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Capsule filling studies using an mG2 production machine

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A production mG2 G36 machine has been employed to study the effects of compression on the capsule filling properties of four particle size fractions of lactose having a range of flow properties. The effect of the surface texture of the dosator nozzle bore on capsule filling is also investigated. Fine, cohesive powders gave uniform fill weights over a whole range of compression settings but as increasingly free-flowing powders were used, this range diminishes. For both types of powder, the upper limit on compression is set by compaction of powder which produces poor fill weights; coarse, free-flowing powders, which are less compressible, compact at lower compressions. Free-flowing powders, in particular, also require a minimum compression to be retained. Resurfaced nozzles produced improved capsule filling. One nozzle surface produced slightly more uniform fill weights and was unaffected by powder coating of the nozzle suggesting that an optimum surface texture exists for capsule filling. The results are similar to those obtained using the mG2 simulator and hence validate the latter's use in studying production capsule filling.

Recently reported studies of the automatic filling of hard gelatin capsules using the continuous motion dosator nozzle principle have employed a specially constructed mG2 simulator (Jolliffe et al 1982). Those experiments are repeated here using a production mG2 machine to test the applicability of the results obtained with the simulator to production machines.

The mG2 simulator was designed so that the dosator nozzle movement was confined to the vertical plane, enabling instrumentation to be attached. This means that, unlike production machines, the lower part of the filling turret holding the dosators does not rotate and a special mechanism was required for positioning the rotating powder feed tray. Experiments conducted using lactose size fractions showed that powder retention and hence capsule fill weight uniformity was affected by the amount of compression applied by the piston during the filling process. Free-flowing powders were more sensitive to this than the more compressible, cohesive powders. Free-flowing powders required the application of a certain amount of compression to be retained and both types of powder had a maximum compression limit where compaction of powder prevented regular retention (Jolliffe & Newton 1982). Further work studied the effect of angle of wall friction between the powder and the nozzle wall surface on retention ability and capsule filling (Jolliffe & Newton 1983). This indicated that the wall surface texture affected fill weight uniformity. We have now used a production mG2 machine to test the validity of results obtained with the mG2 simulator.

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MATERIALS AND METHODS

Materials

Size fractions from a sample of DMV lactose, 125 mesh originally, split into eight size fractions were used. These had mean volumetric diameters as follows (A) 15·6, (B) 17·8, (D) 37·5 and (H) 155·2 μm (determined using a Model TA Coulter Counter). Before use, the powders were passed through a suitable sized sieve to break up any aggregates.

Description of mG2 production machine

The machine used was an mG2 model G36 (mG2 Bologna, Italy). In this machine, capsules travel in bushes on a continuous chain that passes around five turrets which: (a) rectify and feed capsule shells into the chain, (b) remove the caps from the bodies, (c) fill the bodies with powder (filling turret), (d) replace the caps on the bodies, (d) close the capsules to the required length and, finally, eject the filled capsules. Whilst newer versions of the mG2 machines do not employ the chain mechanism for carrying the capsule, the nozzle design is the same.

The dosator nozzles on the filling turret rotate with the lower part of the turret whilst cams in the top part of the turret, which raise and lower the dosator and/or the piston at appropriate points, are stationary. (This arrangement is reversed in the simulator.) Compression adjustments are made in a similar way to the simulator i.e. by adjusting the height of the compression cam which operates while the nozzle is dipping into the powder bed, causing the piston to move down to compress the powder. Up to twelve dosators may be fitted to the filling turret, each one performing a different operation at a given time in a similar manner to a rotary tablet press.

The powder feed tray has an annular shape as on the simulator, although it accommodates nearly twice the weight of powder. It is driven via gearing from the main machine drive such that it has a different centre of rotation and speed to that of the filling turret. The relative speeds of the two components are such that when the nozzle enters the powder there is no relative motion between the two. Ejection of the powder occurs after approximately 180° rotation of the filling turret. At this point the feed tray (being offset and of a larger radius) does not cover the bush carrying the capsule body, allowing the dosator nozzle to be lowered over a capsule body and collect the ejected powder. Although the powder mixing and levelling device differs from that on the simulator, bulk densities achieved are similar (Table 1). Powder is filled into the feed tray from a vibratory feeder via a covered chute.

Table 1. Differences between mG2 production machine and simulator.

Parameter	mG2	Simulator
(1) Dosator nozzles	Rotating	Stationary
(2) Turret rev min^{-1}	40	30
(3) Zero compression	$Cm = -1{\cdot}5$	$Cm = 0{\cdot}0$
(4) Gap between nozzle and feed tray base	0·41 mm	0·1 mm
(5) Feed tray volume ratio	2	1
(6) Leveller mixer shape		
(7) Feed bed bulk densities g cm^{-3}		
(i) size fraction D	0·787	0·757
(ii) size fraction A	0·418	0·430
(iii) size fraction B	0·608	0·571
(iv) size fraction H	0·855	0·887

Although the machine has 12 nozzles available on the filling turret, only one nozzle was attached at any one time. This allows a study of a particular filling nozzle and also the ability to use a nozzle with different surface texture. Only one size of nozzle was used, namely a size 3 to allow comparison with those used in the previous studies employing this simulator.

General method

Filling conditions were kept as close as possible to those used for the simulator, however, some differences were avoidable; there are listed in Table 1. The slowest turret speed of 40 rev min^{-1} was used and the powder feed bed depth was adjusted to 10·7 mm as used on the simulator.

Since no distance transducers are fitted to this machine, the compression was adjusted to points on the gauge fitted to the machine. Generally, integer values were used to make adjustment simpler and more accurate. This compression setting was given the symbol *Cm*. Values of $Cm = -1{\cdot}5$ (no compression movement of the piston) to $Cm = 6{\cdot}0$ were used. (These approximately corresponded to $Cm = 0{\cdot}0$ and 8·0 respectively on the simulator gauge).

Sufficient powder was weighed out to fill the feed tray and poured into the vibratory feeder. After filling, the feed tray was allowed to rotate five times and the bulk density of the powder determined using the sampling technique described for the simulator (Jolliffe et al 1982). A further five revolutions followed to ensure mixing.

When the feed bed had been prepared, the capsule feed supply (modified so that shells were only fed into positions coinciding with the one dosator nozzle) was switched on and the machine run until empty capsule shells were ejected. Before fitting, the dosator nozzle was cleaned in water and carbon tetrachloride (to remove both lactose and grease).

Capsule filling was then carried out with each capsule being collected consecutively (with the aid of self-adhesive tape) for numbering and weighing. The contents of 50 capsules were weighed for each condition.

Treatment of results

Mean fill weights and their variance were calculated for the first 20 capsules of each sample (only 20 were used for valid comparison with the simulator results). These values were plotted as a function of *Cm* since the compression ratio could not be calculated without distance transducer data.

RESULTS AND DISCUSSION

Effect of compression setting on capsule filling performance

All four particle size fractions of lactose were used with a range of compression settings. Standard production nozzle A was used.

Particle size fraction A (15·6 μm)

The mean fill weights and their variance underwent little further change after running times exceeding 15 min. This time was therefore chosen as the 'running-in' time to produce a constant powder coating on the nozzle bore surface, i.e. a coated nozzle. Similar fill weights were obtained for both clean and coated nozzles with slightly larger values of variance for the coated nozzle. Results for the coated nozzle are shown in Fig. 1. A gradual decrease in fill weight is seen from $Cm = -1·5$ to $Cm = 6·0$, above which the decrease is much greater. There is apparently little change in variance. Highest fill weights are achieved when no compression is applied and they decrease at higher settings probably as powder is lost (behind the piston). At $Cm = 6·0$ to 8·0 powder is compacted on the nozzle wall, preventing the piston moving and resulting in a higher proportion of zero fill weights. These observations are similar to those seen for the simulator (Jolliffe & Newton 1982).

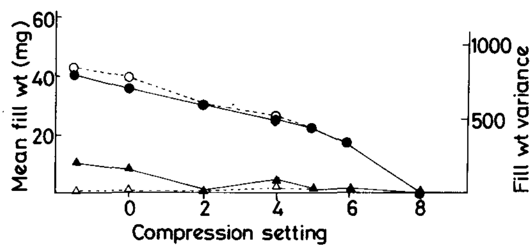

FIG. 1. Mean capsule fill weight and fill weight variance as a function of production machine compression setting (*Cm*). Size Fraction A. Nozzle A coated. ● = Mean fill weight. ▲ = Fill weight variance. ○, △ as above but excluding zero fills.

Particle size fraction B (17·8 μm)

As for size fraction A, a time to constant coating of 15 min was established and fill weights and variance for the coated nozzle A (Fig. 2) show a similar trend to size fraction A. Highest fill weights were achieved at the lowest compression settings, whereas at high compression settings low fill weights result from the piston jamming in powder compacted in the nozzle. Higher fill weights are seen than with size fraction A reflecting the slightly less cohesive nature of this powder. The different flow properties of B are also shown by the sharpness of the fall in fill weights with increasing *Cm*, i.e. a sudden decrease at $Cm = 6·0$. These results also follow a similar trend to those obtained for the simulator.

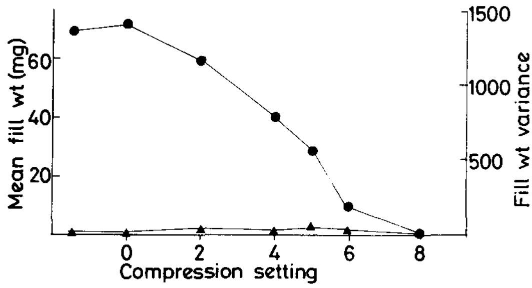

FIG. 2. Mean capsule fill weight and fill weight variance as a function of production machine compression setting (*Cm*). Size Fraction B. Nozzle A coated. ● =Mean fill weight. ▲ = Fill weight variance.

Particle size fraction D (37·5 μm)

Fill weight results for size fraction D using a constantly coated nozzle A are presented in Fig. 3. Generally, higher fill weights are seen at lower compression settings with high uniformity at $Cm = -1·5$ and 1·0. (At $Cm = 0·0$ an unexpected result is obtained compared with those for other nozzle surfaces as discussed later). Low fill weights are recorded above $Cm = 7·0$, again probably due to powder compaction. This is especially prevalent at $Cm = 4·0$ where a large number of zero fill weights were measured. This powder is retained at low compression stresses, but is more sensitive to compaction at lower compression settings than size fractions A or B as seen in experiments with the simulator (Jolliffe & Newton 1982).

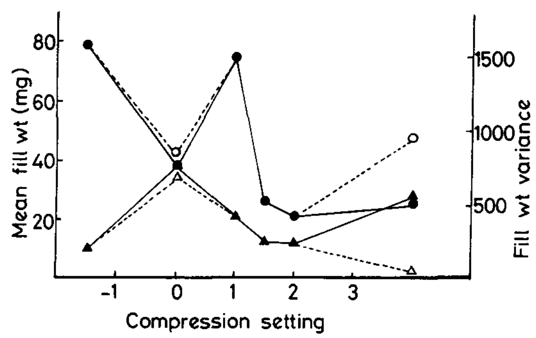

FIG. 3. Mean capsule fill weight and fill weight variance as a function of production machine compression setting (*Cm*). Size Fraction D. Nozzle A coated. ● = Mean fill weight. ▲ = Fill weight variance. ○, △ as above but excluding zero fills.

Particle size fraction H (155·2 μm)

Fig. 4 shows that size fraction H could not be retained in a constantly coated nozzle when no

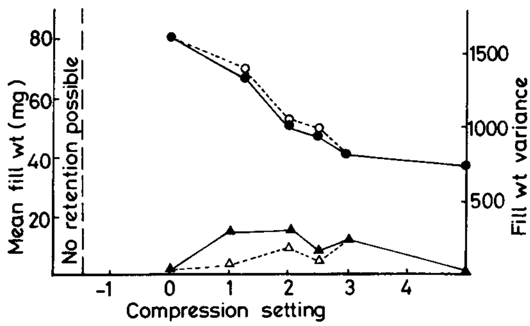

FIG. 4. Mean capsule fill weight and fill weight variance as a function of production machine compression setting (*Cm*). Size Fraction H. Nozzle A coated. ● = Mean fill weight. ▲ = Fill weight variance. ○, △ as above but excluding zero fills.

compression (*Cm* = −1·5) was applied, but application of a small amount of compression, *Cm* = 0·0, gives the highest fill weights with the lowest variance. Above *Cm* = 0·0 fill weights decreased and variance increased except for the low fill weights at *Cm* = 5·0. These results can be interpreted in a similar way to those for the simulator, i.e. size fraction H requires a certain amount of compression to ensure retention but slightly greater compression causes decrease in fill weight due to powder compaction.

General discussion

The patterns of fill weights observed for these size fraction using this production machine are similar to those obtained using the simulator. Fine, cohesive powders give uniform fill weights over a wide range of compression settings but with increasingly free-flowing powders this range decreases. Compaction of powder at high compression results in poor filling and, since free-flowing powders are less compressible, this occurs at a lower compression setting for this type of powder. Free-flowing powders, in particular, require a minimum compression to be applied to ensure powder retention within the nozzle and hence regular filling. Between these two limiting factors the most uniform fill weights are achieved.

Fill weights are slightly higher using the production machine than those obtained with the simulator. This may result from the slightly higher feed bed bulk densities or the rotation of the nozzle aiding retention by exerting another force ('centrifugal') on the powder other than gravity.

When compression settings (*Cm* values) for simulator and production machines were compared with the events occurring, it was apparent that there was a difference of 1 to 1·5 units between the two compression scales.

Capsule filling using the resurfaced nozzles

In addition to the standard production nozzle A, three nozzles, X, Y and Z, whose bore surfaces had been modified by lapping, were employed. (The surface texture of the nozzles can be characterized by the *Ra* values of 1·6 (A), 0·8 (X), 0·15 (Y) and 0·35 (Z) μm respectively. Their method of preparation and use with the mG2 simulator has been discussed previously (Jolliffe & Newton 1983). Particle size fraction D was used in these experiments. For all four nozzles in clean and powder-coated states the highest fill weights were obtained at *Cm* = −1·5 and 1·0 (Fig. 5A, B). Above this, fill weights decrease due to powder compaction as discussed earlier. Nozzle A gives the lowest fill weights between *Cm* = −1·5 to 1·0. Although mean fill weights for all the resurfaced nozzles are similar using this range of compression, those recorded for nozzle Z remain apparently unaffected by a nozzle becoming coated with powder whilst those for X and Y undergo a small change.

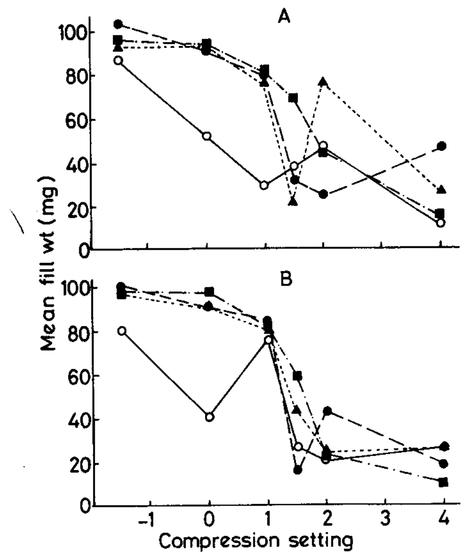

FIG. 5. Mean capsule fill weights as a function of production machine compression setting for size fraction D using nozzles A, X, Y and Z (A) clean and (B) coated. ○——○ = nozzle A, *Ra* (μm) 1·6. ●– –● = nozzle X, *Ra* (μm) 0·8. ▲----▲ = nozzle Y, *Ra* (μm) 0·15. ■---■ = nozzle Z, *Ra* (μm) 0·35.

Plotting capsule fill variance as a function of *Cm* (Fig. 6A, B) shows that variance is increased at high *Cm* settings where compaction occurs. Generally between *Cm* −1·5 to 1·0 variance is low for clean nozzles and increases with coating. However, the variance of nozzle Z does not change significantly as the nozzle becomes coated (over this range of compression settings).

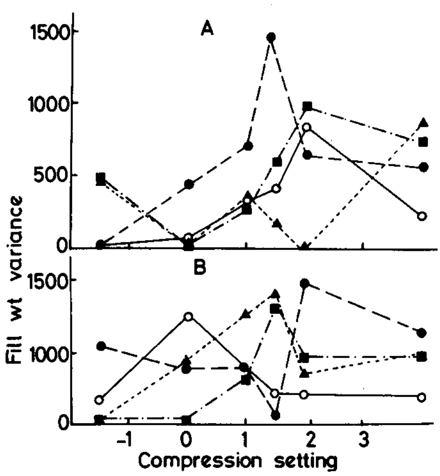

FIG. 6. Capsule fill weight variance as a function of production machine compression setting for size fraction D using nozzles A, X, Y and Z (A) clean and (B) coated. ○——○ = nozzle A, *Ra* (μm) 1·6. ●– –● = nozzle X, *Ra* (μm) 0·8. ▲----▲ = nozzle Y, *Ra* (μm) 0·15. ■–■ = nozzle Z, *Ra* (μm) 0·35.

These results suggest that the resurfaced nozzles are generally able to produce higher fill weights. Nozzle Z appears to produce slightly higher and more uniform fill weights than the others and is apparently unaffected by powder coating the nozzle. Since this nozzle has an intermediate *Ra* value it appears that there is an optimum surface for good filling performance.

These observations agree with those obtained using the simulator where nozzle Z produced the most satisfactory capsule filling.

Conclusions

The results show that capsule fill weights obtained using fine, cohesive powder are relatively insensitive to the compression applied by the piston and are able to give uniform fill weights over a wide range of compression settings. Coarse, free-flowing powders require a minimum compression to be uniformly filled and fill uniformity will only occur for small increases in compression above this. For both types of powder, application of compression which attempts to compress the powder beyond the limits of its ability to pack more tightly by powder rearrangement, will cause powder compaction resulting in poor fill uniformity. This occurs at lower compressions for coarse, free-flowing powders because of their lower compressibility.

Filling experiments employing nozzles with different bore surface textures showed that all the modified surfaces had improved filling ability and that one of these surfaces was slightly better than the others. This indicates surface roughness can affect filling properties and suggests that an optimum surface exists for good capsule filling performance.

Comparison with results obtained with the mG2 simulator shows that observations are similar and hence, for powders of this type, the mG2 simulator gives a valid representation of capsule filling using a production machine.

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