was lower in comparison to the other tested powders resulting in greater drained angle. The tendency of drained angle to increase if the flow rate decreases is in accordance with the complex nature of the powder flowability.

Mass flow rate is the crucial characteristic of free-flowable powders. Testing of mass flow rate is accomplished due to the fact that the mass flow rate is proportional to the 2.5 power of effective diameter of the test hopper orifice (Tighe and Sperl 2007). To estimate parameters of the flow equation, therefore, the measurement of material flow rates from hopper of different orifice sizes is needed. In contrast, no significant influence of drained angle by the different diameters of hopper orifice was found (Zatloukal and Šklubalová 2008). Then, the estimation of drained angle in testing of flowability is much easier using only one hopper, assuming the filling of the powder into a hopper in conformity with the standard conditions of bulk density measuring in a graduated cylinder. Moreover, the results of drained angle estimation are more reproducible than the ones of the angle of repose.

In principle, the estimation of drained angle is similar to the estimation of the optimal wall angle of the conical hopper needed for the powder mass flow and the complete emptying of hopper. Wherever the wall slope is lower, the funnel flow may occur resulting in residue of powder in the hopper except for powder gliding along the smooth walls. Unfortunately, this way, the flow kinetic might be influenced. In such situations, the steady state flow conditions should carefully be defined. The test funnels with different angles and orifice diameters are used to test flowability in pharmaceutical technology according to the European Pharmacopoeia. In conclusion, the wall angle of minimum 70° could be recommended to maintain the uniform flow of powder and to prevent any powder residue in the hopper.

Experimental

1. *Materials*

Four different powder excipients of pharmaceutical quality were used: sodium chloride, potassium chloride, sodium citrate dihydrate, and potassium citrate monohydrate. Size fractions of 0.200–0.250, 0.250–0.315, 0.315–0.400, 0.400–0.500, and 0.500–0.630 mm were obtained using a vibrating screen Pulverisette® 0 (Alfred Fritsch, Laborgerätebau, Idar Oberstein, Germany).

2. *Methods*

In accordance with the European Pharmacopoeia (‹616› Bulk density and tapped density), the bulk volumes of powder fractions were measured at the controlled ambient conditions (25 ± 1 °C, relative humidity of 40 ± 2.5%) and the bulk densities d_b (g/cm^3) were expressed.

The apparatus used for testing of flowability consisted of the model stainless steel cylindrical hopper with a flat bottom having a height of 10 cm and the inner diameter D equal to 4.0 cm. In the flat bottom, there was a concentric orifice having a height of 0.2 cm (i.e., equal to the thickness of the hopper bottom wall) and diameter $d = 1.0$ cm. Avoiding all vibrations, the model hopper was uniformly filled with the powder sample using the funnel of the outlet size 0.6 cm, maintained upright and placed 2 cm from the model hopper. The uniform mass discharge rate Q (g/s) was estimated as the time required to free steady-state discharge of the minimal amount of 80 g of powder from the hopper.

The discharged hopper with the residual powder was carefully weighed and the mass of the residual powder M (g) was estimated. Drained angle α (°) was calculated from Eq. (1) using the actual values of the hopper inner diameter D = 4.0 cm and the diameter of the circular orifice d = 1.0 cm:

$$\alpha = \mathrm{arctg}\left[\frac{\mathrm{M}}{10.6 \cdot \mathrm{d_b}}\right] \tag{2}$$

Each powder size fraction was characterized with an arithmetic mean of ten measurements of the mass discharge rate Q, the bulk density d_b and the mass of the residual powder M, the relative standard deviation of which was lower than 2% (for volume density and drained angle) and/or 1% (for mass flow rate), respectively.

Acknowledgement: This work was supported by the research project MSM 002160822 of the Ministry of Education of the Czech Republic.

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