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The effect of dosator nozzle wall texture on capsule filling with the mG2 simulator

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The effect on capsule filling, using an mG2 simulator, of the surface texture of the bore of the dosator nozzle has been investigated for size fractions of lactose. Since the angle of powder-wall friction between the powder and the nozzle cannot be readily measured in-situ, this was determined using flat plates with similar surface textures fitted into a Jenike shear cell apparatus. The problems of reproducing surface textures on both types of surface are discussed and a lapping process employed as the most suitable method. Angles of wall friction for the nozzle surfaces were extrapolated from the values obtained from flat plates of similar roughness. Capsule filling experiments, using the mG2 simulator, showed the three resurfaced nozzles produced more uniform fill weights with smaller measured compression and ejection stresses than an untreated nozzle surface. One lapped nozzle surface produced a slightly greater improvement than the others. This supports the concept of an optimum angle of wall friction for powder retention (and hence uniformity of fill) with a minimum of applied compression stress.

Automatic hard gelatin capsule filling using the dosator nozzle system requires that the dosator nozzle retains a powder dose, allowing it to be transferred from a feed tray to a capsule shell. The application of hopper design theory to the retention process (Jolliffe et al 1980) has shown that it is controlled not only by powder properties, but also by the interaction between the powder and the nozzle wall as measured by the angle of wall friction, ϕ. The theory allowed the calculation of the compressive stress required to be applied to the powder to ensure retention for a given powder and angle of wall friction. It also indicated that for a given powder there is a certain angle of wall friction for which the compression required for retention is a minimum. Such correlations between retention ability and interaction with a wall surface have been presented for simple systems (Jolliffe & Newton 1982a).

The present study has employed an instrumented mG2 simulator (Jolliffe et al 1982) which is able to measure compression and ejection stresses that can be correlated with the capsule fill weights achieved. By preparing nozzles with bores having a range of surface textures, the effects of the powder-nozzle wall interaction on these parameters can be investigated.

Preparation of similar surface textures on flat stainless steel plates has enabled the angle of wall friction to be determined and hence an estimation of the value of ϕ existing between the powder and the wall surface within the dosator nozzle. Wall friction determinations and the difficulties encountered in producing suitable surface textures are discussed before capsule filling studies are described.

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MATERIALS AND METHODS

Materials

Size fractions of a sample of DMV lactose—125 mesh, which had been split into eight size fractions (A–H) with mean volumetric diameters 15·6–155·2 μm (Coulter Counter), were used. The size fractions employed were A (15·6 μm), D (37·5 μm) and H (155·2 μm) representing a range of flow properties. Before use, the powders were passed through a suitable sized sieve to break up any agglomerates.

Surface preparation

The angle of wall friction between the powder and the nozzle wall cannot be measured directly because the small diameter of the nozzle bore makes its surfaces inaccessible. To overcome this, resurfacing methods which can be used to produce similar surface textures on both a nozzle wall and a flat metal surface, suitable for fitting to the Jenike shear cell for wall yield locus determinations, were used.

Examination of the original nozzle surface showed that it was a turned surface, produced by a single point boring tool. However, to produce a range of turned surfaces with different roughnesses requires that a wide range of parameters be defined, e.g. the tool material, geometry and holding, cutting speed and feed rate and the machine used. In addition, there are particular problems with this method; (a) the internal diameter of the nozzle bore (4 mm) requires the use of a very small tool which would be unsuitable for producing a texture on 8 cm diameter plates (required for the Jenike shear cell) as it would rapidly wear, (b) stainless steel (used for both nozzles and plates) requires a high cutting speed which would be difficult in the small diameter of the nozzle, (c) although the cutting speed would remain constant inside the nozzle bore, it would become less as the tool moved towards the centre of the plate producing a change in cutting characteristics.

For these reasons, two methods were tried for producing closely matching surface textures on the nozzle bore and the flat plate (the same grade of stainless steel was used for each). These were spark erosion and lapping.

Spark erosion. This technique uses electron bombardment to vaporize metal on the surface. The more rapidly metal is removed, the rougher the surface produced, and this gives a non-directional texture, unlike, e.g. turning. A revolving planing tool was employed to produce a range of six surface textures on the flat wall friction plates. Nozzle surface were not produced (see later).

Lapping. This involves moving an abrasive paste over the metal surface, the grade of paste determining the finish. A range of surface textures was produced on the nozzle surfaces and the wall friction plates using similar diamond pastes. Whilst this should ensure surface features of a similar shape, the different orientation of the two surfaces required the use of different machining techniques which resulted in the flat plate having a close intermesh of small diameter circles, and the nozzle a criss-cross pattern. Six wall friction plate surfaces and three nozzle surfaces were produced (a fourth nozzle was retained with its original surface).

Surface texture measurement

The measurement of surface texture is complex since, not only is there variation in the height and depth of peaks and troughs between and within samples, but also the shape of these features varies with the use of different cutting methods. One approach to the measurement of surface texture is considered in detail in BS 1134: 1972. In practice, the arithmetic mean deviation, *Ra*, is commonly used in the comparison of similar surfaces. *Ra* is calculated as the sum of the areas of the profile of a surface above and below a reference line per unit length of surface measured, i.e. large values of *Ra* indicate rough surfaces. Since no account is taken of the shape of surface features, surface texture cannot be fully defined by *Ra* alone and other factors may be derived.

In practice, the *Ra* value is determined using an electrically indicating stylus instrument (e.g. Talysurf 4) which produces a magnified recording of the surface profile and calculates and displays an *Ra* value.

Ra values and recordings were determined for all the surfaces produced and are presented in Table 1. The flat metal plates could be tested directly, but replica castings with a special resin were made of the nozzle bore since it was too narrow for direct measurement.

Table 1. *Ra* values for surface plates and nozzles.

Surface description	Designation	*Ra* (μm)
(a) Plates		
Face ground	S	0.10
Turned	R	0·55
Spark eroded	7	0·60
,, ,,	6	0·90
,, ,,	5	2·00
,, ,,	4	3·50
,, ,,	3	5·00
,, ,,	2	5·50
Lapped	A	0·02
,,	B	0·10
,,	C	0·50
,,	D	0·25
,,	E	0·45
,,	F	0·30
(b) Nozzles		
Untreated	A	1·60
Lapped	X	0·80
,,	Y	0·15
,,	Z	0·35

Determination of angle of wall friction (ϕ)

The method used to measure angle of wall friction for the size fractions over the range of surfaces produced has been described (Jolliffe & Newton 1982a), and discussed in detail (Jolliffe 1980). Essentially it consists of a Jenike shear cell with the lower half of the cell replaced by the test surface plate. The top part of the shear cell is filled with the test powder and the shear stress required to push the powder along the surface under an increasing normal load is measured. ϕ is calculated from the slope of a plot of shear stress as a function of normal load (the wall yield locus). Generally, each wall yield locus was plotted from six points each repeated five times. For all the results obtained here wall yield loci were linear ($r = 0{\cdot}9$–$1{\cdot}0$).

Test surfaces were cleaned between each determination in hot water (to remove lactose) and then carbon tetrachloride (to remove any grease) before being thoroughly dried in air.

Values of ϕ were determined for the six spark-eroded and six lapped surfaces for size fractions A, D and H. Values of ϕ have been previously presented (Jolliffe & Newton 1982a) for a turned ($Ra = 0{\cdot}55$ µm) and a face ground surface ($Ra = 0{\cdot}1$ µm).

Capsule filling

Capsule filling was studied using the mG2 automatic capsule filling machine simulator (Jolliffe et al 1982). Twenty capsules were filled in each experiment and their contents weighed and correlated to the compression and ejection stresses and piston movements measured. The nozzle bores were initially cleaned as described above.

Results are presented for size fraction D (37·5 µm) which has been shown to be sensitive to the amount of compression used (i.e. it cannot be filled without a minimum compression stress but gives poor uniformity of fill at high compression stress because of powder compaction) (Jolliffe & Newton 1982b). The results are presented as mean capsule fill weight and mean compression and ejection stresses (and variance) as a function of compression ratio, *Pr*.

$$Pr = \frac{\text{change in height of the powder bed on compression}}{\text{original height of powder bed}}$$

(Takagi et al 1969).

RESULTS AND DISCUSSION

Values of angle of wall friction, ϕ, for the test surfaces

In the production of the resurfaced plates and nozzles, it was the intention to provide surface textures with a range of *Ra* values extending above and below that of the original nozzle, i.e. nozzle A ($Ra = 1{\cdot}6$ µm). Table 1 shows that this objective was only achieved for the spark eroded plates, the values for all other surface treatments producing surfaces with *Ra* values less than 1·6 µm. Spark erosion therefore, appeared to be the method of choice in terms of providing different surface textures.

Spark eroded surfaces. The smoothest spark-eroded plate had a similar *Ra* value to the turned surface used previously (Jolliffe & Newton 1982a), while all the other plates prepared by this process had considerably higher *Ra* values (see Table 1).

For equivalent *Ra* values, the spark eroded surface had a value of wall friction ϕ greater than that for surfaces produced by either turning or lapping. Close examination of the recordings of the surface profile of the surfaces of the plates revealed that for approximately equal values of *Ra*, the number of peaks and valleys per unit length was much greater for the spark eroded surface than the turned surface. Thus if the surfaces are produced by very different processes, comparison of surface texture by use of *Ra* values alone may not be adequate, a more detailed characterization being required.

The wide range in the values of *Ra* produced by spark erosion did not produce a large variation in the angle of wall friction, see Fig. 1. An examination of the surface profile recordings indicated that as the

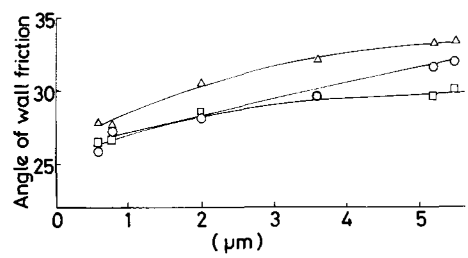

FIG. 1. Angle of wall friction (ϕ) of lactose size fractions as a function of *Ra* (µm) of spark-eroded surface textures. Size fraction △ A, ○ D, □ H.

value of *Ra* increased, peaks and valleys became higher, deeper and further apart, and it is presumably these changes which resulted in the increase in the values of *Ra* which did occur. Eventually the valleys became of sufficient depth to trap particles within their depth and when this occurs, the moving particles will in fact be sliding over a surface of trapped particles rather than the metal surface itself. That the value of wall friction approaches the angle of internal friction of these powders (Jolliffe & Newton 1982a) suggests that particle/particle friction is occurring for these surfaces.

The small range of values of wall friction achieved for this type of surface suggested the technique to be unsuitable for the purpose of producing nozzles of different *Ra* values and was not therefore used for this purpose. The results, however, clearly indicate that the type of surface preparation is extremely important and care in characterization of surface texture is essential.

Lapped surfaces. The *Ra* values of surfaces produced by this method are lower and cover a smaller range than those for the spark-eroded surfaces. These lapped surfaces have a profile of small irregular peaks and troughs. When ϕ is plotted as a function of *Ra* for size fractions A, D and H (Fig. 2), straight lines can be drawn for size fractions D and H (r = 0·971 and 0·966 respectively). The results for size fraction A are best fitted by a curve with levels off at higher values of ϕ. The difference in interaction of the different size fractions with a given surface has been discussed (Jolliffe & Newton 1982a).

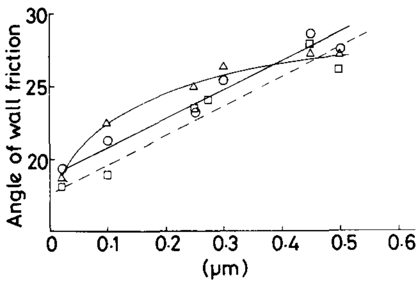

FIG. 2. Angle of wall friction (ϕ) of lactose as a function of the value of *Ra* (μm) of lapped surfaces. △——△ 15.6 μm size fraction. ○——○ 37.5 μm size fraction. □- - - -□ 155.2 μm size fraction.

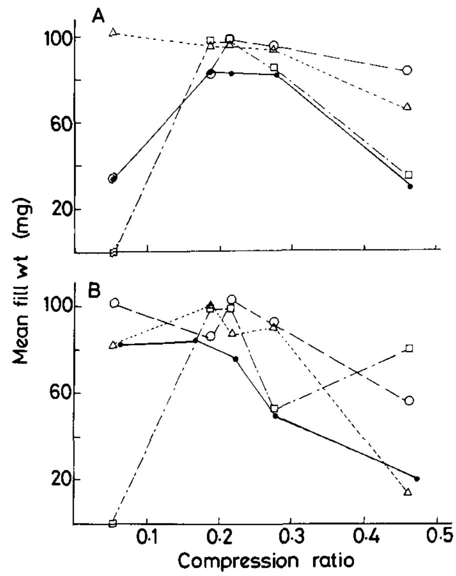

FIG. 3. Mean capsule fill weight as a function of simulator compression ratio for 37·5 μm size fraction. Nozzles A, X, Y and Z. (A) clean, (B) coated. ●——● Nozzle A, *Ra* 1·6 μm. ○— —○ Nozzle X, *Ra* 0·8 μm. △- - -△ Nozzle Y, *Ra* 0·15 μm, □—·—□ Nozzle Z, *Ra* 0·35 μm.

The values of ϕ for the range of *Ra* values measured for these surfaces covers a range of approximately 10° and therefore this method was used to produce the nozzle surfaces.

Capsule filling

The four nozzle surfaces used were A an original nozzle (*Ra* = 1·6 μm) and three lapped nozzles X, Y and Z (*Ra* values 0·8, 0·15 and 0·35 respectively). The fill weights for both clean nozzles and those on which a constant powder coat had been built up are shown in Fig. 3 (a) and (b) and the corresponding fill weight variances in Fig. 4 (a) and (b) achieved highest and most uniform fill weights between *Pr* = 0·19 and 0·22 showing that a certain degree of compression is required to ensure powder retention but that excessive amounts of compression may lead to poor uniformity of fill due to powder compaction (as discussed in Jolliffe & Newton 1982b).

The original nozzle, A, generally gave lower fill weights and higher fill weight variance at a given *Pr* than the resurfaced nozzles. For nozzle surfaces X and Y the compression ratio giving the highest mean fill weights and greatest uniformity changed when the nozzle becomes coated. However, with nozzle Z this change was not observed and it appeared to give the most uniform fill weights.

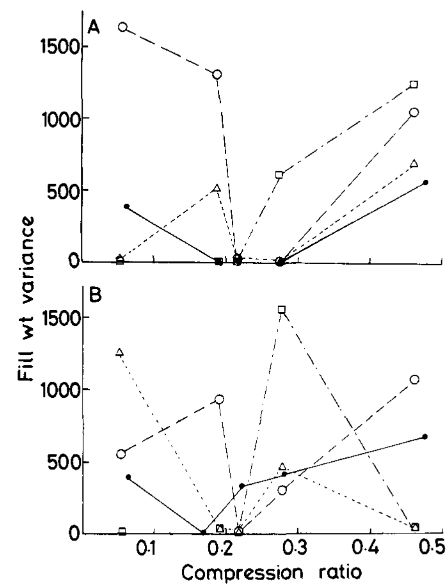

FIG. 4. Capsule fill weight variance as a function of simulator compression ratio. 37·5 μm size fraction. Nozzles A, X, Y and Z, (A) clean, (B) coated. ●——● Nozzle A, *Ra* 1·6 μm. ○— —○ Nozzle X, *Ra* 0·8 μm. △- - -△ Nozzle Y, *Ra* 0·15 μm, □—·—□ Nozzle Z, *Ra* 0·35 μm.

Comparison of compression and ejection stresses measured over the range of *Pr* used for each nozzle (not presented) showed that small or zero stresses were recorded at $Pr = 0{\cdot}06$ but at $Pr = 0{\cdot}28$ and 0·46 very large stresses were observed indicating the compaction of powder mentioned above. Between these extreme *Pr* values, intermediate stresses were observed; nozzle A produced the highest and most variable stress values of the coated nozzles and generally nozzle Z had the lowest measured values of compression and ejection stress. Stresses for nozzles X and Y were slightly more variable than for Z.

Compaction of powder on the nozzle wall at *Pr* values greater than 0.2 was confirmed by calculation of the weight of powder per unit length coating the nozzle wall which showed a considerable increase above this setting.

Extrapolation of values of ϕ for the resurfaced nozzles from the determinations using the flat plates for size fraction D gave the following values of ϕ:

Nozzle	*Ra* (μm)	$\phi_{extrap.}$	
X	0·80	>30 °	(Roughest plate *Ra* =
Y	0·15	21·7	0·6 μm)
Z	0·35	25·6	

Capsule filling studies indicated that all the resurfaced nozzles gave higher, more uniform fill weights with less measured stress than the original nozzle A. Of the resurfaced nozzles, nozzle Z ($Ra = 0{\cdot}35$ μm), $\phi_{extrap.} = 25{\cdot}6$ °, produced slightly better fill weight uniformity (with lower measured stresses) than X and Y. This supports the idea of an optimum angle of wall friction for powder retention (and hence uniformity of fill weight) with minimum stress. The modification of the value of ϕ with powder coating should be borne in mind, since ϕ has been shown to increase with repeated powder movement of a metal surface (Jolliffe & Newton 1980).

Conclusions

The production of identical surface textures on flat friction plates and the cylindrical inside bore of the dosator nozzle is not readily achieved. A lapping method was selected as most acceptable. Powder wall friction studies showed that very rough surfaces have similar values of ϕ probably due to the predomination of powder-powder over powder-wall friction. Surfaces prepared by different methods, but having similar *Ra* values, may produce different values of ϕ because of differences in the shapes of the surface features (not accounted for by *Ra* values). Values of ϕ for resurfaced nozzles were extrapolated from wall friction determinations with the flat plates.

Capsule filling with resurfaced nozzles showed an increase in fill weights and uniformity with reduced stresses compared with the original nozzle. One lapped nozzle proved to slightly exceed the others in this improvement in filling performance, supporting the concept of an optimum angle of wall friction for powder retention (and hence uniformity of fill weight) with minimum stress.

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REFERENCES

BS 1134: 1972 Part 1 'Method for assessment of surface texture'.

Jolliffe, I. G. (1980) PhD Thesis, University of Nottingham

Jolliffe, I. G., Newton, J. M. (1980) Acta Pharm. Technol. 26: 324–326

Jolliffe, I. G., Newton, J. M., Walters, J. K. (1980) Powder Technol. 27: 189–195

Jolliffe, I. G., Newton, J. M. (1982a) J. Pharm. Pharmacol 34: 293–298

Jolliffe, I. G., Newton, J. M. (1982b) Ibid. 415–417

Jolliffe, I. G., Newton, J. M., Cooper, D. (1982) Ibid. 230–235

Takagi, K., Sugihara, M., Kimura, S. (1969) Yakuzaiguku 29: 245–249