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## Influence of compression setting ratio on capsule fill weight and weight variability

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### Summary

The influence of the compression setting ratio on the capsule fill weight variability has been assessed for a series of particle size fractions of Avicel, calcium carbonate, lactose, maize starch and Starch 1500, on a capsule filling simulator. For most of the systems tested, the most uniform weights were achieved when no compression was applied to the powder during the filling process. The finest particle size fractions of lactose and maize starch do however require some compression to aid powder retention. For the larger particle size fractions, providing high compression settings could induce the piston to jam in the nozzle resulting in failure to fill a capsule at all. The degree of coating of the wall of the nozzle increased with decreasing particle size and increasing compression setting. Lactose produced the greatest degree of coating of the nozzle. For nozzles pretreated by a running in process to induce nozzle coating, most powders behaved in the same manner as those which had started with a clean nozzle. The exception was lactose, which could not be effectively filled using a pretreated nozzle, due to severe binding of powder to the nozzle wall inducing jamming of the piston within the nozzle.

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### Introduction

Hard gelatin capsule filling by the dosator nozzle system requires the accurate dosing, retention and transference of a powder plug from a cylindrical nozzle into an awaiting capsule body. In order to minimise the risk of powder loss during transference, a compressive force may be applied on the piston during dosing to aid retention. Taka-

gi et al. (1969) studied the capsule filling properties of corn starch, lactose, calcium carbonate and calcium phosphate dibasic on a dosator nozzle type automatic machine and found that the coefficient of variation of fill weight was dependent on the compression ratio. They concluded that powder particles should have cohesion but little adhesion to the piston. Studies by Chowhan and Chow (1980) on a Zanasi machine showed that the consolidation ratio (defined as the intercepts of the plots of logarithm of the changes in volume versus the log of the applied pressure) was linearly related to the coefficient of variation of capsule fill weight. Using an instrumented mG2 simulator (Jolliffe et al., 1982), Jolliffe and Newton (1982)

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studied the effects of compression ratio settings on the capsule fill weight uniformity and the compression and ejection stresses for four different lactose size fractions. They found that the range of compression ratios over which satisfactory filling could be achieved was dependent on particle size; fine size fractions produce uniform fill weights over a wide range of compression ratio settings whilst the range over which satisfactory filling was possible decreased with increasing particle size.

The present work seeks to extend the findings of Jolliffe and Newton (1982) to size fractions of five different pharmaceutical excipients (i.e. microcrystalline cellulose, Avicel PH 101, calcium carbonate, maize starch, crystalline lactose and pregelatinized starch, Starch 1500). An instrumented mG2 simulator (similar to that described by Jolliffe et al., 1982) was used to study the influence of compression ratio settings on capsule fill weight and weight uniformity for the different powder systems. The influence of a coated nozzle was also investigated.

## Materials and Methods

Five pharmaceutical excipients i.e. microcrystalline cellulose (Avicel PH101), calcium carbonate, crystalline lactose, maize starch and pregelatinized starch, Starch 1500, classified into different size fractions and characterised as described previously (Tan and Newton, 1990a) were used for the present study. Experiments were carried out on three size fractions of Avicel (A1, A2 and A3), calcium carbonate (C1, C2 and C3), Starch 1500 (S1, S2 and S3), lactose (L1, L2 and L3) and 2 size fractions for the maize starch (M1 and M2). Particle size data relating to these powders are given previously (Tan and Newton, 1990a).

The method used to fill capsules on the mG2 simulator has been described elsewhere (Tan and Newton, 1990a). For the present study, prior to capsule filling, the bulk density of the powder feed bed was determined using a specially constructed sampler to remove samples from different locations of the tray. The bulk density was calculated from the weight of the sample removed and its vol-

ume (determined from the original height of the powder sample and the cross-sectional area of the sampling cylinder). A mean value of bulk density (from five samples) was determined for each powder. The powder tray was then rotated for another 10 revolutions to fill the holes left by the sampler. Powder removed during sampling was not replenished as there was excess powder always trapped within the mixing and levelling device. Details of the capsule filling process are described elsewhere (Tan, 1987). For each powder, capsules were filled over a range compression ratio (Cr) settings where:

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

Experiments were carried out with both 'clean' and 'coated' nozzles. In order to achieve a constant coating on the nozzle for the latter experiments, approximately 500 powder plugs were filled prior to capsule collection. For each experiment, the influence of compression ratio setting on the mean and coefficient of variation of capsule fill weight was examined.

## Results

### *Experiments with a clean nozzle: Effect of compression setting on capsule fill weight and weight uniformity*

#### *Starch 1500*

Fig. 1 shows how the mean and the coefficient of variation (CV) of fill weight varies with increasing compression ratio (Cr) for different size fractions of Starch 1500. It can be seen that the coarser size fractions, S3 and S2 have higher mean fill weights than the finer size fraction S1 at all compression settings. Increasing the compression settings result in slight decreases in the mean fill weights of S3 and S2, but a steeper fall for S1. These observations are reflected in the values of the CV (Fig. 1) where those for S2 and S3 remain low and constant while that of S1 increases quite significantly

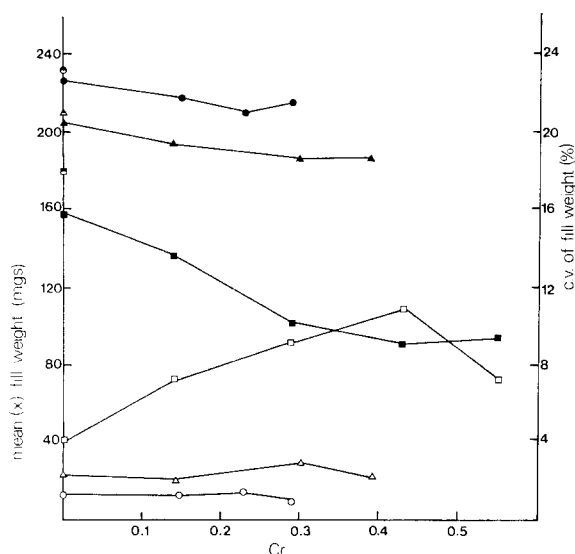


Fig. 1. Capsule fill weight as a function of compression ratio (Cr) for Starch 1500. ('clean' nozzle).

| Powder code | Fill weight |      |             |
|-------------|-------------|------|-------------|
|             | x           | c.v. | Theoretical |
| S3          | ●           | ○    | ●           |
| S2          | ▲           | △    | ▲           |
| S1          | ■           | □    | ■           |

with increasing Cr. For the three powders, slight increases in the mean fill weights are observed at the highest compression settings with corresponding decreases in the CV values. For S2 and S3, the limits of compression settings (at Cr = 0.39 and 0.29, respectively) are reached when the compaction of the powders and overloading of the machine occur. Whilst S2 and S3 show good agreement between the initial observed mean fill weights and the theoretical fill weights (calculated from the powder bed density and fill volume), a significant deviation between observed and expected fill weight was observed for S1.

#### *Avicel PH101*

For Avicel powders (Fig. 2), the highest mean fill weight is exhibited by the fine size fraction A1 and the lowest fill by the coarse size fraction A3. When the values of CV are considered, the highest value is shown by A1 with that of A2 fractionally lower than A3. Increasing the Cr settings has very little influence on the mean fill weights and values

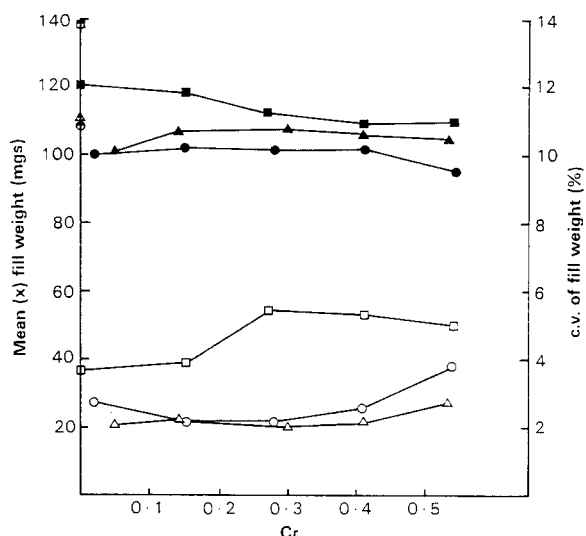


Fig. 2. Capsule fill weight as a function of Cr for Avicel PH 101. ('clean' nozzle).

| Powder code | Fill weight |      |             |
|-------------|-------------|------|-------------|
|             | x           | c.v. | Theoretical |
| A3          | ●           | ○    | ●           |
| A2          | ▲           | △    | ▲           |
| A1          | ■           | □    | ■           |

of CV for A2 and A3 generally, but a small decrease in the fill weights and a corresponding rise in the values of CV are seen at the highest compression settings. In the case of A1, a slight decrease in the mean fill weight and an increase in the CV values is seen at Cr settings 0.15 to 0.27. The mean fill weight and value of CV then remain relatively unaffected by further increases in Cr. The greatest deviation of the observed from the theoretical initial mean fill weight is also shown by A1.

#### *Calcium carbonate*

The results in Fig. 3 show that the coarse size fractions C2 and C3 have much higher mean fill weights than the fine size fraction C1. Increasing the Cr settings results in a sharp fall in mean fill weight of C1 and also to a lesser extent for C2 and C3. For C2 and C3, capsules can only be filled over a limited compression range as over-compaction of the powders and machine overloading prevent experiments being carried out beyond Cr settings

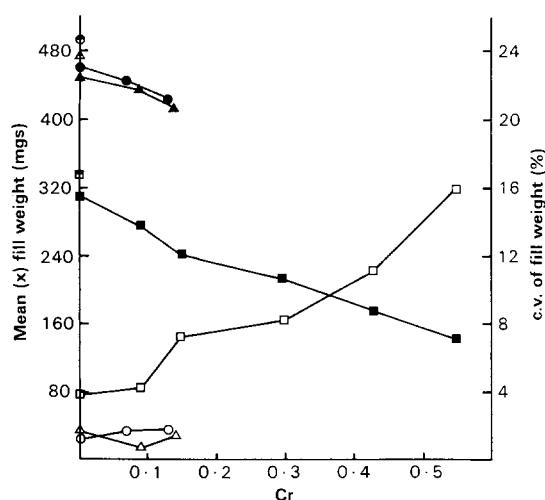


Fig. 3. Capsule fill weight as a function of Cr for calcium carbonate. ('clean' nozzle).

| Powder code | Fill weight |      |             |
|-------------|-------------|------|-------------|
|             | <i>x</i>    | c.v. | Theoretical |
| C3          | ●           | ○    | ●           |
| C2          | ▲           | △    | ▲           |
| C1          | ■           | □    | ■           |

of approx. 0.14. In contrast, C1 may be filled over the whole Cr range without exceeding the compression limit. The trends seen for the mean fill weights are reflected in the results for the values of CV, with C1 showing the highest value and increasing significantly with Cr, while that of C2 and C3 generally remain low and constant over the limited Cr range available. All three size fractions show similar degree of deviation of the observed from the theoretical initial mean fill weights.

#### Maize starch

The graphs for maize starch powders are presented in Fig. 4. A higher mean fill weight and a lower CV value is generally obtained with the coarser size fraction, M2. Increasing the Cr causes a decrease in the fill weight and a slight increase in the CV value. A notable feature here is the presence of some zero fills for M1 at Cr = 0 (i.e. no piston compression). This results in a much lower mean fill weight and an excessively higher CV value than when results are considered excluding the zero fills. M1 also shows a larger deviation of

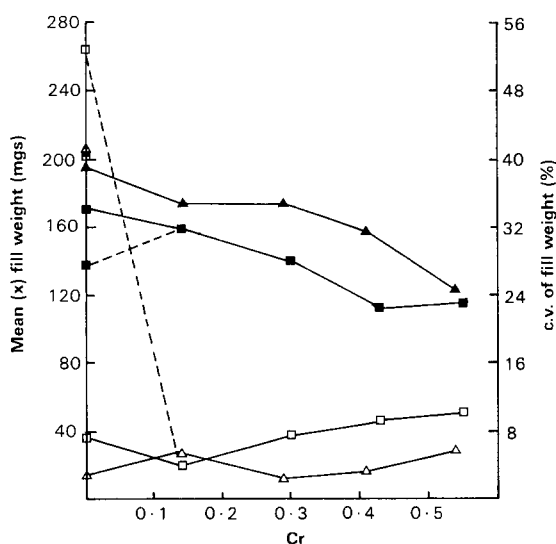


Fig. 4. Capsule fill weight as a function of Cr for maize starch. ('clean' nozzle).

| Powder code | Fill weight |      |             |
|-------------|-------------|------|-------------|
|             | <i>x</i>    | c.v. | Theoretical |
| M2          | ▲           | △    | ▲           |
| M1          | ■           | □    | ■           |

(---) Include values for zero fills.

the observed from the expected initial mean fill weight when compared to M2.

#### Lactose

Fig. 5 shows the results obtained with lactose powders. The highest mean fill weight is generally obtained for the coarse size fraction, L3 and the lowest fill with the fine size fraction, L1. Increasing the Cr settings to 0.15 results in a gradual decrease in the mean fill weight of L3 but a very steep fall is observed at Cr = 0.27. For L2, there is a steady decrease in the mean fill weight with increasing compression to Cr = 0.44. In the case of L1, an initial increase in the mean fill weight at Cr = 0.15 is followed by a steep fall in the fill weights at higher compression settings. For L3 and L2, capsules cannot be filled above Cr settings of 0.27 and 0.44 respectively due to the jamming of the piston inside the nozzle. Whilst L1 can be filled to a higher compression setting, the piston also starts to jam at Cr = 0.55.

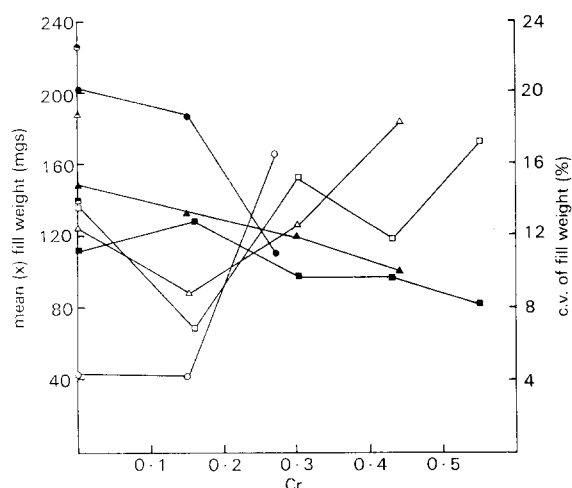


Fig. 5. Capsule fill weight as a function of Cr for lactose. ('clean' nozzle).

| Powder code | Fill weight |      |             |
|-------------|-------------|------|-------------|
|             | x           | c.v. | Theoretical |
| L3          | ●           | ○    | ●           |
| L2          | ▲           | △    | ▲           |
| L1          | ■           | □    | ■           |

### General discussion

Capsule fill weight is dependent on: (i) the fill volume which is a function of the size of the dosator unit and the initial piston height in the nozzle (ii) the powder bed depth and (iii) the bulk density of the powder in the feed tray. Weight uniformity may be influenced by a multitude of factors: poor retention of the powder in the nozzle during ejection of the powder plug from the nozzle; powder coating on the internal wall of the nozzle; loss of powder behind the piston tip during dosing; non-uniformity of the powder feed bed; magnitude of the applied compression stress during dosing; particle size of the powder and its flow properties; wall texture of the nozzle and machine filling speed.

As the fill volume, powder bed depth and machine speed (set at 30 capsules per minute) are constant, the very high initial mean fill weights seen for calcium carbonate powders (Fig. 3) are due to their high powder bed bulk densities (Table 1). In contrast, Avicel powders have much lower bed bulk density values, thus their initial mean fill

TABLE 1

*Bulk densities of the different powders in the feed tray*

| Powder code | Powder bed bulk density<br>$\gamma_b$ (g cm <sup>-3</sup> ) | $\gamma_{b,cv}$ (%) |
|-------------|---|---------------------|
| S3          | 0.626   | 1.31                |
| S2          | 0.569   | 2.11                |
| S1          | 0.485   | 2.82                |
| A3          | 0.294   | 1.14                |
| A2          | 0.300   | 0.21                |
| A1          | 0.378   | 0.97                |
| C3          | 1.334   | 0.87                |
| C2          | 1.283   | 0.57                |
| C1          | 0.913   | 2.47                |
| M2          | 0.557   | 2.58                |
| M1          | 0.550   | 3.96                |
| L3          | 0.609   | 1.82                |
| L2          | 0.504   | 2.55                |
| L1          | 0.374   | 2.93                |

$\gamma_{b,cv}$  = coefficient of variation of powder bed bulk density.

weights are low (Fig. 2), while Starch 1500, maize starch and lactose powders show intermediate bed bulk densities (Table 1) and initial mean fill weights (Figs 1, 4 and 5). For calcium carbonate, Starch 1500, maize starch and lactose powders, bed bulk densities and initial mean fill weights are direct functions of particle size with the coarse size fractions of each excipient (i.e. C3, C2, S3, M2, L3 and L2) showing higher bed bulk densities and mean fill weights than the fine size fractions (C1, S1, M1 and L1). In the case of Avicel, the fine size fraction, A1 has a higher bulk density than the coarser size fractions, A2 and A3 (Table 1), hence accounting for the higher initial mean fill weight of A1. This anomalous bulk density behaviour of Avicel powders has earlier been observed during the loose and tapped bulk density measurements of these powders during tapped consolidation (Tan, 1987).

The decrease in the mean fill weights with increasing values of Cr seen for the various powders (Figs 1–5) is generally associated with the powder loss behind the piston tip and/or its coating on the nozzle wall. For coarser size fractions of Starch 1500 and calcium carbonate (S3, S2, C3 and C2) powder loss behind the piston tip is the main contributory factor to the decrease in mean fill weights with increasing values of Cr. In contrast, the marked decrease in the mean fill weights seen

for the fine size fractions of these excipients (S1 and C1) with increasing values of Cr may be ascribed to a combination of powder losses behind the piston tip and their coating on the nozzle wall.

The low limits to compression for S3, S2, C3 and C2 (at Cr = 0.29, 0.39, 0.13 and 0.14 respectively) are due to the ability of the particles of these powders to rearrange and consolidate down rapidly to their maximum packed states during compression by the piston. Further piston penetrations and volume reductions result in compaction of these powders and machine overloading. In contrast, the upper limit to compression for the fine size fractions of these excipients, S1 and C1, is not reached even at a Cr setting of 0.55. The differences in the compressional behaviour of the powders are primarily due to the variation in their particle size and flow properties (Tan, 1987). The large and regularly shaped particles of S3, S2, C3 and C2 are free flowing and could only undergo small changes in volume (and bulk density) between loose and tightly packed states during consolidation. In fact, for C3 and C2, movements of the powder feed tray and the slight consolidating action of the powder bed leveller are sufficient to cause the particles of these powders to consolidate down rapidly into closely packed uniform beds.

In the case of S1 and C1, the powders consist of small and regularly shaped particles interspersed with a high proportion of very small, irregularly shaped fines which exhibit high cohesive and frictional forces and form agglomerated powder beds with high porosities. Thus particle slippage and rearrangement of these powders do not occur readily even during piston compression. The marked decrease in the fill weights of S1 and C1 with piston compressions are due to powder losses behind the piston tip and their heavy coatings on the nozzle wall. This is confirmed from visual examination of the nozzle and piston after experimentations and from the results presented in Figs 6 and 8. Loss of powders behind the piston tip is facilitated by the small particles of S1 and C1 and their high feed bed porosities which allow the piston to push through these powders before they are sufficiently consolidated to undergo further compressions. For C1 especially, the high proportion of fines and their great tendency to stick (as shown by the large

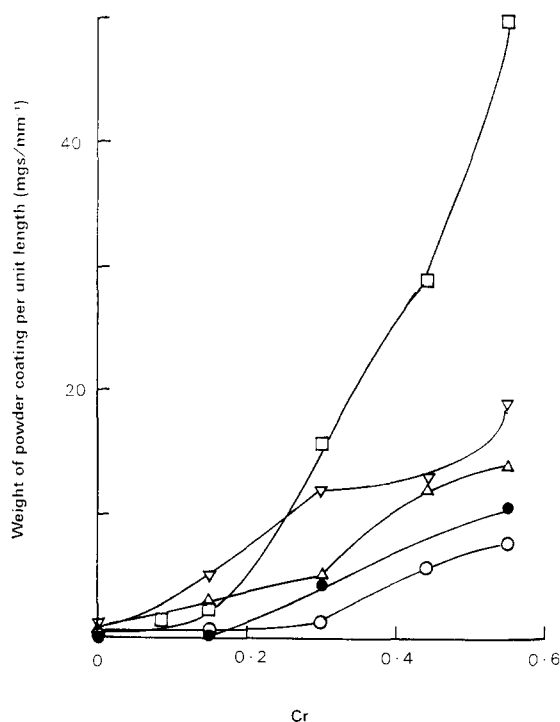


Fig. 6. Weight of powder coating per unit length of nozzle as a function of the compression ratio, Cr, for those powder systems showing greater coating than 2 mg/mm. Calcium carbonate C1 (□), lactose L1 (○), L2 (●), maize starch M1 (▽), Starch 1500 S1 (Δ).

angle of wall friction (Tan and Newton (1990b)) and compaction on the wall surface constitutes a major source of powder loss with increasing compression. The decrease in the fill weights with powder coatings for both C1 and S1 may also be partly explained by the reduction in the nozzle volume due to the coatings. In addition to these factors, the high porosities and variable nature of their powder beds may contribute towards the high values of CV of fill weight seen for these powders.

Whilst a decrease of fill weights of C2, C3, S2 and S3 with increasing Cr values may be similarly explained in terms of their losses behind the piston tip and powder coatings on the nozzle wall, their effects are much less pronounced than those of C1 and S1. The low and constant values of the CV of fill weights are also indicative of the more uniform nature of the powder beds of C3, C2, S3 and S2.

For Starch 1500 powders (S1, S2 and S3), the slight increase in the fill weights and the corresponding decrease in the values of the CV of fill weights at the highest compression setting in each case are probably due to the more compacted nature of the plug, hence less likely powder loss during the transfer and ejection stages.

In the case of maize starch, the decrease in the mean fill weights with increasing values of Cr for M2 and M1 is also associated with increasing powder coatings on the nozzle wall and their losses behind the piston tip. The larger deviation of the observed from the expected initial mean fill weight shown by M1 and its tendency to produce zero fill at  $C = 0$  suggests that its smooth and rounded particles may be poorly retained in the nozzle due to insufficient frictional support provided by the wall as the result of its low angle of wall friction (Tan and Newton, 1990b).

When Avicel powders are considered, it is apparent that the anomalously low bulk density of A3 is caused by the very elongated nature of its particles as is evident from the electron micrographs (Tan, 1987). This results in the powder having a high bed porosity. Slippage and rearrangement of the particles into a more closely packed state may not occur as readily as those of A1 and A2 during consolidation and compression.

For A3, loose powder was initially filled into the capsule bodies at  $Cr = 0.02$ . Increasing Cr settings results in the formation of firmer plugs until very solid ones are formed at  $Cr = 0.54$ . The constant mean fill weights and low CV values observed between Cr settings of 0.15 and 0.41 may be attributed to the good plug formations and the negligible powder losses from nozzle wall coating or its loss behind the piston tip. Fractionally lower fill weights and higher values of CV seen at Cr settings of 0.02 and 0.54 may be due to powder loss outside the vicinity of the capsule body during powder ejection and its loss behind the piston tip respectively. Comparable trends seen with the mean and CV values of fill weight for A2 may be similarly explained.

In the case of A1, there is a greater tendency for the small particles to coat on the nozzle wall or be trapped behind the piston tip and this undoubtedly contributes to the decrease in the mean fill weight

and higher CV. The above factors may also be responsible for the higher initial deviation of the observed from the expected mean fill weight seen with A1.

It is also apparent from Figs 1–5 that coarse size fractions of Avicel (A3, A2) and maize starch (M2) powders are much more compressible than the corresponding size fractions of Starch 1500 (S3, S2) and calcium carbonate (C3, C2) as the compression limits of A3 and A2 have not been reached even at a high Cr setting of 0.54. This again may be related to the differences in the bulk and flow properties of the powders (Tan and Newton, 1990a).

As with other excipients, the high mean fill weight shown by L3 at  $Cr = 0$  is due to its higher powder bulk density (Table 1). The decrease in the mean fill weight at  $Cr = 0.15$  is due to powder coating on the nozzle wall and its loss behind the piston tip. However, unlike other materials discussed so far, one of the major problems encountered during filling of lactose powders is the tendency for these powders to stick and bind onto the nozzle and piston. This is observed with L3 and it becomes quite difficult to detach the nozzle at the end of the experiment. As the compression setting is further increased to 0.27, greater compaction and binding of the powder eventually caused the piston to jam in the nozzle. This prevents the piston from exerting sufficient compression on the powder for it to be retained (as is evident from the very loose powder being filled into the capsules). As piston movement becomes restricted, there is incomplete ejection of the powder into the capsule. Consequently, there is an overall reduction in the nozzle volume available for powder fill. The drastic fall in the fill weight at  $Cr = 0.27$ , may thus be ascribed to a combination of all these factors. Problems encountered during filling of L2 and L1 may be similarly explained but due to their more compressible nature, the piston tends to jam at higher compression settings (at  $Cr = 0.44$  and 0.55 for L1 and L2, respectively).

When the graphs for the CV of fill weight are examined (Figs 1–5), they clearly reflect the problems discussed so far. Very high CV values are observed for each lactose powder when the piston jams or begins to jam in the nozzle (Fig. 5). For L2

and L1, a Cr value of 0.15 appears to be optimal for filling the most uniform capsules. For these powders, high CV values are again seen at  $Cr = 0$  and this may be caused by poor powder retentions and their losses during dosing, transfer and ejection from the nozzle.

It is thus evident from the results presented that generally optimum filling conditions for powders of S3, S2, S1, C3, C2, C1, M2, A3, A2, A1 and L3 are achieved when no piston compression is applied during dosing. In contrast, some compression is required to aid powder retention and achieve uniform fill weights for powders of M1, L2 and L1. Whilst S3, S2, C3, C2, A3, A2, and M2 may also be satisfactorily filled over their optimal compression ranges, large piston compressions are very detrimental to the filling of S1, C1, M1, L3, L2 and L1. In addition to the marked decrease in the fill weights and the large increase in the values of the CV of fill weights, high Cr settings result in the jamming of the piston inside the nozzle when powder of L3, L2 and L1 are used.

#### *Weight of powder coating the nozzle*

Powder coating occurs mainly on the area of the interior nozzle wall after the powder has been compressed and also to some extent on the piston, especially behind the flared end of the piston tip. Since it is impractical to dismantle and weigh the whole dosator after each experiment, the weight of powder coated on the nozzle wall is taken as a guide to assess the material's performance. As the cross-sectional area of the nozzle is constant, results are expressed as the weight of coat per unit length of nozzle wall. The length of compressed powder in contact with the nozzle wall is calculated from the piston displacement (Tan, 1987).

### **Discussion**

Increasing the values of Cr results in the compression and eventual compaction of the particles of S1, C1, M1, L1, L2 and on the nozzle walls (Fig. 6). The decrease in the powder coat for A1 at  $Cr = 0.54$  is probably due to a greater cohesion of the particles when they become more compacted and hence less adhesion to the wall surface. Alternatively,

it may just be an artefact caused by powder loss when the nozzle was detached for weighing. With L3, L2 and L1, greater compactations also result in the binding of these powders onto the nozzle wall. As larger particles of L2 and L3 are less compressible than those of L1, they tend to compact and bind onto the nozzle wall at lower Cr settings. For L3, this starts to occur at  $Cr = 0.15$  and at  $Cr = 0.27$ , the piston becomes solidly jammed in the nozzle due to the thick, compacted powder coat. It is very difficult to detach the nozzle for weighing and during the process, powder may be lost from the nozzle wall giving rise to possible errors and an underestimate of the actual weight of powder coat per unit nozzle length. In the case of L2, powder starts to bind and compact on the wall at  $Cr = 0.3$  and at  $Cr = 0.44$ , it becomes quite difficult to detach the nozzle for weighing, again due to the compacted powder coat formed. For L1, powder compaction and its binding on the wall starts to occur only at  $Cr = 0.43$  and becomes prominent at  $Cr = 0.55$ .

In contrast, the negligible coating and relative insensitivity to compression observed for S3, S2, A3 and A2 indicates the little surface affinity of these powders for the nozzle wall surface as the irregularities on it are probably too small to accommodate the powder particles. Overall, greater amounts of coating are shown by fine size fractions of calcium carbonate (C1), Starch 1500 (S1), maize starch (M1) and lactose (L1 and L2). Another noticeable feature is that whilst powder coatings of Starch 1500, Avicel and maize starch are easily removed by brushing or blowing, those of calcium carbonate and lactose are not readily dislodged after experimentation and thin films of the powders remained on the wall surface.

#### *Experiments with a coated nozzle fill weight, weight variation and powder coating*

The results obtained for Starch 1500 powders when capsules were filled after the machine had been 'run in' and the nozzle coated with a constant coat of each powder, are essentially similar to those of Figs 1 and 5 when a clean nozzle was used. A minor difference is the increase in the value of the CV and a slight decrease in the mean fill weight of S1 at  $Cr = 0.55$  when a coated nozzle was used.

For S2 and S3, there is only a slight tendency for the powders to coat on the nozzle, and any coating would be constant after filling the first few capsules. Hence, the similarity in the profiles between the use of a clean and coated nozzle is to be expected. In the case of S1, powder coating below a Cr setting of 0.55 is of a loose and non compacted nature. A constant coat is formed after some loose capsules have been filled and further powder build-up on the nozzle wall is automatically dislodged and prevented by the piston action and machine motion during the 'running in' period. At Cr = 0.55, compaction of powder on the nozzle wall starts to occur and during the 'running in' period, there is a gradual build-up of this powder coat, which may not be dislodged so readily. Examination of the results for Starch 1500 shows a greater amount of coating at a Cr setting of 0.55 when compared in Fig. 6. Filling capsules with the coated nozzle at this Cr would thus result in more variable fill weights as the movement of the piston would be somewhat restricted by the powder coat on the wall.

For Avicel, calcium carbonate and maize starch powders, results from the use of coated nozzles were comparable to those with clean nozzles. Similar explanations (as in the case of Starch 1500) may be used to account for these observations. Minor differences amongst the results are to be expected due to the inherent variability of the experimental work. A notable difference between the two nozzle states is the absence of zero fills at Cr = 0 when the fine size fraction of maize starch, M1 is filled with a coated nozzle. This may be ascribed to the increase in the angle of wall friction caused by the powder coat, hence aiding powder retention through better frictional support at the wall. For calcium carbonate, the decrease in the amount of powder coat observed at Cr = 0.55 with a coated nozzle may be similarly explained as there is less adhesion at the wall surface when the powder becomes compacted (Tan, 1987).

Attempts to fill lactose powders with a coated nozzle proved unsuccessful due to the severe binding of the powders onto the nozzle wall and piston, resulting in the jamming of the latter.

## Conclusions

(i) For most powder systems, the highest, most uniform fill weights are generally achieved when no piston compression is applied during dosing. An exception to this trend is exhibited by the fine size fractions of maize starch, M1 and lactose, L1 where some piston compression is required to aid powder retention. Whilst coarse powders may be satisfactorily filled over their available compression ranges, high Cr settings tend to be very detrimental to the filling of fine powders. For lactose powders, high Cr values result in the jamming of the piston.

(ii) Fine powders show greater coatings of nozzle walls than coarser ones and the coatings usually become more pronounced with increasing Cr settings. For the three lactose size fractions, there is severe binding of the powders onto the nozzle wall and piston.

(iii) With the exception of lactose, filling powders with a coated nozzle gives comparable results to those obtained with a clean one. Lactose powders cannot be filled with a coated nozzle due to their severe binding onto the nozzle wall and piston and the eventual jamming of the latter.

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