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Measurement of the flow properties of powders with special reference to die fill

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Abstract

The flow behaviour of four pharmaceutical powders was investigated using a model shoe-die-filling system. The variation of mass delivered to the die as a function of shoe velocity provides a measure of flowability. The paper discusses the concept of critical velocity, above which incomplete filling is observed, in the context of pharmaceutical powders. The filling process was recorded using a high-speed video system, which allowed the different flow patterns to be observed, and how this influences the critical velocity to be evaluated. The influence of humidity, which was investigated in detail for one of the powders, was found to be small. The initial conditioning of the material, the die opening and if die filling takes place in air or in vacuum, however, were found to change the flow behaviour significantly.

Keywords: Powder flow; Shoe-die-filling rig; Critical velocity; High-speed video; Filling in air; Vacuum

1. Introduction

In this paper, we examine the flow characteristics of pharmaceutical powders. We start by describing the importance of powder flow in pharmaceutical powder processing and identify a range of techniques, which are routinely employed for measuring their flowability. Different techniques can provide a different rank order of the flowability of powders, and it is argued that any technique employed should capture the major history effects that the powder is subjected to during a particular unit operation. A model shoe-die-filling system is described, which captures some of the features of a rotary tableting press. High-speed video images of the delivery of four representative powders are presented and three major different types of flows are identified. A practical measure of the flowability of these powders is discussed and related to the observed flow mechanisms.

1.1. Powder flow in pharmaceutical processing operations

The flow properties of powders are important in manufacturing operations of solid dosage forms. These are determined by a combination of (i) powder characteristics, such as particle size, size distribution, shape, packing, density and surface properties and (ii) operating conditions, such as moisture, temperature, static charge, aeration, or history of applied stress. Powders

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flow under the influence of applied stresses, and during flow they may dilate, contract or flow at constant volume. The applied stresses arise from gravity, air pressure, external loading, vibrations and the constraints imposed by the containers in which flow takes place. These conditions dictate the behaviour of the powder when flowing through a hopper orifice, during pile formation or delivery into the die of a tablet compression machine.

A vast amount of research has been undertaken to develop experimental techniques suitable for characterising the flow properties of powders. When a powder is poured onto a surface, a pile is formed. One of the simplest indicators of the flow properties is the angle of repose, which is formed between the free surface of the powder and the horizontal surface. A good flowing powder spreads out and forms a low angle of repose. Hopper design is generally assisted by determining the flow properties of the powder through an orifice. This has resulted in the development of a variety of flowmeters over the years, which can be used to determine quantities such as the critical or minimum orifice size (Carr, 1965, Guyoncourt and Tweed, 2003) at which flow starts to occur, or the time for a fixed mass of powder to flow through a standard orifice (German, 1994). The initial condition of a powder is important and in order to obtain repeatable experimental results (especially for poorly flowing powders) pre-conditioning devices, such as a series of chutes or an upstream funnel, are often employed for the initial filling of devices such as the Scott volumeter (USP Chapter 616) or Flodex flowmeter (Manufactured by Hanson Research, Chatsworth, CA, USA). Other apparatus, such as the Freeman rheometer (Freeman, 2001), are based on measurement of the work done as the powder is stirred using a paddle. This type of device can also be employed to pre-condition a powder bed in a repeatable way prior to being tested.

One of the methods widely used in the pharmaceutical industry is based on measuring the poured and tapped densities of a powder, which are used for calculating indices such as the Carr index (Carr, 1965), Hausner ratio (Hausner, 1967) or angle of internal flow (Varthalis and Pilpel, 1975), which are related to the flow properties of the powder. A particular class of flow-meters, such as the Aero-Flow (manufactured by TSI incorporated, St. Paul, MN, USA) examines the formation of small avalanches and the position of powder in a rotating drum.

Finally, flow properties are also related to the cohesion and internal friction angle of the powder, which can be examined using low pressure triaxial testing (Li and Puri, 1996) or shear cells, which can either work by translation (Jenike, 1964) or rotation of the material. Rotational shear cells can be annular or full circle (Peschl, 1989). Parameters determined from these tests are often used to calibrate Mohr–Coulomb constitutive models, which originate from the soil mechanics literature and have been adapted for hopper design. A detailed description of these methods can be found in specialist books (Howard and Lai, 1992) and standards.

The flow measurement methods listed above can be used to characterise and classify the flow properties of powders. It is interesting however to note that these methods may give inconsistent classification for a given material. For example, zirconia powder flows very well through an orifice flowmeter, however, it can be regarded as a poor flowing powder when a model die-filling system is used (Guyoncourt and Tweed, 2003; Schneider et al., 2004). This behaviour is brought about by the flow properties being a consequence of the combined effect of the powder characteristics and the processing parameters discussed above. Therefore, it is important that the flow characterisation is carried out using a device which captures the physical phenomena involved in the process under consideration.

1.2. Die filling in rotary presses

In this section we examine the flow behaviour of powders within the delivery system of a rotary tablet press. During the tablet compression cycle the powder flows through a hopper into a feed frame and then into moving dies. Hopper flow has been investigated extensively and many of the flow characterisation methods described above have been developed specifically to assist hopper design. However, flow in the feed frame and into closed cavities, i.e. dies, has received less attention. In the following we examine the die filling process of a typical rotary tablet production press, using the characteristics of a Fette 1000 production press (Manufactured by Fette GMBH, Schwarzenbek, Germany), which can run at speeds between 0 and 75 rpm.

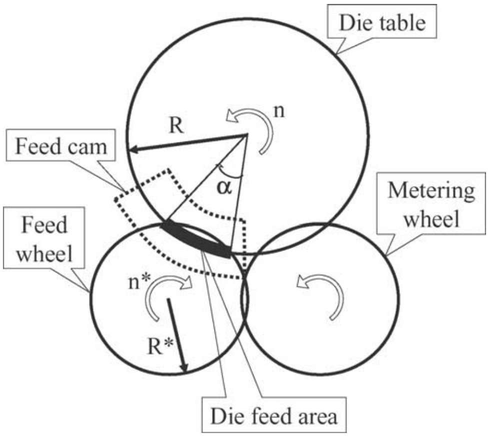

Fig. 1. Feed frame and die table for a rotary press.

The die table is presented diagrammatically in Fig. 1. The filling system consists of a hopper connected to a feed frame. The feed-frame is motor driven and consists of three wheels, for dispensing, feeding and metering, respectively. All three wheels operate at the same speed.

Of interest are the components in the immediate vicinity of the die table: the feeding and the metering wheel of the feed frame above the die table; and the fill cam and weight adjustment mechanism located below the die table, as presented in Fig. 1. The geometrical characteristics of the feed system (Fil-o-matic type) were measured, taken from the operating manual or estimated from available drawings as presented in Table 1.

The sense of rotation for the die table and feed and metering wheels are presented in Fig. 1. The powder is fed into the die under the feeding wheel in the area highlighted. In this process it passes under a number of spokes (N_p) of the feed wheel. This parameter is important because it relates to the experimental system described in the following sections where the number of passes necessary for complete die filling can be established experimentally.

Table 1
Characteristics and operating conditions of a rotary press feed system

Description	Symbol	Value
Press speed	n	0–75 rpm
Fill-o-matic speed, 0–100 rpm	n^*	0–100 rpm
Radius of die table	R	157 mm
Effective radius of feed wheel. The nominal radius of the wheel is 100 mm, the effective radius is taken to the centre of a die	R^*	78 mm
Angle where die receives powder	α	35°
No. of spokes of the feed wheel	N_s	16

Using elementary geometry, we obtain:

$$N_p = \frac{N_s \alpha}{360}\left(\frac{R}{R^*} - \frac{n^*}{n}\right) \qquad (1)$$

where all the symbols are defined in Table 1. If N_p is established experimentally, the required speed of the Fill-o-matic can be obtained by solving Eq. (1) for n.

Using the parameters presented in Table 1 and choosing a press speed and feed frame speed of 40 and 20 rpm, respectively, the resulting velocity of the die is 657 mm s^{-1}, while the velocity of the spokes is 163 mm s^{-1} in the same direction. This gives a relative velocity of the die with respect to the spokes of 494 mm s^{-1}. Also, $N_p = 2.35$, indicating that there will be a little more than two passes of the spokes over the die cavity while the die is under the feeding zone.

However, the previous discussion is rather simple and assumes gravity feed only. In practice, the following complex dynamics takes place: gravity feeding, force feeding, suction feeding, centrifugal forces and weight adjustment as described below.

Gravity feeding occurs as material from the powder bed above the die table deposits in the die under the effect of gravity. The arms of the feeding and metering wheel can be profiled such that the powder is forced into the die cavity, this effect is referred to as force feed. Also, there is a clearance between the top of the die table and the arms of the feed wheel of around 6 mm. This implies that there may be a zone where the powder is exposed to high shear conditions. The feed cam, which is shown as a dashed line in Fig. 1, is positioned such that the bottom punch has a linear movement downwards, which starts prior to the empty die being exposed to powder in the feed-frame and continues while the die passes through the filling zone. This creates a negative pressure (vacuum) between the punch and the powder bed, facilitating the fill of the die through a suction effect.The operating conditions can be chosen such that $n^* = n \times R/R^* \sim 2n$ then $N_p = 0$. In this situation there is no relative movement of the powder with respect to the die and the contribution to die filling from the sweeping movement of the feeding wheel is reduced. n^* and n can be chosen such that the relative velocity of the powder driven by the feeding wheel is in the same or the opposite direction to the rotation of the die table.

After passing through the fill cam, the bottom punch reaches the weight adjustment mechanism, where it is moved up rapidly to eject the excess weight of powder from the die. Then the top of the powder bed in the die passes under the metering wheel while the vertical velocity of the bottom punch is zero. It is not clear whether the die is filled completely after passing under the feed wheel or if there is some additional filling after weight adjustment when the die passes under the metering wheel. The inherent vibration of the press during operation may also facilitate gravity feed or packing of the powder as it is fed from the hopper to the feed frame.

The above analysis (which is conducted based on a Fette type rotary press feed system—other press manufacturers use similar or different arrangements) illustrates that die filling on a rotary press is a complicated phenomenon. The contribution of gravity feed, suction filling, centrifugal forces, weight adjustment, the effect of the metering wheel, vibrations from the press and air pressure can however be studied separately. The relative importance of these factors depends also on the characteristics of the powder, which also influence the permeability to air.

1.3. The effect of die fill on tablet compression

The importance of die fill has also been recognised in other industries, which use powder compaction, such as powder metallurgy or ceramics. Typical die-filling systems in these operations consist of a rectangular feed shoe moving at a given velocity across a stationary die. During this process, the material is deposited non-uniformly into the die. A number of shakes when the feed shoe is above the die may improve the filling process. Effects resulting from non-homogeneous die fill can propagate through the compaction process and influence the final properties of the compact. In order to study this process an experimental system was developed by Wu et al. (2003).

The die-fill phenomenon on a rotary tableting press is complex as described above. The design of a die-feed system is mostly based on empirical considerations. Nevertheless, die fill is an important and often neglected aspect; for example, situations have occurred when the appearance of the two halves of a capsule shaped tablet were different, raising elegance issues. Taking the effect of centrifugal forces into account resulted in re-design of the orientation of the dies with respect to the die table. Nonetheless, rules of thumb and knowledge of what has worked in the past still form the basis of feed frame design and choice of process parameters in pharmaceutical tableting operations.

Contributing factors to rotary press die fill, such as shoe velocity, environment, shoe and die design, or pre-conditioning, can be studied individually using the linearly moving shoe-filling system described by Wu et al. (2003). These studies can be directly related to some of the effects experienced in a rotary press. In this paper, we focus on gravity feed with special reference to the effect of feed shoe velocity. The contribution of air pressure is examined by performing experiments in air and vacuum.

2. Experimental details

2.1. Powders

Four powders were used in this study as presented in Table 2. Two of these powders, microcrystalline cellulose (Avicel PH102, manufactured by FMC BioPolymer, Cork, Ireland) and anhydrous lactose (Anhydrous Lactose—Direct Tableting, manufactured by Quest International) are common direct compression ingredients. A mixed placebo powder, which is based on these two excipients, was chosen to compare the flow behaviour of a mixture with its pure ingredients. The placebo mix also contains a disintegrant (croscarmellose sodium) and a lubricant (magnesium stearate). The microcrystalline cellulose described above has a nominal particle size of 100 μm. Finally, a 50 μm particle size microcrystalline cellulose, Avicel PH101 (manufactured by FMC BioPolymer, Cork, Ireland), was chosen to examine the effect of particle size on the flow properties of the powder into the die. In this work these two grades of microcrystalline cellulose are referred to as coarse and fine, respectively. Based on the Carr index values presented in Table 2, the anhydrous lactose is classified as good flowing

Table 2
Powders and characteristics

Powder	Description	Bulk density ($Mg\,m^{-3}$)	Tapped density ($Mg\,m^{-3}$)	Carr index	Nominal particle diameter (μm)
Placebo mix	Lactose, coarse MCC[a], disintegrant (croscarmellose sodium), lubricant (magnesium stearate)	0.52	0.72	29	N/A
Lactose	Lactose anhydrous direct tableting	0.68	0.81	16	100
Coarse MCC	Avicel PH102	0.34	0.48	29	100
Fine MCC	Avicel PH101	0.32	0.45	28–29	50

[a] MCC, microcrystalline cellulose.

while the other three powders are considered poor flowing.

2.2. Die-filling rig

A model shoe-die-filling system (Wu et al., 2003), see Fig. 2, has been used to allow the interaction between a stationary die and moving delivery system to be studied. The shoe, which is a rectangular shaped box, is filled with powder and translates over a transparent die with a steady state velocity between 10 and $1000\,mm\,s^{-1}$. The initial acceleration of the shoe can be varied between 1 and $100\,m\,s^{-2}$; in all the experiments presented here, however, the acceleration was kept constant at $50\,m\,s^{-2}$. As indicated previously, the condition of the powder has a strong influence on the flow characteristics and careful pre-conditioning is required to perform experiments representative of a large batch of material and to achieve good repeatability. Before placing it into the shoe, the powder was therefore pre-conditioned in a mixer/blender (Turbula T2F manufactured by Willy A. Bachofen AG Maschinenfabrik Basel/Switzerland) to avoid segregation due to transport and to loosen up the material after storage. The packing condition of the powder within the shoe can change due to the kinematics and the shearing of the powder on the fixed baseplate. The powder was therefore also conditioned between each test either by stirring it within the shoe or by refilling the shoe with fresh powder. The entire die-filling system is located in a transparent vacuum chamber. By conducting tests in air and vacuum, the influence of air, which is dis-

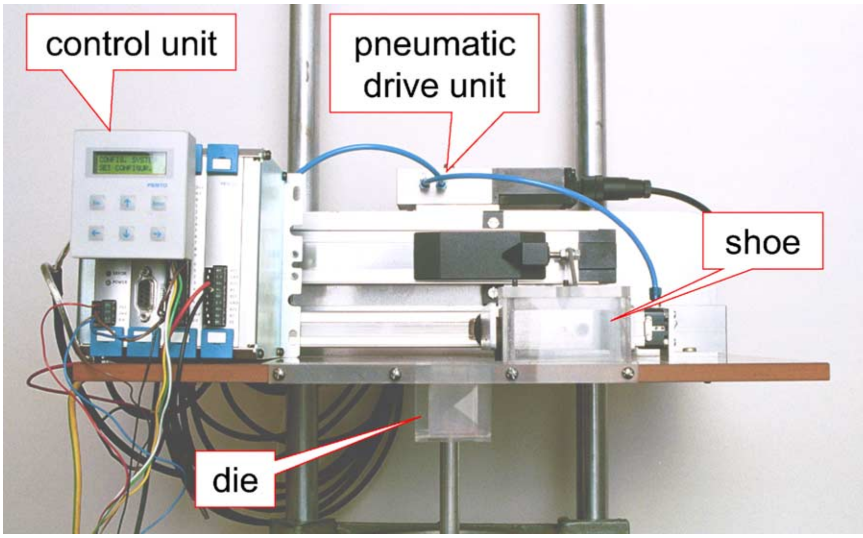

Fig. 2. Model shoe-die-filling system (Wu et al., 2003).

placed by the powder entering the die, can be determined. A high-speed video camera (NAC500 digital) was used for qualitative observations. More details of the shoe-die-filling system and studies on a range of different materials are presented elsewhere (Wu et al., 2003; Cocks et al., 2001). Before the shoe passes over the die a number of shakes, i.e. forward and reverse motion over a short distance, were employed to create a repeatable initial state of the material. This is particularly relevant for experiments in vacuum to allow trapped air to escape and to break down any air vents, which develop during evacuation. Apart from shoe velocity and environment, other parameters such as the height of the powder in the shoe, the die geometry and the shoe design can be varied to determine their influence on the filling behaviour.

As discussed previously, flowability can be defined and measured in various ways, but correlation between the results using the different techniques may be difficult to achieve. The present model shoe-die-filling system has the advantage that it provides a direct measure of flowability since it is similar to the die filling process. Results generated are therefore more representative of the real situation. High-speed video was used to evaluate the flow behaviour visually. It allows flow patterns, such as nose, intermittent and bulk flow, to be identified. When a fixed mass of powder is placed in a shoe, the initial acceleration of the shoe and friction between the powder mass and the baseplate forces the powder towards the back of the shoe, forming a nose shaped profile. As the tip of this nose translates across the die opening material can flow over the surface of the nose to the tip and avalanche into the die. We refer to this as *nose flow*. At high speeds, or if the die opening is small, the tip of the nose rapidly moves across the opening and powder is delivered into the die by detaching from the bottom free surface of the powder mass. If this flow is reasonably continuous we refer to it as *bulk flow*. For some materials, flow occurs as a result of a series of discrete instabilities, which releases large chunks of agglomerated powder into the die. This is a random, often infrequent, process and we refer to it as *intermittent flow*. More details on these three types of flow and how they are influenced by parameters such as shoe velocity and die opening are given elsewhere (Cocks et al., 2001; Wu et al., 2003; Schneider et al., 2004). High-speed video is also important in allowing differences between filling in air and vacuum to be visualised. Quantitative results are obtained by measurement of the mass of powder in the die as a function of shoe velocity. High-speed video studies were undertaken using stepped and simple rectangular shaped dies to examine different features of the material behaviour; all the quantitative results presented below were, however, determined using only the simple rectangular shaped die.

3. Results and discussion

3.1. High-speed video images

In this section we present a series of images taken from the high-speed videos. This provides information about the different flow mechanisms exhibited by the powders tested in this study, which can be used to explain the quantitative results presented in Section 3.2. Fig. 3 shows a series of still images from videos of the filling of a stepped cavity using the placebo and the fine microcrystalline cellulose powder. The filling of the two materials took place in air with a shoe velocity of 100 mm s^{-1}. The die opening has a width of 23 mm and a length of 20 mm, and this opening extends to a height of 20 mm; a step then creates a narrow section downstream of the direction of shoe motion of width 3.2 mm, which extends a further 18 mm. This profile is not representative of practical tablets, however the experiments allow us to illustrate some interesting features of the material behaviour, particularly the influence of air on the flow process. The top row of images in Fig. 3 shows the filling behaviour of the placebo mix. As the shoe moves over the die, powder is initially delivered from the front of the shoe. The forward velocity of the powder imparted by the shoe motion causes the powder initially to build up on the centre of the step and as the shoe moves forward the point of impact of the powder with the step moves towards the narrow section, until eventually powder is delivered directly into this section of the die. Also, as the shoe moves forward and powder is removed from the front of the shoe a nose type profile is created and there is a rapid avalanche of powder into the die. This results in a bridging of powder over the narrow section, trapping air in this section of the die. The pressure created opposes further flow of powder into this part of the die. Eventually, the powder mass completely

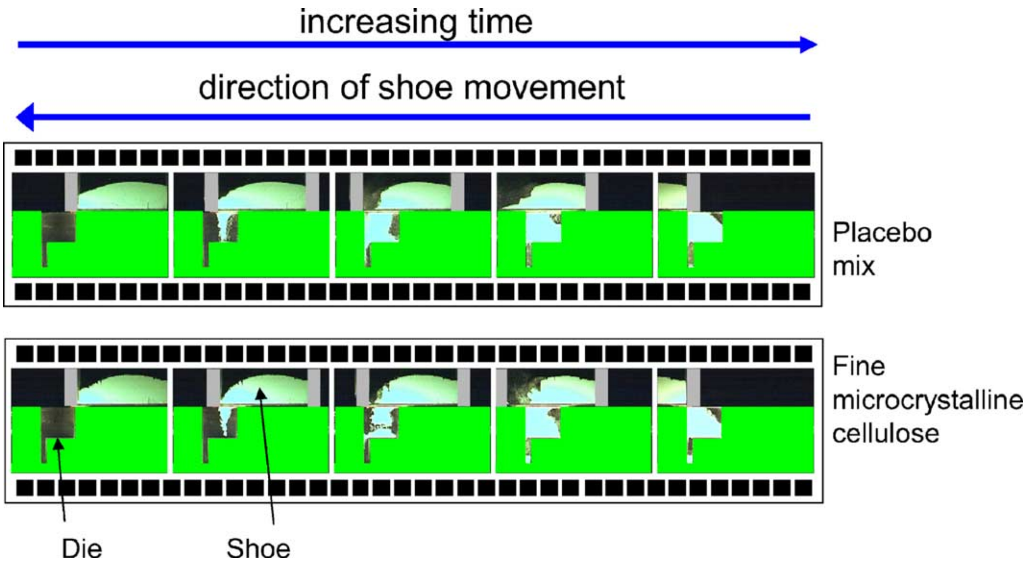

Fig. 3. High-speed video of filling of a stepped die with placebo mix (top) and fine microcrystalline cellulose (bottom). Filling in air with a shoe velocity of 100 mm s^{-1}.

covers the die. Powder now detaches from the bottom surface of the powder in the shoe and the upstream section of the die is gradually filled by powder cascading down the heap created on the step and flowing back towards the near corner of the die. The rate of filling gradually slows down as air becomes trapped and air pressure increases in the upper section of the die. The area through which powder is delivered into the die decreases to a width similar to that for the simple rectangular die shown in Fig. 4. The flow then becomes more intermittent and slower, preventing complete filling of the die and a depression remains in the top right corner after the shoe has passed. The process of air escape sometimes continues after the shoe has passed and the level of powder may drop in the die.

The filling process for the placebo mix is reasonably smooth. This can be contrasted with that of the fine microcrystalline cellulose powder, which is shown in the bottom row of Fig. 3. The initial stage of the flow process is similar to that for the placebo mix, but the flow is more intermittent, consisting of large agglomerates. The tip of the nose created by the initial flow then becomes unstable and a series of large blocks of powder inside the shoe detach and fall in a random manner into the die. The cohesive nature of the powder can be observed in Fig. 3 as steep sided fissures form and grow from the surface of the powder. These defects create the necessary imperfections for the creation of the blocks of powder that fall into the die. As with the placebo mix, the narrow section eventually bridges over and the delivery rate into the bulk of the die slows. The reasons for this are the same as for the placebo mix. The surface of the powder in the die after the delivery process is jagged, indicating the presence of large cohesive blocks of powder.

Fig. 4 shows a series of snapshots for the filling of a die with a 10 mm × 10 mm opening and a height of 38 mm using the placebo mix. Friction between the powder and the shoe wall can influence the flow of powder into the die. To minimize this powder-shoe wall effect the die was placed under the centre-line of the shoe. The shoe is 30 mm wide, i.e. transverse to the direction of shoe movement, and it is 60 mm long in the direction of movement. In all the experiments reported here the initial height of powder in the shoe was kept constant at 18 mm. The top set of images are for filling in air, while the bottom set are from an experiment conducted in vacuum. In each case the steady state velocity of the shoe as it traversed over the die was 50 mm s^{-1} and the sequence of images are for the same times in the filling process. For this die opening, most of the powder is delivered from the bottom sur-

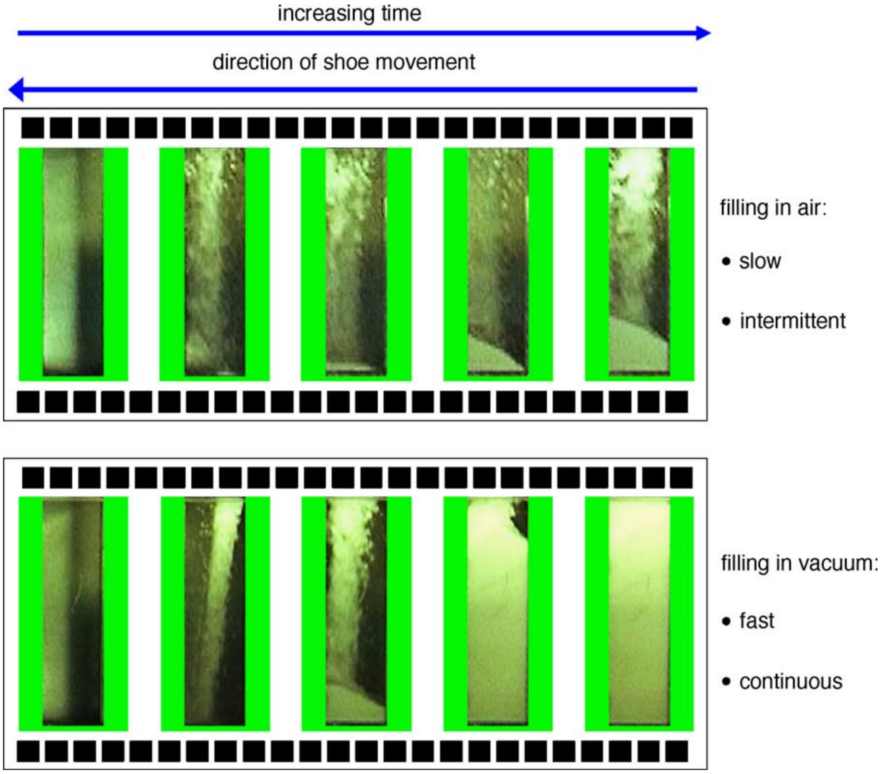

Fig. 4. Filling with placebo mix in air (top) and vacuum (bottom). The shoe velocity is $50\,mm\,s^{-1}$.

face of the powder mass in the shoe. The flow in vacuum is relatively smooth (bulk flow) and the die can be completely filled at this velocity. The flow in air is, however, intermittent. It is evident from the images that there are periods during the filling process when very little powder is flowing into the die. At other periods small clumps of agglomerated powder drop into the die, and at two instants during the entire filling process larger agglomerates detach from the bottom surface of the powder and fall into the die (as illustrated in the last image on the top row of Fig. 4). This latter type of event is responsible for delivering most of the powder into the die. Examination of videos of a number of filling events reveals that the size and number of these agglomerated clumps of powder, which fall into a die varies from one filling to the next; thus, there is significant scatter in the mass of powder that is delivered into a die when this type of intermittent mechanism dominates. This intermittent flow process is similar to that observed towards the end of the filling process described for the stepped die, Fig. 3, when the remaining area through which the powder is delivered to the die is similar to that for this simple die profile. It is also evident from these images that the details of the flow process is very sensitive to the environment (i.e. the presence of air has a significant effect).

Fig. 5 shows a series of images for filling the simple rectangular die in air for all four powders listed in Table 2. The shoe velocity is $50\,mm\,s^{-1}$ and the snapshots were taken when the shoe was in the same position over the die for all the materials investigated. As noted above, the placebo mix flows in an intermittent manner. The flow of the coarse microcrystalline cellulose is smoother, but at this velocity less material is delivered into the die than for the placebo mix. Also, only a small amount of the lactose and the fine microcrystalline cellulose is delivered into the die at this velocity and even at the slowest velocity used in the experiments ($10\,mm\,s^{-1}$) it was never possible to completely fill this die. A similar effect was observed for these two powders in vacuum, while the flow of

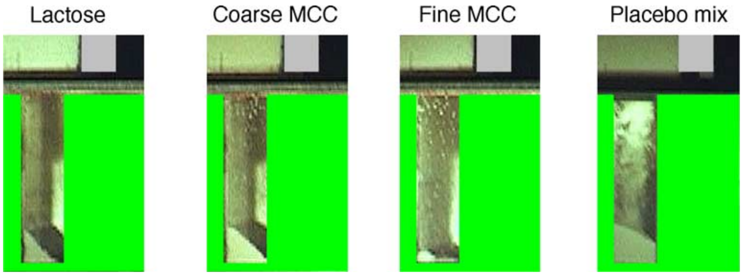

Fig. 5. Snapshots of filling behaviour of the different materials in air with a shoe velocity of 50 mm s^{-1}.

the placebo mix and the coarse microcrystalline cellulose powder was smooth in vacuum at all velocities. We discuss the significance of this observation in more detail in Section 3.2.

3.2. Critical velocity

Wu et al. (2003) have defined a critical shoe velocity v_c as a measure of flowability. The die is filled completely by a single pass of the shoe if the shoe velocity is below v_c. The influence of variables such as die geometry and environment (air/vacuum) on the critical velocity have been investigated. Here we base our measurement of the critical velocity on a series of experiments conducted using the simple rectangular die of Fig. 4, which were described in Section 3.1. The mass of powder in the die was measured as a function of shoe velocity. To compare materials and to determine critical velocities, the fill ratio, which is the ratio of the mass in die to the mass required to fill the die completely, was determined. The experimental data for the fill ratio δ and shoe velocity v were fit using a function of the form:

$$\delta = \left(\frac{v_c}{v}\right)^h \quad (2)$$

where v_c and h are fitting parameters. Fig. 6 shows the data for the placebo mix in air (Fig. 6(a)) and vacuum (Fig. 6(b)); a fill ratio of unity indicates that the die was filled completely; it can be seen that the scatter in fill ratio results for a given shoe velocity is large since for this die opening the material falls as large agglomerates into the die in a random manner, as shown in Fig. 4. The scatter in results, however, is less in vacuum, due to the smoother delivery process. The critical velocity is given as the intersection of the horizontal straight line, which passes through unity on the ordinate (Fig. 6), with the curved line at higher velocities, which is given by the fit to the data using Eq. (2). Data points in Fig. 6, which are not used to fit

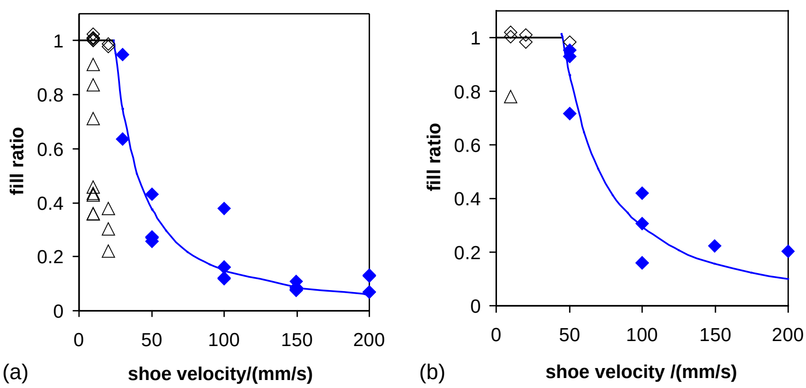

Fig. 6. Determination of the critical velocity for placebo mix using Eq. (2) to fit the data. Tests in (a) air and (b) vacuum. Solid markers indicate incomplete die fill. Triangles indicate data not used for fitting.

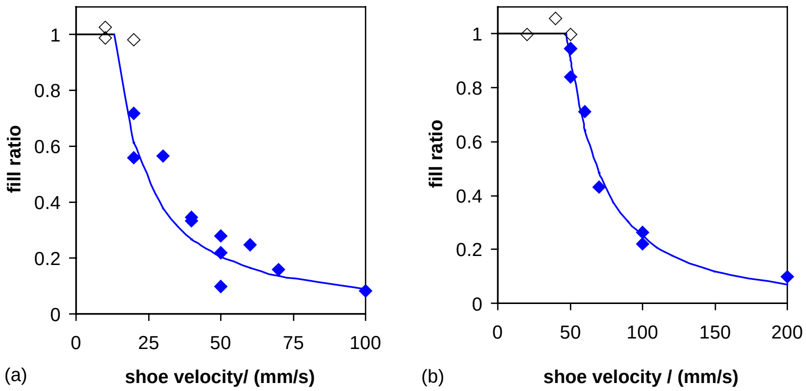

Fig. 7. Determination of the critical velocity for coarse microcrystalline cellulose using Eq. (2) to fit the data. Tests in (a) air and (b) vacuum. Solid markers indicate incomplete die fill.

the data are shown as triangles. The critical velocity determined when not taking into account all points is therefore a maximum possible critical velocity.

The experimental results for the coarse microcrystalline cellulose, however, follow a more consistent pattern, Fig. 5. The flow of this powder is much smoother and continuous, resulting in a greater reproducibility of the experimental results, as illustrated in Fig. 7 for air and vacuum.

As indicated before, it was not possible to determine critical velocities using the current die for the other two powders, the fine microcrystalline cellulose and lactose. Even at the slowest possible shoe velocity of 10 mm s^{-1}, or just by opening a shutter below the shoe while it is located over the die, it was not possible to completely fill the die.

For the coarse microcrystalline cellulose and the placebo mix the critical velocities in air are lower than measured in vacuum, as illustrated in Fig. 8. Smoother flows are generally observed in vacuum, and the filling rate is faster at all shoe velocities, as illustrated in Fig. 4, while the build up of pressure in the die in air opposes the flow process and promotes the development of instabilities. The densities achieved in vacuum are generally higher (Fig. 9) since a denser packing is achieved due to the absence of air repelling the filling process. Fig. 9 also indicates that the shoe filling process achieves a slightly higher fill density compared to the apparent density measured using the standard USP method for tap density measurement. Shoe filling is a more dynamic process and the powder particles can rearrange to create a denser packing.

Temperature and relative humidity can have a strong influence on the properties of pharmaceutical powders. For the set of experiments carried out here the temperature and relative humidity were recorded. For the placebo mix the relationship of fill ratio as a function of shoe velocity was determined at relative humidities of 30–65% and temperatures between 10 and 25 °C. To reduce the moisture content the material was heated at 100 °C for 2 h. Intermittent flow was observed in all cases and neither the critical velocity nor the scatter in fill ratio for a given shoe velocity was changed if the sample was dried or not.

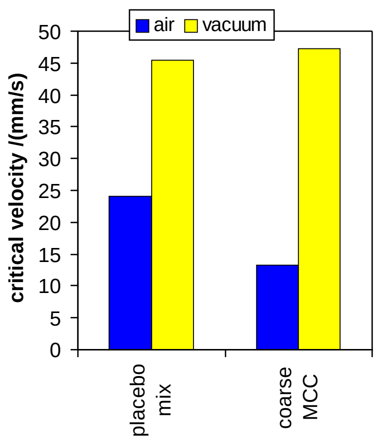

Fig. 8. Comparison of critical velocities of coarse microcrystalline cellulose and placebo mix in air and vacuum. The die and shoe geometry and initial powder height in the shoe were kept constant.

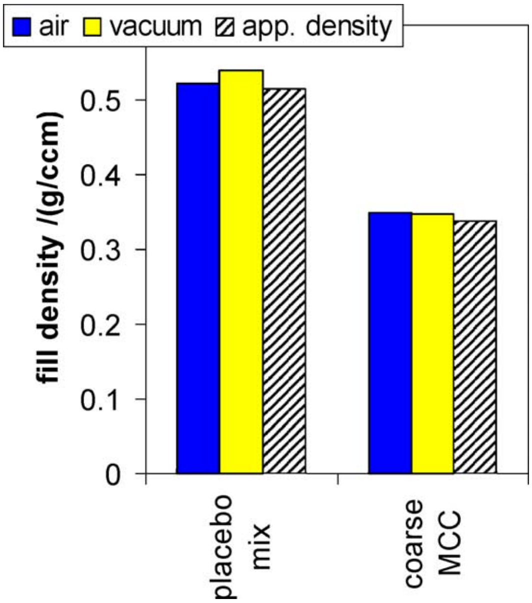

Fig. 9. Influence of environment on fill density and comparison with apparent density.

Schneider et al. (unpublished work) have shown that one parameter, the geometry of the die, has a significant influence on the flow behaviour and the scatter in the fill ratio-shoe velocity relationship. They show that the critical velocity generally increases if the die height is reduced or if the die opening size is increased. Schneider et al. (unpublished work) also found for the placebo mix that doubling the die opening significantly reduced the scatter shown in Fig. 6(a) in the fill ratio-velocity relationship. This effect is due to the fact that for the larger die opening, there is a greater probability that a local instability will occur at a given time to initiate a detachment event. Thus on average, over the filling period an equal number and scale of detachment events will occur creating a uniform net filling rate.

The critical velocities determined for the present simple die allows the flowability for different materials to be compared using a test whose kinematics are closer to that experienced in practical tableting presses. Schneider et al., 2004 demonstrate that the Beverloo equation (Beverloo et al., 1961), which is an empirical relationship originally developed for hopper flow, can be used to derive a relationship between die geometry and the critical velocity, which allows the data generated using a standard die to be used for a number of different die filling situations. The relationship is valid provided the flow is relatively smooth, i.e. not intermittent.

According to the Carr index data presented in Table 2, anhydrous lactose is good flowing while the other three powders are poor flowing. The model die-filling system however, demonstrates that the coarse microcrystalline cellulose and the placebo mix flows well compared with anhydrous lactose or the fine microcrystalline cellulose. It is therefore suggested that for die filling purposes the flow properties of powders should be measured using apparatus similar to the process under investigation.

The critical velocity measured using the model die-filling system can be related to the practical situation on a rotary press. Using the parameters presented in Table 1 we have compressed a small amount of placebo mix into acceptable tablets. This suggests that the press was operated below the critical velocity corresponding to the rotary press feed shoe system, i.e. the die was completely filled. A quantitative correlation of the critical velocity of the model die-filling system and the rotary press feed system requires a detailed analysis of the geometrical and operating parameters and is presented in detail elsewhere (Schneider et al., unpublished work). It is suggested that suction filling and the other effects described in Section 1.2 result in an increase of the apparent critical velocity of the rotary press feed system.

4. Conclusions

The present work compares the flow behaviour of four pharmaceutical powders. The flowability was evaluated using a model shoe-die-filling system. High-speed video was used to identify the dominant flow mechanism, i.e. nose, bulk or intermittent flow. If nose or bulk flow dominate, a critical velocity can be determined, which provides a measure of the flowability of the powder.

The large difference of critical velocities determined for filling in air and vacuum indicates that the effect of the build up of air pressure is significant. The critical velocity obtained using the experimental system can be related to the rotary press situation and it is suggested that the contribution from other factors (such as force feeding, suction feeding, centrifugal force, weight adjustment, vibrations, etc.) is significant.

The flowability of anhydrous lactose and that of the placebo mix, which consists of approximately 60% of lactose, were found to be significantly different. The flowability of the placebo mix is however similar to its other main ingredient, the coarse microcrystalline cellulose, suggesting that the flow properties of mixtures into dies should not be generalised.

The results obtained using the model die shoe system may or may not be consistent with the classification of powders using other flow measurement techniques. The die-filling system, however, reproduces some of the features of the industrial process; therefore, it is argued that the results may be more relevant from a die-fill point of view than other flow measurement methods.

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