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Practical implications of theoretical consideration of capsule filling by the dosator nozzle system

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Eight lactose size fractions with mean particle sizes ranging from 15·6 to 155·2 μm were characterized by their failure properties using a Jenike shear cell. The effective angle of internal friction was found to be constant for all size fractions, with a mean value of 36·2°. Jenike flow factors could only be obtained for the two most cohesive size fractions presumably due to limitations of the shear cell. Angles of wall friction, ϕ, were determined for all size fractions on face ground and turned stainless steel surfaces. These decreased with increasing particle size up to around 40 μm, above which they became effectively constant for both surfaces. The rougher turned plate gave consistently higher values of ϕ for each particle size. Simple retention experiments with a dosator nozzle and a range of powder bed bulk densities showed good retention was possible only up to a particle size of around 40 μm. Retention was difficult or impossible above this size. Values of ϕ were applied to equations derived in the theoretical approach described previously (Jolliffe et al 1980). This showed that the strength required within a powder to ensure arching increases with increasing particle size up to around 40 μm. Above this size, this strength requirement becomes constant. This is related to the powder retention observations. Finally, the failure data was used to calculate the minimum compressive stresses required to ensure powder retention within the dosator nozzle, by employing the equations described by Jollife et al (1980). This suggested that, as powders became more free flowing, a larger compressive stress is necessary and that the angle of wall friction should be lower to ensure stress is transmitted to the arching zone.

Some theoretical considerations on the automatic filling hard gelatin capsules by machines operating on the dosator nozzle principle have been reported by Jolliffe et al (1980). Such machines transfer powder from a powder feed bed to a capsule shell in an open ended tube (the nozzle). To assist powder retention by the nozzle, the powder entering it may be compressed by a piston to achieve transfer without powder loss.

By applying the hopper design theories of Walker (1966) and Walters (1973) to the problem of forming a stable powder system (i.e. an arch) in the nozzle, it was possible to calculate the strength required within the powder for arching to occur and the vertical compressive stress to be applied to the top of the powder by the piston to ensure this strength required for arching exists at the nozzle outlet. It was found that a major factor affecting the vertical compressive stress required to cause arching in a given powder was the angle of wall friction (ϕ) between the powder and the nozzle wall. An optimum value of ϕ could be calculated for which the vertical compressive stress applied to the top of the powder bed was a minimum. The application of minimal compressive stress is desirable both for rapid drug release (soft powder plugs have been shown to promote drug release (Mehta & Augsburger 1981) and for reducing machine wear.

The theoretical approach also suggested that cohesive powders could be retained with only small compressive stresses and were insensitive to angle of wall friction. However, free flowing powders required much larger compressive stresses and small deviations from the optimum value of ϕ produced a sharp increase in the magnitude of the required compressive stress. The practical significance of these theoretical predictions will be considered.

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MATERIALS AND METHODS

Materials

A sample of DMV-125 mesh lactose monohydrate was classified into eight particle size fractions. Their designation (A–H), method of prepartion and mean volumetric diameter (determined using a Coulter Counter) are given in Table 1.

Table 1. Particle size classes of lactose (DMV).

	Size fraction							
	A	B	C	D	E	F	G	H
	Nominal size range (μm)							
	−18·7	+18·7–26·5	+26·5–37·5	+37·5–53	+53–75	+75–105	+105–150	+150
	Method of preparation							
	Zig-zag classifier			Air-jet sieve				Vibrational sieve
Mean volumetric particle diameter (μm)	15·6	17·8	27·7	37·5	41·3	80·8	99·5	155·2
± standard deviation	±11·1	±9·4	±10·5	±14·3	±14·8	±22·0	±34·7	±59·6

Methods
Experimental work was performed in less than 40% relative humidity.

Determination of flow and failure properties
The flow and failure properties of each size fraction were determined with a Jenike type shear cell (Jenike 1961) essentially as described by Butcher (1973) with modifications and method details as described by Jolliffe (1980). Yield loci were plotted for four consolidating loads (each determined from 16 experiments) for each particle size. This enabled the effective yield locus to be determined and hence the calculation of the effective angle of internal fraction, δ.

In addition, the Jenike flow factor, FF, was calculated, where possible, from measurements of the unconfined yield stress f_c, and the major consolidating stress, σ_1.

Determination of angle of powder wall friction, ϕ
The angle of wall friction was determined using a modified shear cell system, originally described by Jenike (1961). The upper part of the shear cell (the shearing ring) remained unchanged, but the lower part was replaced by a metal plate (Fig. 1). The shearing ring was placed on the plate and filled with the test powder. The powder was scraped level, a shearing lid placed on top and a yoke and weight holder attached. The smallest weight was then hung on the weight holder and, after powder subsidence had ceased, a shear stress applied to the shearing ring and lid. The maximum shear stress, as measured by a load cell, was recorded. The normal load was then increased and shearing continued to give the shear stress corresponding to this normal load. This was repeated for at least six normal loads and the experiment repeated four times.

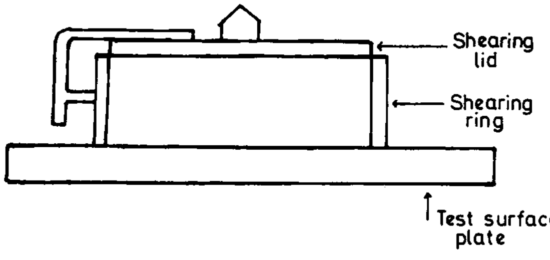

FIG. 1. Apparatus for wall yield locus determination.

Two wall surfaces were studied; these were turned (Ra = 0·55 μm) and face ground (Ra = 0·1 μm) finishes, representing rough and smooth surfaces respectively. These were cleaned initially in hot water (to remove any lactose) and then, after drying, carbon tetrachloride (to remove any grease), again followed by drying in air. Experiments were conducted with size fractions on each surface.

Wall yield loci were calculated by plotting shear stress, τ, as a function of normal load, σ. These were linear with a correlation coefficient of 0·9–1·0. Angles of wall friction were calculated from the tangent of the slope of the line. In all cases, the intercept was found to be effectively zero indicating there was no powder-wall adhesion.

Simple powder retention studies
A preliminary assessment of the ability of a number 3 sized dosator nozzle to remove and retain powder plugs of each size fraction was made, using powder beds of a range of bulk densities.

The dosator nozzle (without piston) was attached to a pneumatic piston so that it could be pushed into and raised out of a powder bed (Fig. 2). Powder beds were prepared to different bulk densities in the shear cell by the loading and twisting procedure used in the shear cell experiments. These powder beds were positioned centrally under the raised nozzle and the nozzle pushed into the powder bed under a pneumatic pressure of 2·5 kgf cm^{-2}. The nozzle was then raised under a minimal pressure so that the powder

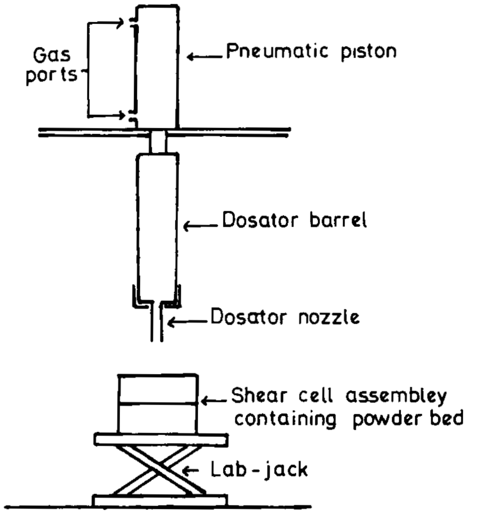

FIG. 2. Diagram of static rig for powder retention studies.

plug in the nozzle was not disturbed. The sampling powder bed was then removed and the nozzle raised and lowered through 5 cm under a pressure of 1 kgf cm^{-2} so that the plug was jolted at each end of the nozzle's travel. Retention was measured by the number of tapping strokes required to cause all the powder to drop out of the nozzle.

RESULTS AND DISCUSSION

Powder flow and failure

Values obtained for the effective angle of internal friction and flow factor are given in Table 2.

Flow factors could only be reliably determined for the two finest size fractions, A and B. The other size fractions produced yield loci with very small or even apparently negative intercepts on the τ axis which prevented reliable calculation of f_c. This is probably due to the inability of this type of shear cell to detect small changes in the flow properties of powders with low interparticle interaction or which are free flowing. The difficulties in obtaining accurate values of τ at low normal loads has been discussed elsewhere (Jenike 1961; Eckhoff & Leversen 1974; Eckhoff et al 1978).

Table 2. Values of effective angle of internal friction (δ) and flow factor.

Size fraction	Mean diameter (μm)	δ°	FF
A	15·6	37·09	20·03
B	17·8	35·47	44·38
C	22·7	37·97	—
D	37·5	36·81	—
E	41·3	35·77	—
F	80·8	35·12	—
G	99·5	35·52	—
H	155·2	35·76	—
Mean		36·20	
s.d.		1·00	

The effective angle of internal friction is constant for all the particle sizes tested, suggesting that, for this size range, the value of δ is independent of particle size and reflects only friction between lactose particles. The mean value (36·2°) is close to the value of 38·3° obtained by Kocova & Pilpel (1971) for crystalline lactose with a mean particle size of 13·5 μm. Their work also showed that δ decreases sharply from a value of 49° at 4 μm but decreases only gradually for particle sizes above 7·0 μm.

Angle of wall friction

Values of ϕ are plotted as a function of mean particle size for both surfaces in Fig. 3. These results show that, for both the face ground and turned surfaces, ϕ decreases with increasing particle size up to around 40 μm. Above this size ϕ becomes effectively constant. Values of ϕ for the face ground surface are consistently lower, showing that the friction between the powder and this smoother surface is less as would be expected. Frictional effects are also affected by particle size. Apparently, this is not just a simple reflection of particles to fit into the surface (as has been described by Strijbos et al (1977) for ferric oxide powders) since the change in the curve occurs at approximately the same particle size for both surfaces, despite the differences in the magnitude of the surface features. The answer may be related to powder properties since angle of repose, angle of internal flow, uniformity coefficient and constants

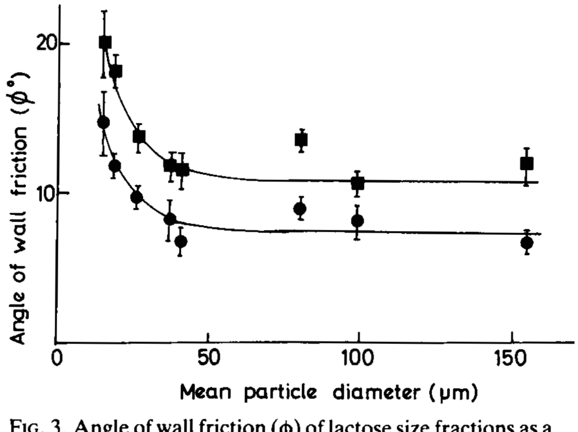

FIG. 3. Angle of wall friction (ϕ) of lactose size fractions as a function of particle size ● = Face ground stainless steel surface. ■ = Turned stainless steel surface. I = one standard deviation.

derived from the equation of Kawakita & Ludde (1970) for these powders all underwent a major change at around the 40 μm size (Jolliffe 1980).

Powder retention studies

The results of this work are presented in Table 3. Because this method is rather crude, it was not possible to establish an exact relationship between bulk density and retention (although there was a definite trend of increased retention with increased bulk density); however, the experiment did show the ease with which the particle size fraction could be retained. The results indicate that powder retention occurs readily under these conditions for particle sizes up to 40 μm, but above this size powder is retained only with difficulty.

Table 3. Ability to retain powder slugs from powder beds of different particle size fractions using a dosator nozzle.

Size fraction	Mean vol diam μm	Range of bulk densities tested $g\,cm^{-3}$	Retention ability
A	15·6	0·380–0·735	Retained with all the bulk densities tested and cannot be removed by this method
D	37·5	0·693–0·898	Can be retained at all bulk
E	41·3	0·683–0·866	densities but cannot be removed at the highest bulk densities tested
F	80·8	0·743–0·872	Retained only at the very highest bulk density and readily removed
G	99·5	0·738–0·835	Not retained at any bulk density
H	155·2	0·734–0·835	tested

Application of experimental results to the theoretical considerations

The basis of the theoretical approach to powder retention within a dosator nozzle is Walker's (1966) theory for calculating arching conditions in parallel sided hoppers (with its modification by Walters 1973). Walker's theory is founded firstly on the stress-span relationship i.e. the maximum span across which arching can occur is limited by the maximum shear which can be developed at the walls to support the powder; and, secondly, on the concept that since a powder arch has a free surface, the strength of the powder forming the arch has a free surface, the strength of the powder forming the arch must be the unconfined yield strength, f_c. The strength required within a powder for arching can be calculated from equation (1) derived from these concepts:

$$f_c = \frac{r \gamma g}{\sin 2\phi} \qquad (1)$$

where r is the span radius, γ the bulk density and g the acceleration due to gravity.

Calculation of the strength required in a powder for arching to occur in a nozzle

The strengths required within a powder for arching to occur in a parallel sided cylinder of size number 3 dosator nozzle dimensions were calculated for each particle size, using values of φ determined earlier (a value of γ = 800·0 kg^{-3} was used as this represents a tightly packed state for each particle size, and a nozzle diameter of 0·004 m). The values of f_c obtained are plotted as a function of particle size in Fig. 4. This shows that the rougher, turned surface (giving a higher value of φ) gives lower values of f_c than the smoother, face ground surface, for each particle size. Rough wall surfaces, therefore, promote arching by reducing the strength required within the powder for arching to occur.

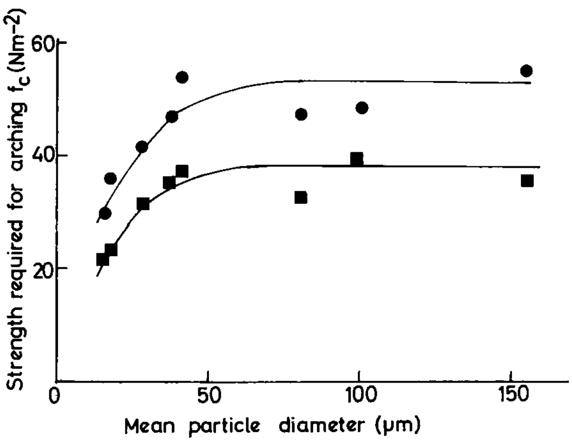

FIG. 4. The strength required in the powder for arching to occur (f_c) as a function of mean particle size of lactose.
● Face ground surface. ■ turned surface.

Fig. 4 also shows that, for a given surface, the strength required within the powder for arching to occur increases with increasing particle size up to 40 μm. Above this size the strength required is constant, i.e. the strength required for arching increases as the powders become more free flowing, but the larger free flowing particle sizes all require the same strength. This effect can be related to the powder retention experiments. The results in Table 3 indicated that, while powder retention was possible over a wide range of bulk densities for size fractions up to around 40 μm (with especially strong retention at the smallest particle sizes), retention was generally not possible with the larger, free flowing particle sizes.

This suggests that the low arching strength requirements of cohesive materials can be met under the conditions of the retention experiments; but the much larger strength required within free flowing powders (for the same surface) cannot be achieved.

Calculation of the minimum compressive stress required to be applied to the powder in the nozzle to ensure retention

The theoretical considerations were developed further by Jolliffe et al (1980), enabling the vertical compressive stress required to act on the powder at the arching zone, $\bar{\sigma}_{z\ req}$, for the powder to possess strength f_c and hence form a stable arch (eqn 2):

$$\bar{\sigma}_{z\ req} = \frac{FF\, r \gamma g}{\sin 2\phi} \qquad (2)$$

Finally, an equation (equation (3)) was produced for calculating the compressive stress that must be applied to the top of the powder bed ($\sigma_{z,o\ req}$) to ensure sufficient stress ($\bar{\sigma}_{z\ req}$) is transferred to the arching zone to satisfy arching strength requirements:

$$-\sigma_{z,o\ req} = \frac{\bar{\sigma}_{z\ req} - r\gamma g(1 - e^{-2BD\frac{z}{r}})}{e^{-2BD\frac{z}{r}}} \qquad (3)$$

where BD is a function of ϕ and δ and describes the distribution of stress through the powder bed, z is the powder depth.

Jolliffe et al (1982) showed that $\sigma_{z,o\ req}$ passes through a minimum when plotted as a function of ϕ. This showed that to ensure retention of a given powder within the nozzle, there was an optimum value of angle of wall friction for which the compressive stress applied to the top of a powder bed is a minimum.

Equations (2) and (3) require a knowledge of the powder flow factor, *FF*, which in practice could only be obtained for the most cohesive size fractions A and B. Hence, calculations were only possible for these size fractions. The data and results of the calculations are given in Table 4.

The calculations show that the more free flowing particle size, B, requires a larger minimum compressive stress to ensure retention and that this minimum occurs at a smaller angle of wall friction than for size fraction A. This agrees with the conclusions drawn from the theoretical considerations, i.e. as powders become more free flowing, a greater compressive stress is required for the powder to be retained and that, to transmit sufficient stress to the arching zone, the angle of wall friction should be lower.

Table 4. Data used in the calculation of the minimum value of $\sigma_{z,o\ req}$ and the value of ϕ at which the minimum occurs.

Data used	Calculated values: Minimum $\sigma_{z,o\ req}$ Nm^{-2}	when $\bar{\sigma}_{req}$ Nm^{-2}	at ϕ°
(i) *Size fraction A*			
E 0·475			
ρ 1529·0 kg m^{-3}	1012·7	451·9	20·0
γ 802·7 kg m^{-3}			
r 0·002 m			
d 0·010 m			
δ 37·09°			
FF 20·03			
(ii) *Size fraction B*			
E 0·475			
ρ 1530·0 kg m^{-3}	2571·0	1113·2	18·0
γ 802·3 kg m^{-3}			
r 0·002 m			
d 0·010 m			
δ 35·47°			
FF 44·38			

The compressive stress values calculated are very small and much smaller than those measured during capsule filling studies (Jolliffe 1980; Jolliffe et al 1982) and at present beyond the sensitivity of our measuring equipment.

Conclusion

The use of experimentally determined angle of wall friction for eight lactose size fractions (on two different surfaces) in equations described previously (Jolliffe et al 1980) has shown that the strength required within a powder for arching, and hence retention in a dosator nozzle, increases with increasing particle size up to a constant value at around 40 μm. Simple retention experiments apparently confirm this by showing that powder retention is readily achieved up to this particle size, but that above this size retention is very difficult or impossible.

In powder failure studies, flow factors could not be obtained for particle sizes over 17·8 μm; apparently due to the limitations of the shear cell technique. Effective angles of internal friction, determined for each size fraction, were found to be effectively constant for all size fractions and gave a mean value of 36·2°.

Application of the failure data to the equations described by Jolliffe et al (1980) enabled the minimum vertical compressive stress required to be applied to the powder within the nozzle to ensure retention to be calculated and the optimum angles of wall friction for which these occur. These results, although limited to the two size fractions for which flow factors could be determined, agree with the conclusions of the theoretical approach.

Symbols

BD	function of ϕ and δ accounting for stress distribution through the powder bed (See Jolliffe et al 1980).
E	porosity
FF	flow factor
f_c	unconfined yield stress
g	acceleration due to gravity
r	powder bed radius
Ra	arithmetic mean deviation
z	powder bed depth
γ	bulk density
δ	effective angle of internal friction
ρ	particle density
σ	normal stress
$\bar{\sigma}_{z\ req}$	mean vertical compressive stress required at the arching zone for arching to occur
$\bar{\sigma}_{z,o\ req}$	vertical compressive stress required to be applied to top of powder bed for arching to occur
τ	shear stress
ϕ	angle of wall friction

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