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An investigation of the relationship between particle size and compression during capsule filling with an instrumented mG2 simulator

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An instrumented mG2 capsule filling machine simulator has been employed to study the effects of the amount of compression (compression ratio) on the capsule fill weight uniformity and measured compression and ejection stresses. Four size fractions of lactose were studied (mean particle sizes 15·6, 17·8, 37·5 and 155·2 μm). The range of compression over which satisfactory filling could be achieved was large for fine, cohesive powders but decreased with increasing particle size. The lower limit of filling ability was the ability to retain the powder and the amount of compression needed to achieve retention increased with increasing particle size. The upper limit on compression, was the compaction of the powder which prevented the piston acting to cause retention. Large particle sizes were able to undergo only a small change in volume before compaction occurred whilst fine, cohesive powders were considerably more compressible and hence could be filled satisfactorily at higher compression settings.

The particle size of a powder has an important influence on the flow properties of a bulk powder, and for a given material, the smaller the particle size the less free flowing, i.e. the more cohesive, is the powder. Jolliffe et al (1980) have shown theoretically that the ability of a powder sample to be retained within a dosator nozzle of a capsule filling system is related to the flow properties of the powder. This arises from two factors: (i) the ability to form a stable arch at the exit to the nozzle and (ii) the transmission of the compressive stress applied to induce retention. According to the theoretical approach of Jolliffe et al (1980), cohesive powders require only small amounts of compression to be retained by the dosator nozzle during the powder transfer stage of the filling process. Free flowing powders, however, need larger compressive stresses to achieve powder retention and hence uniform fill weights. In addition to reflecting the poor stress transmission in powder beds where interparticulate friction is low (i.e. free flowing powders), this also relates to the interaction between the powder and the nozzle wall surface. For a given surface texture, the angle of wall friction has been shown to be greater for cohesive powders which, as a result, are more readily retained (Jolliffe & Newton 1982). Simple retention experiments have supported these theoretical predictions (Jolliffe & Newton 1982). The current work extends these studies to conditions which reflect those of production, i.e. the use of a simulator.

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The design and method of use of an mG2 capsule filling simulator has been described (Jolliffe et al 1982). This machine employs an mG2 G36 filling turret, but has a modified drive mechanism which moves the dosator nozzle vertically only, rather than causing it to rotate as on production machines. A mechanical linkage positions a rotating powder feed bed under the nozzle for picking up the powder and later in the cycle, moves it away to expose a capsule shell into which powder can be ejected. This simplification of dosator nozzle motion allows it to be instrumented. Three measurements are possible from this instrumentation:

(1) Compression and ejection stresses exerted by the piston on the powder within the dosator nozzle. These are measured by strain gauges attached to the piston.

(2) Movement of the piston relative to the nozzle during compression and ejection (measured by a distance transducer).

(3) Movement of the whole dosator nozzle up and down through its cycle. This is also measured by a potentiometer type distance transducer.

The use of this machine to relate these measurements to capsule filling performance, using certain size fractions of crystalline lactose is described here. In particular, the relationship of compression stress to fill weight uniformity is investigated.

MATERIALS AND METHODS

Four size fractions (A, B, D and H) of a sample of DMV lactose-125 classified into eight size fractions as described previously (Jolliffe & Newton 1982) were used. These had mean volumetric diameters of (A) 15·6 μm, (B) 17·8 μm, (D) 37·5 μm and (H) 155·2 μm. Before use, these were passed through a suitable size sieve to break down any agglomerates.

Method

The basic method of use of the mG2 simulator has been described elsewhere (Jolliffe et al 1982). In these experiments capsules were filled over a range of compression settings for each particle size. Capsule contents were weighed and correlated with the compression and ejection stresses measured. Results were obtained for filling with a freshly cleaned nozzle and one which had achieved a constant coating of powder, after a period of filling at that particular compression setting. The running time to achieve a constant coating of powder on the nozzle was established at 15 min for the powder samples used here.

The results are presented as mean fill weights, compression and ejection stresses (and their respective variances) as a function of compression ratio, *Pr* (change in height of powder with compression divided by the original powder height, Takagi et al 1969).

RESULTS AND DISCUSSION

The results for each particle size will be discussed individually before a final comparison is made. Only results from the use of the coated nozzle will be considered since these match the normal operation most closely.

Size fraction A (15·6 μm)

Three principal problems were encountered during the use of this fine powder:

(i) Its tendency to aggregation was detrimental to feed bed uniformity.

(ii) High feed bed porosity means that the piston in the dosator nozzle tends to push through the powder before it is sufficiently compressed to undergo further compression. This can result in powder being trapped behind the piston tip.

(iii) Because the powder has a high porosity and is therefore highly compressible, stresses at low compression settings (where piston movement is small) can be too small to measure.

However, the following observations were made during filling with this size fraction:

The mean fill weights and the fill weight variance are plotted as a function of *Pr* in Fig. 1. (Since inclusion of zero fill weights has a marked effect, points where they have been excluded are also shown.) Mean fill weights decrease gradually as compression is increased. This is unlikely to be due to retention problems (except at *Pr* = 0·84) since no zero or very low fill weights occur. This is reflected in

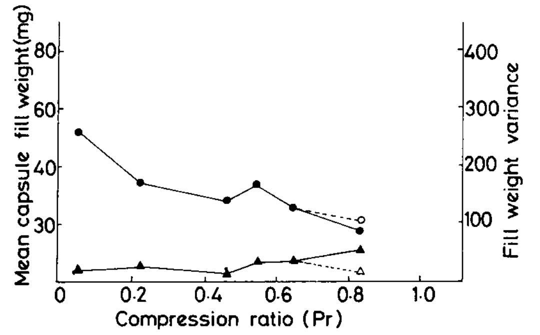

FIG. 1. Mean capsule fill weight and fill weight variance as a function of simulator compression ratio. Size fraction A (15·6 μm). Nozzle coated. ● Mean fill weight. ▲ Fill weight variance. ○, △ As above but excluding zero fills.

the fill weight variance which increases only slightly up to *Pr* = 0·84. The gradual decrease in fill weight is likely to result from powder losses behind the piston. The changes occurring at *Pr* = 0·84 are explained when Fig. 2 is examined. Both the mean compression and ejection stresses remain very small (and have only small variances) until *Pr* = 0·84 where they become comparatively very large.

It therefore appears that, at *Pr* = 0·84 either compaction of the powder was occurring on the nozzle walls, jamming the piston and preventing it

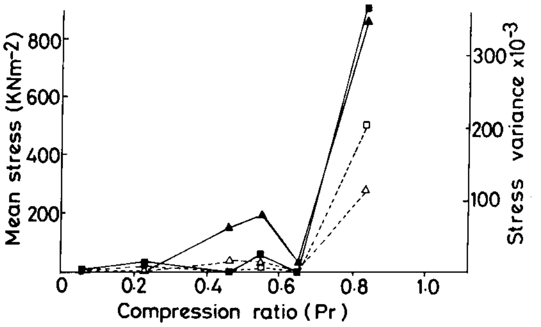

FIG. 2. Compression and ejection stress as a function of simulator compression ratio. Size fraction A (15·6 μm). Nozzle coated. ■——■ Mean compression stress. □ - - □ Compression stress variance. ▲——▲ Mean ejection stress. △ - - △ Ejection stress variance.

from compressing or ejecting powder, or the whole powder plug was compacted and could not be removed by the ejection stress exerted by the piston. In either case, zero fill weights and large values of stress would be recorded.

When the weight of powder coating per unit length of nozzle is plotted as a function of *Pr* (Fig. 3), it can be seen that, although there is a slight increase in powder coat weight with increasing *Pr* up to 0·84, at *Pr* = 0·84 a considerable increase occurs. This supports the idea of compaction occurring at this high compression setting.

Size fraction B(17·8 μm)

The mean particle size of this fraction is only slightly greater than that for size fraction A but the values of the Jenike flow factor differ significantly. The problems mentioned earlier are evident, although to a lesser extent, and compared to samples D and H, the material was relatively cohesive. As is the case for size fraction A, the mean fill weight decreases with increasing compression ratio; this decrease becomes steeper at *Pr* = 0·54, 0·63 and 0·80. This is likely to be due to losses behind the piston since the fill weight variance remains, suggesting there are no

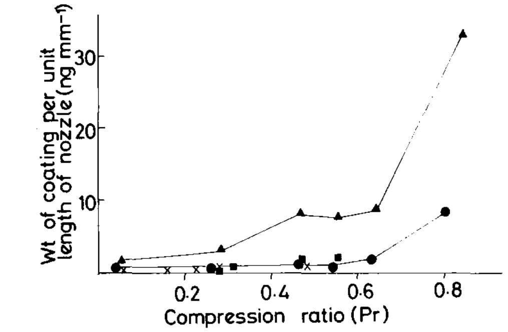

FIG. 3. Weight of powder coating per unit length of nozzle as a function of simulator compression ratio for size fractions A, B, D and H. Size fraction (μm): ▲ = A 15·6; ● = B 17·8; X = D 37·5; ■ = H 155·2 μm.

retention problems. The mean compression and ejection stresses are similar to those for size fraction A. They are minimal up to *Pr* = 0·63 when they increase slightly before a sharp increase at *Pr* = 0·80. Examination of Fig. 3 shows that at *Pr* = 0·63 the weight of powder coating the nozzle also increases slightly before increasing steeply at *Pr* = 0·80. This suggests that the powder is beginning to be compacted at *Pr* = 0·63 and this compaction is fully effective at *Pr* = 0·80.

Size fraction D (37·5 μm)

A value for the Jenike flow factor could not be obtained for this sample, and hence on this classification the powder would be considered to be free flowing. Simple flowability tests (Jolliffe 1980) indicated that it was slightly less free flowing than size fraction H.

The mean fill weights and their variances are plotted as a function of *Pr* in Fig. 4. It will be observed that the largest fill weight is achieved at *Pr* = 0·17 and that this setting also gives the lowest fill weight variance. (The variance at *Pr* = 0·06 is distorted by the inclusion of zero fill weights. The effect of excluding these is shown. At *Pr* = 0·06 capsules were either completely filled or empty, whereas at higher settings partial fills were obtained).

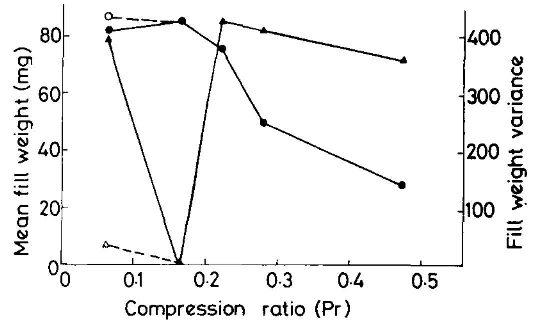

FIG. 4. Mean capsule fill weight and fill weight variance as a function of simulator compression ratio. Size fraction D (37·5 μm). Nozzle coated. ● Mean fill weight. ▲ Fill weight variance. ○, △ As above but excluding zero fills.

In Fig. 5 the mean compression and ejection stresses (and their respective variances) are plotted as a function of *Pr*. These show a similar trend to the fill weight variation. The smallest compression and ejection stresses are recorded at *Pr* = 0·06, as would be expected. Stresses measured for *Pr* = 0·17 are also extremely small and uniform but at *Pr* = 0·22 both stresses are in the region of 1000 kNm^2.

For compression ratios above 0·22 both the stresses (and their variances) increase considerably and values off the recorder scale (i.e. greater than 8000 kNm^{-2}) were observed.

These results demonstrate that at low values of *Pr* there was insufficient compression stress exerted by the piston to ensure that the powder was regularly retained, especially when a clean nozzle was used. The improved fill uniformity as the nozzle becomes coated is likely to result from an increase in angle of powder-wall friction, as discussed elsewhere (Jolliffe & Newton 1980). At high values of *Pr* poor

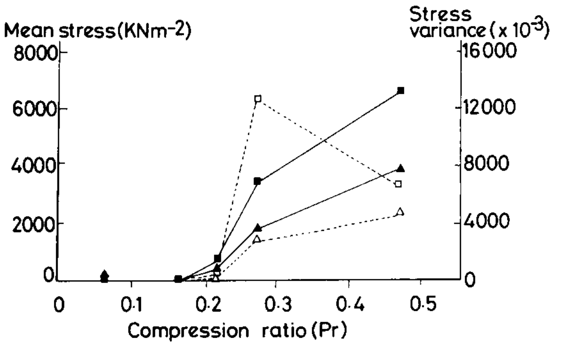

FIG. 5. Compression and ejection stresses as a function of simulator compression ratio. Size fraction D (37·5 μm). Nozzle coated. ■——■ Mean compression stress. □ - - □ Compression stress variance. ▲——▲ Mean ejection stress. △ - - △ Ejection stress variance. ↑ Includes value exceeding recorder scale.

uniformity of fill again occurs. However, in this case, the large and erratic values of stress recorded suggests that powder was compacted on the nozzle walls jamming the piston. This prevents the piston exerting sufficient compression on the powder for it to be retained (although large values of compression and ejection stress are recorded). At $Pr = 0{\cdot}17$ the compression applied was sufficient to achieve retention of the powder without it becoming compacted on the nozzle wall.

The compaction of powder on the nozzle wall was apparently confirmed by Fig. 3 which shows the weight of powder coating per unit length of nozzle as a function of *Pr*. The powder coating the nozzle wall was a minimum at $Pr = 0{\cdot}17$. At $Pr = 0{\cdot}22$ it had increased slightly but increased considerably at $Pr = 0{\cdot}28$ and 0·47. This suggests that a much thicker, denser powder coating was built up on the nozzle wall at these high values of *Pr*.

Size fraction H (155·2 μm)

This powder was free flowing and did not produce the problem associated with the use of fine cohesive powders discussed earlier.

Fig. 6 shows that no retention is possible below $Pr = 0{\cdot}17$, but that between $Pr = 0{\cdot}28$ and 0·36 high fill weights with low weight variance are achieved. Above this setting, i.e. for $Pr = 9{\cdot}47$ and 0·55 fill weights decrease and variance increases considerably.

Stresses measured during the filling of this size fraction (Fig. 7) are zero for settings where no fill occurs and increase steeply from $Pr = 0{\cdot}28$ to 0·31. Compression settings giving $Pr = 0{\cdot}36$, 0·47 and 0·55 result in compression stresses which are frequently off the recorder scale and generally very high ejection stresses. These stresses also exhibit a high variance, which examination of individual results shows, is due to erratic stresses and fill weights being recorded.

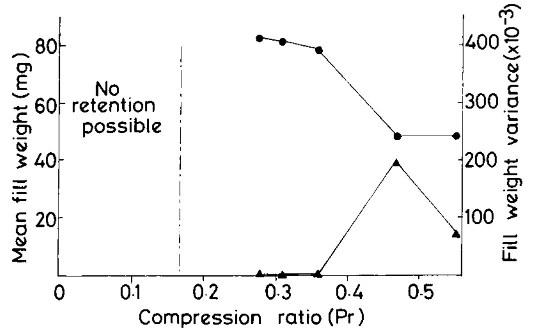

FIG. 6. Mean capsule fill weight and fill weight variance as a function of simulator compression ratio. Size fraction H (155·2 μm). Nozzle coated. ● Mean fill weight. ▲ Fill weight variance.

This indicates that compaction of the powder on the nozzle wall occurred over this range of *Pr*, causing the piston to jam and resulting in poor powder retention with large stresses on the piston. The weight of powder coating the nozzle (Fig. 3) increases at $Pr = 0{\cdot}32$ from virtually zero at $Pr = 0{\cdot}28$ with a further gradual increase at higher values of *Pr*. This also suggests compaction begins to occur at $Pr = 0{\cdot}32$.

GENERAL DISCUSSION

Principally, the results show that the range of compression ratios over which uniform fill weights can be achieved with minimum stress requirements is much greater for fine, cohesive powders than for the

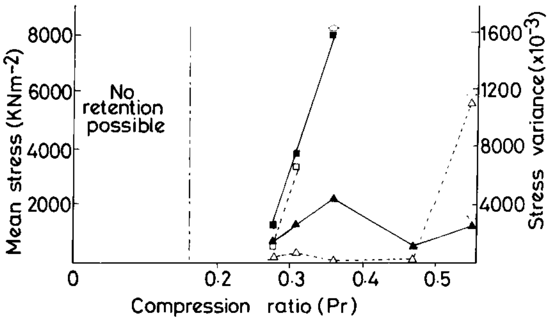

FIG. 7. Compression and ejection stresses as a function of simulator compression ratio. Size fraction H (155·2 μm). Nozzle coated. ■——■ Mean compression stress. □ - - □ Compression stress variance. ▲——▲ Mean ejection stress. △ - - △ Ejection stress variance. ↑ Includes value exceeding recorder scale.

coarser, free flowing samples. This is summarised diagrammatically in Fig. 8.

Size fractions A and B (15·6 and 17·8 μm) can be retained with little or no compression in the coated nozzle giving uniform fill weights. These fractions have been shown to give larger angles of wall friction for a given wall surface than the free flowing size fractions (Jolliffe & Newton 1982). This provides support for the powder, reducing the amount of compression required to ensure retention. Consequently, free flowing powders having less frictional wall support require larger compression stresses to be retained.

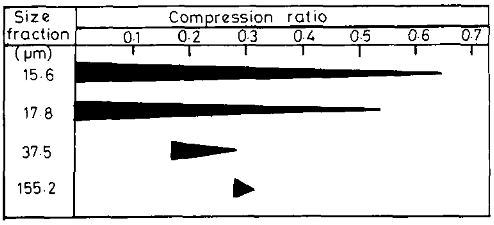

FIG. 8. Range of compression ratios over which fill weight uniformity is satisfactory for the size fractions studied. ▶ zone over which satisfactory capsule filling is feasible. Widest part of bar indicates the best conditions.

The value of the compression ratio at which the weight uniformity begins to deteriorate, is associated with the compaction of the powder at the nozzle wall which, in turn, is particle-size dependent. Cohesive size fractions A and B are able to undergo large volume changes and compaction will occur only at very high compression values. However, the free flowing powders D and H are much less compressible and hence compaction occurs once a certain volume reduction is exceeded. Bulk density studies on these powders by observing the volume changes of powder held in a tapped measuring cylinder (Jolliffe 1980) have shown that the larger particle sizes undergo only a small increase in bulk density between the loose and tightly packed states but loosely packed smaller particle size increase in bulk density considerably on tapping.

Generally, the highest most uniform fill weights with the smallest measured compression and ejection stresses are obtained when the minimal amount of compression that produces retention is employed. This amount of compression apparently depends on the powder flow properties (resulting from the particle size) as predicted by the theory of Jolliffe et al (1980).

Conclusions

The range of the compression ratios over which uniform fill weights can be achieved with minimum stress in a dosator nozzle system depends on the particle size; fine lactose size fractions produce uniform fill weights over a wide range of compression settings but, as particle size increases the range over which satisfactory filling is possible decreases.

Powder retention ability provides the lower limit of the range and the amount of compression to cause retention increases with increasing particle size. The upper limit on filling ability is the compaction of the powder. This is higher for fine cohesive powders since they are able to undergo a greater volume reduction than coarse free flowing powders.

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