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Observed and expected powder plug densities obtained by a capsule dosator nozzle system

S.B. Tan* and J.M. Newton

The School of Pharmacy, University of London, Brunswick Square, London WC1N 1AX (U.K.)

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Summary

The influence of compression setting on the observed powder plug density (γ_{po}) and its deviation from the expected plug density (γ_{pe}) during capsule filling of different size fractions of some pharmaceutical powders on an mG2 simulator have been investigated. Values of γ_{po} were particle size and material dependent. Deviations in the values of γ_{po} from γ_{pe} were attributed to differences between the observed and expected fill weights. Larger deviations in the values of γ_{po} from γ_{pe} were generally observed with fine powders and higher compression settings.

Introduction

Automatic capsule filling machines which employ a dosator nozzle system rely on accurate powder dosing, the formation and retention of the powder plug within the dosator nozzle and subsequent transference and ejection of this plug into an awaiting capsule body. Powder retention within the dosator nozzle may be aided by the application of a compressive force on the piston whilst it is dipping into the powder bed during the dosing phase. Ideally, minimal piston compression should be used to assist retention as capsule filling performance (Jolliffe and Newton, 1982; Tan, 1987; Tan and Newton, 1990b) and drug release (Mehta and Augsburger, 1981) are known to be influenced by piston compression settings.

This paper assesses the influence of piston compression settings on the observed powder plug density (γ_{po}) and its deviation from the expected plug density (γ_{pe}) during capsule filling of different size fractions of five pharmaceutical excipients on an instrumented mG2 simulator.

Materials and Methods

Materials

Size fractions of microcrystalline cellulose, (Avicel PH101), pregelatinised starch (Starch 1500), calcium carbonate, maize starch and lactose B170, fractionated and characterised as described pre-

Correspondence: J.M. Newton The School of Pharmacy, University of London, Brunswick Square, London WC1N 1AX, U.K.
*Present address: Evans Medical Ltd., Langhurst, Horsham, West Sussex RH12 4QD.

TABLE 1

Mean particle length (A) μm and standard deviation of powder samples

	Starch 1500			Avicel PH101			Calcium carbonate			Maize starch		Lactose B170		
Code:	S1	S2	S3	A1	A2	A3	C1	C2	C3	M1	M2	L1	L2	L3
A	13.7	29.7	66.7	16.7	58.9	88.7	6.8	14.5	25.2	10.2	15.2	6.6	13.2	25.9
(±S.D.)	(4.3)	(9.3)	(15.0)	(9.3)	(25.9)	(34.3)	(1.9)	(3.4)	(5.1)	(3.3)	(4.1)	(2.4)	(6.5)	(10.0)

viously (Tan and Newton, 1990a) were used for the present study. The coding of the samples and their mean particle size (expressed as a length) are given in Table 1.

Capsule filling

Capsule filling studies on the different powder systems were carried out with an instrumented mG2 simulator at various compression settings using a clean size 1 dosator nozzle of medium texture, M, as described elsewhere (Tan and Newton, 1990a).

The powder bulk density (γ_b) of the feed bed on the simulator was determined as described elsewhere (Tan and Newton, 1990c).

The observed powder plug density (γ_{po}) of each powder at a specific compression (Cr) setting was calculated from the weight of the powder plug ejected divided by its volume.

As the cross-sectional area of the dosator nozzle was constant, the expected powder plug density (γ_{pc}) of each powder at a particular Cr setting could be calculated directly from Eqn 1:

$$\gamma_{pc} = \frac{\gamma_b \cdot L_0}{L_p} \qquad (1)$$

where γ_b denotes powder bulk density, L_0 is fill length (distance between the piston tip and nozzle outlet without piston compression) and L_p is length of the powder plug (determined from the displacement of the piston during compression). For each powder, values of γ_{po} and the deviation of γ_{po} from γ_{pc} were plotted against Cr settings.

Results and Discussion

From the results presented in Fig. 1, it is clear that larger particle size powders (S3, S2, C3, C2, M2, L3 and L2) generally have higher γ_{po} values than the finer size fractions (S1, C1, M1 and L1). An exception to this trend is the case for Avicel powders where the fine size fraction, A1, shows fractionally higher γ_{po} values than the coarser fractions A2 and A3. For powders of C3, C2, S3 and

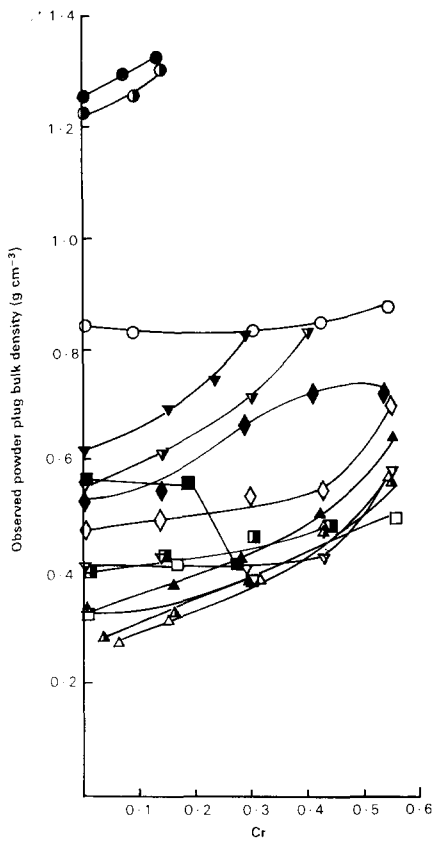

Fig. 1. Observed powder plug density as a function of compression ratio Cr. Avicel PH101: (▲) A3, (△) A2, (▲) A1; Starch 1500: (▼) S3, (▼) S2, (▽) S1; Maize starch: (◆) M2, (◇) M1; Calcium carbonate: (●) C3, (◑) C2, (○) C1; Lactose: (■) L3, (◨) L2, (□) L1.

S2, the high initial γ_{po} values at Cr = 0 confirm the ease of consolidation of these powders into closely packed states during powder bed rotation and leveller action without piston compression (Tan and Newton, 1990b). With increasing Cr settings, corresponding increases in the γ_{po} values for these powders are seen but their compression limits are rapidly reached (at Cr 0.14 for C3, C2 and at Cr = 0.29, 0.4 for S3 and S2, respectively) when the plugs are in highly compacted states and further volume reductions are not feasible. Whilst a similar trend is observed for powder M2, it is much more compressible (than C3, C2, S3 and S2) and its compression limit is only reached at Cr = 0.54 (as shown by a levelling off of the γ_{po} value).

In the case of the largest size fraction of lactose, L3, the initial increase in the γ_{po} value at Cr = 0.15, accompanied by a sharp fall in the γ_{po} value at Cr = 0.27 is indicative of some initial consolidation of the powder bed followed by piston jamming, commencing at a higher Cr setting. When this happens, a compacted powder coat binds onto the nozzle and piston preventing the free movement of the latter during powder dosing and ejection. This results in poor retention and incomplete ejection of the powder. In addition, there is powder loss through its coating on the wall and behind the piston tip and all these factors contribute to a decrease in powder fill into the capsule and the low γ_{po} value. With size fraction L2, increasing Cr settings result in some consolidation of the powder and a corresponding increase in the γ_{po} value, but piston jamming again sets in at Cr = 0.44.

In contrast to the larger size fractions of powders just described, fine powders, i.e. S1, C1, M1 and L1 are highly compressible in nature and have high bed porosities (hence low γ_{po} values. For these powders, because of their high porosities and the tendency of powder losses behind the piston tip and on the nozzle wall, resulting in less powder available for compression, increasing Cr values do not usually bring about any noticeable increase in the γ_{po} values. However, at a very high Cr setting (0.55), significant increases in the γ_{po} values are seen for S1, M1 and L1 due to closer packing of the particles of these powders.

As previously mentioned, Avicel powders are unique and distinct from other powders in that all the three size fractions respond to increasing Cr settings with the fine size fraction, A1, showing fractionally higher γ_{po} values than the coarse size fractions A3 and A2. This is due to the lower porosity of the powder bed of A1 and the ability of its smaller and less elongated particles to move into a more tightly packed state during compression (Tan, 1987). The almost identical profiles of A2 and A3 reflect the closeness of their properties, especially their bulk densities and compressional behaviour (Tan and Newton, 1990a).

As the volume of fill is constant, any deviation of γ_{po} from γ_{pc} may be ascribed to the difference in the observed and the expected weight of fill. Factors associated with uniformity of fill have been discussed elsewhere (Tan and Newton, 1990a,b). The results for the deviation of γ_{pc} from γ_{po} are shown in Fig. 2.

In general, fine size powders, S1, C1, A1 and M1 exhibit larger deviations at all Cr settings than coarser powders, S3, S2, C3, C2, A3, A2 and M2. For most powders (except A3 and A2) smallest deviation is observed at Cr = 0; increasing Cr values result in greater deviations. At Cr = 0, poor retention and the loss of powder to the vicinity of the receiving capsule body during the ejection stage constitute the major causes for the deviation of γ_{po} from γ_{pc} for most powder systems. Other contributory factors for the deviations include powder losses through coating on the nozzle wall and their losses behind the piston tip. There is a greater tendency for the fine powders (especially S1, A1 and M1) to be influenced by these factors than the corresponding coarser powders (S3, S2, A3 and M2). For S3 and S2 in particular, there is excellent agreement between the values of γ_{po} and γ_{pc} and this is confirmed by the good correlation between the observed and theoretical fill weights (Tan and Newton, 1990b). This also indicates the good retention ability of the powders and their insignificant losses due to the above factors.

The higher deviations observed for the powders (except A3 and A2) with increasing Cr settings may be associated with greater powder coatings on the nozzle wall and their losses behind the piston tip. Again these effects are more pronounced with fine powders, S1, C1, M1 and A1. Due to their smaller particle sizes, these powders have greater

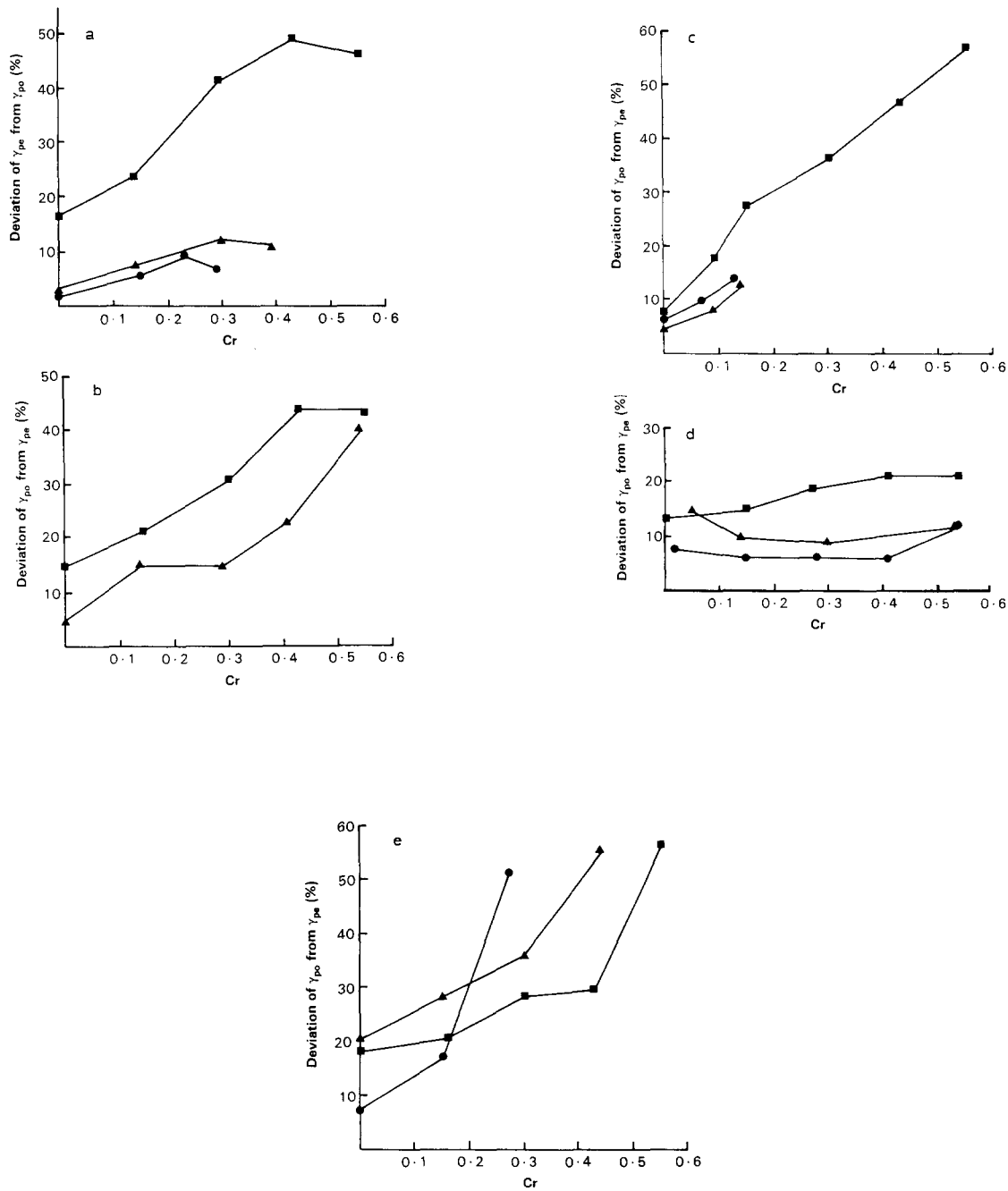

Fig. 2. Deviation of the observed (γ_{po}) from the expected (γ_{pe}) powder density as a function of compression ratio Cr. (a) Starch 1500: (●) S3, (▲) S2, (■) S1; (b) Maize starch: (▲) M2, (■) M1; (c) Calcium carbonate: (●) C3, (▲) C2, (■) C1; (d) Avicel PH101: (●) A3, (▲) A2, (■) A1; (e) Lactose: (●) L3, (▲) L2, (■) L1.

affinities for the wall surface (i.e. the abilities of the particles to fit into the wall surface irregularities) as shown by their higher angles of wall friction (Tan and Newton, 1990c). Their small sizes and high and variable bed porosities (Tan and Newton, 1990b) also allow the piston to push readily through the powder beds, trapping the particles behind the piston tip. In contrast, for coarse powders such as S3, S2, A3, A2, C3 and C2, the large particles are unable to fit into the wall surface irregularities, hence very little powder coating occurs. Loss of powders behind the piston tip is further prevented by the size and morphology of the particles (e.g. the large and elongated particles of A3 and A2 are not easily trapped behind the piston tip).

In the case of A2 and A3, the minimum deviations seen at Cr = 0.14 to 0.3 and 0.15 to 0.41, respectively, suggest that optimum plugs are being formed over these compression ranges, with very little losses due to the above causes. Slightly higher deviations seen at compression settings below 0.15 are due to the poorer retention of the loose powders being filled and their losses outside the vicinity of the capsule bodies during powder ejection as discussed earlier.

The slight decrease in the deviations of γ_{pc} from γ_{po} at the highest Cr settings for S3, S2, S1, M1 and A1 may be associated with the formation of more compacted plugs, thus ensuring less powder losses to the outside of the receiving capsule bodies during the ejection stage.

For lactose powders (L1, L2 and L3; Fig. 2e) the deviations of γ_{po} from γ_{pc} at Cr = 0 are also primarily due to their losses during the transfer and ejection stages. With increasing Cr settings, the problems of powder coatings on the nozzle wall and their losses behind the piston tip become prominent; but, unlike other powders discussed so far there is also a tendency for severe binding of the lactose powders onto the nozzle and piston causing the eventual jamming of the piston within the nozzle at the highest compression settings (i.e. at Cr = 0.27, 0.44 and 0.55 for L3, L2 and L1, respectively). Due to the thick powder build-up, there is a reduction in the fill volumes and the incomplete ejection of the powder plugs, hence accounting for a marked decrease in the amount of powders being filled into the capsules and the large deviations of γ_{po} from γ_{pc}.

Conclusions

(i) Observed powder plug density (γ_{po}) values are particle size and material dependent. For a particular excipient (with the exception of Avicel) coarse powders show higher γ_{po} values than fine size fractions. Whilst there is a marked increase in the γ_{po} values of coarse powders with higher Cr settings, fine powders are relatively insensitive to piston compression (except A1).

(ii) Deviations in the values of γ_{po} from the γ_{pc} are due to differences between the observed and expected fill weights. Powder coatings and their losses behind the piston tip are major contributory factors to the lower observed fill weights and these problems are more pronounced with fine powders. Increased Cr settings generally result in larger deviations of γ_{po} from γ_{pc}.

(iii) With lactose powders, the additional problem of powder binding onto the nozzle and piston and the eventual jamming of the latter account for the very large deviation of γ_{po} from γ_{pc}.

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