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Minimum compression stress requirements for arching and powder retention within a dosator nozzle during capsule filling

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Summary

The theoretical minimum stress requirements for arching and powder retention within a dosator nozzle during capsule filling have been predicted for different size fractions of five pharmaceutical excipients. In general, higher compressive stress requirement for arching, $\sigma_{z\ req}$ at the arching zone also required the application of a greater compressive stress, $\sigma_{z,\ o\ req}$ at the top of the powder bed. The magnitude of these stresses was particle size and material dependent. Whilst these calculated stresses were generally too low to be measured experimentally, the results correlated with the observed capsule filling performance of the powders.

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. Powder retention may often be assisted by the application of a compression force during dosing. This force should be low so that subsequent ejection of the plug can be achieved with minimum effort (Jolliffe et al., 1980). Mehta and Augsburger (1981) observed that soft powder plugs resulting from low compressive stress are desirable for rapid drug release. Tan (1987) and Tan and Newton (1990c) reported the detrimental effects of high compression stress on the capsule-filling performance of pharmaceutical powders, especially fine size fractions.

Based on the hopper design theory of Walker (1966) and Walters (1973), Jolliffe et al. (1980) proposed a theory which states that powder retention within a dosator nozzle during capsule filling requires the formation of a stable arch at the nozzle outlet. This is dependent on the powder properties and the interaction between the powder and the nozzle wall (its angle of wall friction). The maximum span over which a powder can arch depends on the shear developed at the wall supporting its weight. The arch will be stable provided the strength of the powder at its free surface is equal to or greater than its unconfined yield

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strength, f_c. According to Walker (1966), the strength required in a powder bed for arching to occur is given by the equation:

$$f_c = \frac{r\gamma g}{\sin 2\phi} \tag{1}$$

where f_c represents unconfined yield strength, r is span radius, γ denotes powder bulk density, ϕ is the angle of wall friction and g is the acceleration due to gravity.

Retention of powder may be assisted by the application of a vertical compressive force to the top of the powder bed by a piston, whilst the nozzle is dipping into the bed.

Further development of the theory by Jolliffe et al. (1980) enabled the vertical compressive stress required to act at the arching zone to produce f_c within the powder, hence ensuring arching,

$$\sigma_{z\ req} = \frac{\mathrm{FF} r\gamma g}{\sin 2\phi} \tag{2}$$

where FF is the Jenike flow factor. For a given powder, $\sigma_{z\ req}$ decreases as ϕ increases. To achieve $\sigma_{z\ req}$ at the bottom of the powder bed to ensure arching, a higher compressive stress has to be applied at the top of the powder bed. This is because part of the applied stress is used to initiate frictional support at the wall (Brown and Richards, 1970) and the stress transmitted to the bottom of the bed is reduced. Taking this into consideration, Jolliffe et al. (1980) derived another equation to allow for calculation of the stress required at the top of the powder bed, $\sigma_{z,o\ req}$:

$$\sigma_{z,o\ req} = \frac{\sigma_{z\ req} - r\gamma g/2\mathrm{BD}[1 - \exp(-2\mathrm{BD}z/r)]}{\exp(-2\mathrm{BD}z/r)} \tag{3}$$

where z is the powder bed depth and BD is a factor derived from ϕ and δ (the angle of effective friction).

Experimental support of this theoretical approach was provided by different lactose size fractions (Jolliffe and Newton, 1982). The authors concluded that the angle of powder-wall friction is of prime importance in determining the stress distribution of the powder within the nozzle. An optimum value of powder wall friction exists for retention of powders with minimum force. Free flowing powders are sensitive to the angle of powder wall friction but cohesive powders are easily retained over a wide range of wall friction properties.

This paper describes the application of the theory and equations proposed by Jolliffe et al. (1980) and Walker (1966) to the prediction of minimum stress requirement for arching and powder retention within a capsule dosator nozzle during the filling of different size fractions of five pharmaceutical excipients. The theoretical stress requirement for each powder is then related to its observed capsule filling performance.

Materials and Methods

Materials

Size fractions of microcrystalline cellulose (Avicel PH101) (A), pregelatinised starch (Starch 1500) (S), calcium carbonate (C), maize starch (M) and lactose monohydrate (L), fractionated and characterised as described elsewhere (Tan and Newton, 1990a) were used for the present study.

Determination of the flow function (FF), angle of effective friction (δ) and angle of wall friction (ϕ) for the powders

The values of FF and δ for each powder were determined using an annular shear cell, details of which have been described elsewhere (Tan and Newton, 1990a).

As it is not feasible to measure the values of ϕ on the dosator nozzle wall directly, its values have been extrapolated from the results obtained from experiments using a modified annular shear cell (Tan and Newton, 1990b). Values of ϕ obtained for an annular shear wall plate Mt were extrapolated to values of ϕ for a dosator nozzle wall, M of similar wall texture and Ra value to the former.

Capsule filling

Capsule filling studies on the powders were carried out on an instrumented mG2 simulator using a clean size 1 dosator nozzle of medium texture, M (Tan and Newton, 1990a).

The powder bulk density (γ) of the feed bed was determined using specially constructed sampler to remove samples from different locations of the powder bed. Values of γ were calculated from the weight and volume of the sample removed (Tan and Newton, 1990a).

Calculation of the values of f_c, $\sigma_{z\ req}$ and $\sigma_{z,o\ req}$

Values of FF, δ, ϕ and γ obtained by experimentation were fitted into Eqns 1–3 to enable the calculation of the minimum compressive stress required to be applied at the top of the powder bed, $\sigma_{z,o\ req}$, to achieve the vertical compressive stress at the arching zone, $\sigma_{z\ req}$, and the unconfined yield stress, f_c, within the powder to ensure arching.

Results and Discussion

Values of FF, δ, ϕ and γ for the powders and the calculated magnitude of the stresses, f_c, $\sigma_{z\ req}$ and $\sigma_{z,o\ req}$ are presented in Table 1 and Fig. 1.

It is apparent that generally large values of f_c and $\sigma_{z\ req}$ require the application of high compressive stress, $\sigma_{z,o\ req}$. For example, the large values of f_c and $\sigma_{z\ req}$ seen for C2 and C3 mean that higher values of $\sigma_{z,o\ req}$ will be required for arch formation and powder retention. This is due to the fact that the large and free flowing particles (i.e. high FF values) of these powders with their high bulk densities would be less strongly retained than other powders. Hence, the need for higher values of $\sigma_{z,o\ req}$. In contrast, arch formations (hence retention) are readily achieved with cohesive powders (low FF values) L1 and L2 where retentions are also facilitated by the large values of ϕ. Hence, the magnitude of f_c and $\sigma_{z\ req}$ would be low with a corresponding small value of $\sigma_{z,o\ req}$.

From the above discussion, it is apparent that the magnitude of the stresses f_c, $\sigma_{z\ req}$ and $\sigma_{z,o\ req}$ are material as well as particle size dependent. For a particular excipient, values of f_c, $\sigma_{z\ req}$ and $\sigma_{z,o\ req}$ are direct functions of particle size. As the particle size decreases, so does the value of f_c, $\sigma_{z\ req}$ and $\sigma_{z,o\ req}$. This is because a powder with small particles becomes more cohesive (i.e. has a lower value of FF), has a higher angle of wall

TABLE 1

Calculated values of the stresses required for arch formation and powder retention within the dosator nozzle, M

Powder code *	FF	σ^0	γ (kg m^{-3})	ϕ^0	f_c (N m^{-2})	$\sigma_{z\ req}$ (N m^{-2})	$\sigma_{z,o\ req}$ (N m^{-2})
A1	2.2	39.3	378	15.6	21	46	16
A2	4.2	40.2	300	11.8	21	90	49
A3	7.0	38.9	294	10.7	23	161	55
C1	3.8	32.3	913	28.6	31	118	386
C2	7.5	32.8	1 283	25.2	47	356	1 211
C3	10.8	31.3	1 334	21.9	55	592	1 863
L1	2.4	39.5	376	32.3	12	28	32
L2	4.6	39.5	504	35.3	15	70	221
L3	6.3	40.0	609	34.8	19	140	551
M1	2.2	31.9	550	14.0	33	73	73
M2	3.5	35.3	557	11.4	41	143	141
S1	2.6	38.8	485	12.5	33	85	50
S2	3.8	37.2	569	15.7	31	118	120
S3	6.5	34.0	636	13.3	40	258	387

* The letter code indicates the incipient type (as given in Materials) while the number is associated with the particle size fraction — the smaller the number, the smaller the particle size (see Tan and Newton (1990a) for full details).

NB: FF and σ as in previous table; γ, powder bulk density; ϕ, angle of wall friction (extrapolated from values obtained from annular shear cell plate, Mt); f_c = unconfined yield strength; $\sigma_{z\ req}$ = vertical compressive strength required for arching at arching zone; $\sigma_{z,o\ req}$ = applied vertical compressive stress required for arching.

friction (ϕ), and a lower bulk density. When the values of f_c, $\sigma_{z\ req}$ and $\sigma_{z,o\ req}$ are related to x_{cv} non-significant correlations are however observed (Table 2 and Figs 2–4). This implies that the capsule filling performance of the powder studies is not controlled by the magnitude of these stresses.

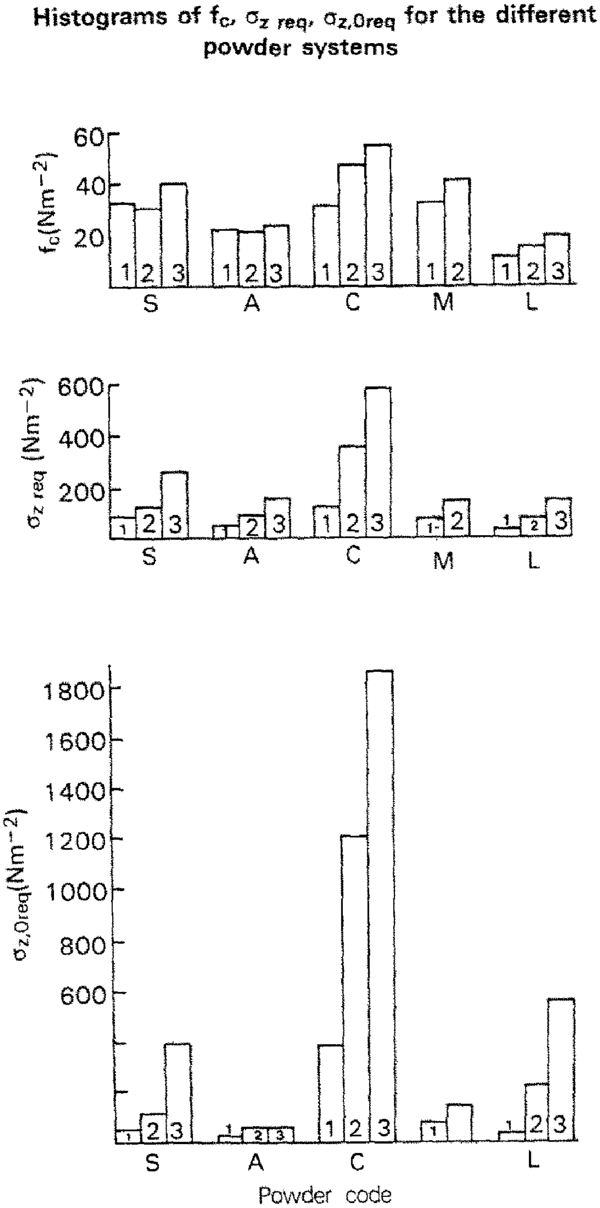

Fig. 1. Histograms of f_c, $\sigma_{z\ req}$ and $\sigma_{z,o\ req}$ for the different powder systems.

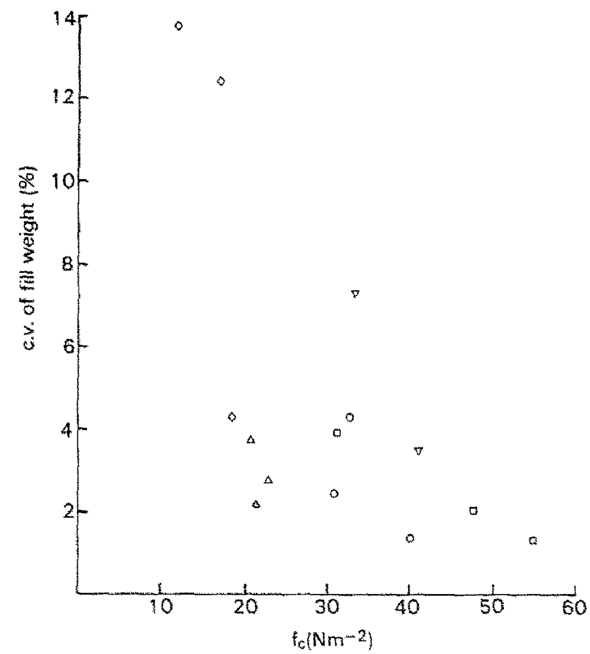

Fig. 2. C.V. of fill weight as a function of f_c ('clean' nozzle M). (○) S, (△) A, (□) C, (▽) M, (◇) L.

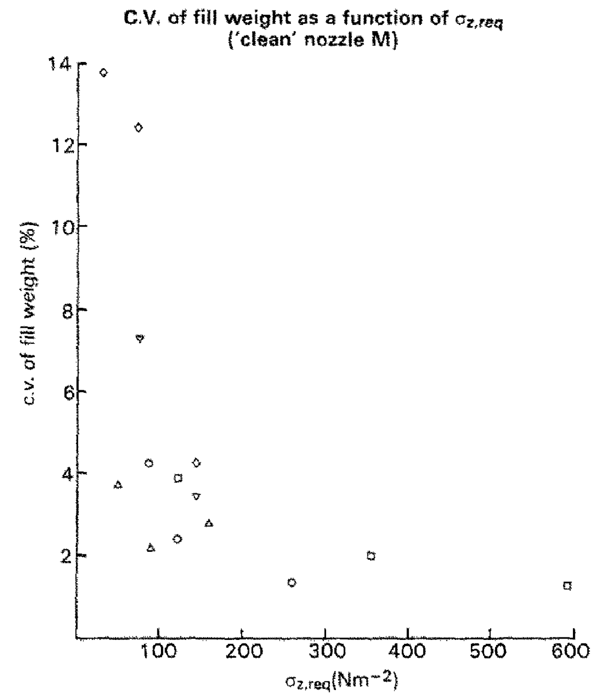

Fig. 3. C.V. of fill weight as a function of $\sigma_{z\ req}$ ('clean' nozzle M). (○) S, (△) A, (□) C, (▽) M, (◇) L.

TABLE 2

Relationship between the coefficient of variation of capsule fill weight (X_{cv}) and the calculated values of the stress requirements (S_c)

S_c	Including values for lactose ($n = 14$)				Excluding values for lactose ($n = 11$)			
	r	c	m	Comment	r	c	m	Comment
f_c	−0.636	10.618	−0.198	[a]	−0.314	4.832	−0.049	n.s.
$\sigma_{z\ req}$	−0.518	6.824	−0.013	n.s.	−0.595	4.304	−6.272	n.s.
$\sigma_{z,o\ req}$	−0.357	5.599	−2.6	n.s.	−0.489	3.694	−1.399	n.s.

[a] Significant at 5% level.

Symbols as in Table 1 except: n, number of pairs of observations; r, correlation coefficient; c and m, intercept and slope of the best fitting line; n.s., non significant.

Examination of Table 1 also shows some anomalous behaviour exhibited by S1, A1, A2 and A3. For these powders, the calculated values of $\sigma_{z,o\ req}$ appear less than those of $\sigma_{z\ req}$. In the theoretical approach of Jolliffe et al. (1980), lower values of $\sigma_{z\ req}$ compared to $\sigma_{z,o\ req}$ have also been reported for cases where the FF values of the powders are low (below ≙ 5). Thus, it appears that for the above cohesive powders, arch formation and powder retention may be achieved within the dosator nozzle during dosing without the need for the application of a vertical compressive stress ($\sigma_{z,o\ req}$). This is facilitated by the low bulk densities and the optimal angles of wall friction (ϕ) of these powders, hence favouring stress transmissions and powder retentions (Table 1).

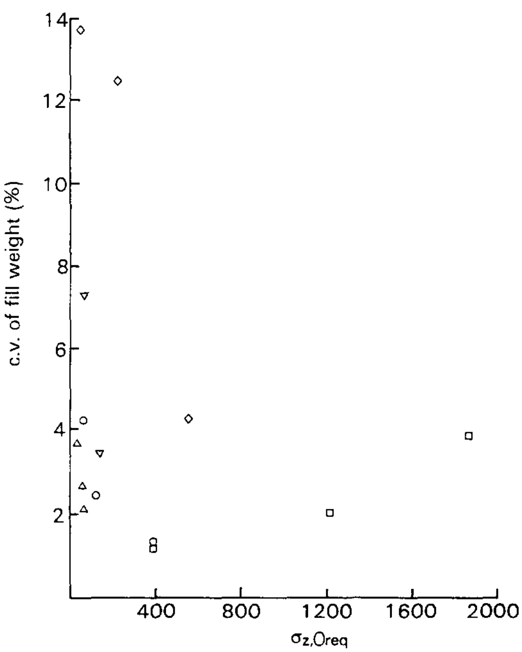

Fig. 4. C.V. of fill weight as a function of $\sigma_{z,o\ req}$ ('clean' nozzle M). (○) S, (△) A, (□) C, (▽) M, (◇) L.

As previously discussed (Tan and Newton, 1990a,c) all the powders used in the present study required little or no piston compression for retention during capsule filling on the mG2 simulator. It is evident from the results presented in Table 1 that even the highest calculated value of $\sigma_{z,o\ req}$ required for arch formation and powder retention (i.e. for C3) is much lower than that encountered experimentally and is beyond the sensitivity of the instrumented equipment used (lowest limit of sensitivity was approx. 10 kN m^{-2}).

Thus, it may be inferred that during the actual filling process, the consolidating of the powder bed during its formation is sufficient for arch formation and retention without the need for piston compression.

Conclusions

Application of the theory and equations proposed by Walker (1966) and Jolliffe et al. (1980) to the present study has made possible the calculation of the theoretical compressive stress requirement for arch formation and powder retention. In general, higher values of $\sigma_{z\ req}$ at the arching zone also require greater compressive stress $\sigma_{z,o\ req}$ at the top of the powder bed. The magnitude of these stresses is material and particle size dependent. With the present powders studied, these stresses are not the main controlling feature of capsule filling performance, although the magnitude of f_c and $\sigma_{z\ req}$ may to some extent influence capsule fill weight uniformity. Whilst the calculated stress requirement for arch formation for all the powders is generally too low to be measured experimentally, this also implies that these powders show good arching and retention ability without the need for piston compression during dosing. It confirms the findings by Tan and Newton (1990a,c) where the optimal filling conditions for most powders are observed at a compression setting of 0 (i.e. no piston compression).

The present results provide further experimental support to the theoretical concept of Jolliffe et al. (1980).

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