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The effect of particle shape on the variation of fill of a tabletting die

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A 12 mm die cavity has been repeatedly filled from batches of granular material of a range of particle shape and size distributions. The variance in the weight contained by the die was determined from the replicates. The variance, expressed as a coefficient of variation to allow for differences in the means of groups of replicates, increased from 0·2635 to 0·7222 as the particle shape, expressed as the Heywood shape coefficient, increased in irregularity from a value of 8 to 13. The mean contained weight was greater for regular than for irregular particles, and a maximum die fill occurred where the ratio of die diameter to particle diameter was about 20 for all particle shapes.

Powders are normally granulated to give a free-flowing material suitable for feeding to the dies of a tabletting machine. The aim is to obtain granules of a uniform size and of a shape not too far removed from the spherical, and it is generally accepted that the ease and accuracy of filling the die will be greater if this aim is achieved. So far as we are aware, no quantitative measurements have been made relating the variability of fill of a die to the degree of departure from the ideal aim of a uniform spherical granulation.

Hersey (1965) reported that the weight uniformity of the contents of a die in a tablet machine simulation apparatus was a function of the method adopted to fill the die, but no quantitative results were given. The die was filled (*a*) as a blind hole with one pass of the feed shoe; (*b*) by lowering the bottom punch whilst ample heaped powder was present at the die mouth; and (*c*) by allowing the lower punch to drop below its required setting as the die filled, then subsequently raising it and levelling off the powder rising above the die mouth with a knife blade. The reproducibility, and the mean weight of powder contained, both increased in the order (*a*) to (*b*) to (*c*).

In the experiments reported here, sand of particle size 60–85, 44–60, 30–36, 18–20 mesh, in batches sorted according to particle shape by the method of Ridgway & Rupp (1969), has been fed into a simple die simulator: the weight of sand contained was then measured. Repetition of the experiment allowed the variance as a function of particle shape to be assessed.

EXPERIMENTAL

The apparatus (Fig. 1) consists of a circular brass disc bored at its centre with an accurate parallel-sided hole. The disc was faced-off and a similar, thinner disc was attached to it by three screws. This assembly formed the die. A flanged cylinder formed the feed shoe.

The sand used was supplied by British Industrial Sand Limited, Reigate and by George Garside (Sand) Limited, Leighton Buzzard. It was divided into size fractions

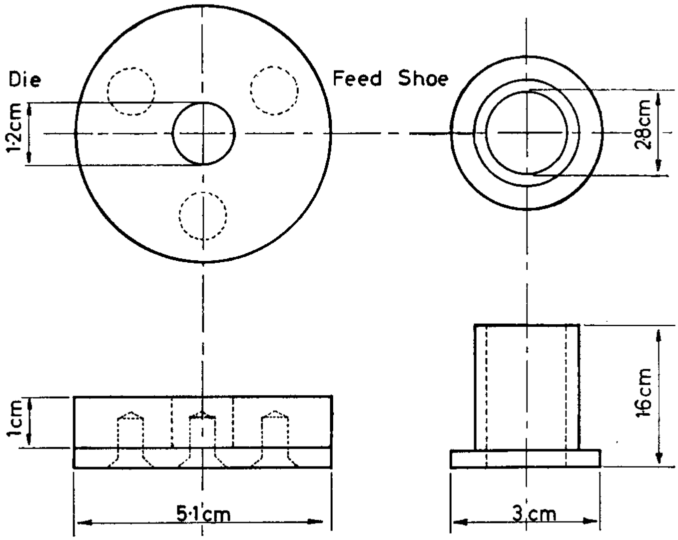

FIG. 1. The die simulator. The feed shoe cylinder is filled with the powder under test and moved manually across the face of the die.

by sieving, and the following size fractions were obtained: 60–85, 44–60, 30–36, 18–20 mesh. Each sieve cut was subdivided into four fractions of different shape by being passed over the deck of a shape-sorting machine (Jeffrey-Galion Ltd., Johannesburg).

Shape coefficients (Heywood, 1969) were determined as described previously (Ridgway & Rupp, 1969).

Sand was loaded into the feed shoe, which was passed over the die manually three times. Any sand grains on top of the die disc were removed, and the sand in the die was tipped out and weighed. Ten replicate determinations were made for each particle size and shape.

At the extremes of shape coefficient for each particle size fraction a further hundred replicate determinations of the weight of die fill for each sample of sand were made; the variance was thus determined. To allow for the change in mean weight of die fill in each group of measurements the variance was represented as the coefficient of variation.

RESULTS AND DISCUSSION

The values of particle size, shape coefficient, mean weight of die fill and variance about the mean are given in Table 1. The variance was estimated for all samples by examining the distribution of the weights of fill about the mean. But ten measurements is not a sufficiently large number from which to obtain a variance estimate of any precision. Accordingly the two samples at each size which were the most and the least spherical respectively were made the subject of 100 replications, and much more reliance can be placed upon the variance estimates derived from these measurements.

As expected the mean weight of die fill is reduced as the shape coefficient increases (Fig. 2). Irregular particles flow less freely from the feed shoe, and are less ready to rearrange into a closer packed random arrangement when they drop into the die.

Table 1. *Variation in die-fill with particle size and shape*

Mesh size mean size and sample no.	Projected diameters (μ)	Shape coeff. f/k	Mean weight g	Variance × 10⁻⁶ with degrees of freedom: 9	99	Coefficient of variation with degrees of freedom: 9	99
60/85 213 μm							
1	214	7·87	1·9579	28	35	0·2702	0·3012
2	218	9·15	1·8989	53		0·3833	
3	220	9·87	1·8726	44		0·3542	
4	228	11·92	1·7843	47	38	0·3841	0·3434
44/60 302 μm							
5	343	8·78	1·9289	127	34	0·5842	0·2964
6	357	10·47	1·8651	46		0·3636	
7	358	11·03	1·8272	17		0·2256	
8	378	13·03	1·7347	72	33	0·4891	0·3248
30/36 461 μm							
9	559	7·66	2·0801	132	31	0·5523	0·2635
10	609	8·70	2·0237	40		0·3125	
11	661	9·81	1·9397	115		0·5528	
12	694	11·54	1·8529	80	75	0·4827	0·4544
18/20 805 μm							
13	909	8·17	2·0668	187	94	0·6616	0·4662
14	967	9·21	2·0038	176		0·6620	
15	1024	9·87	1·9721	291		0·8649	
16	1051	11·04	1·9041	229	191	0·7947	0·7222

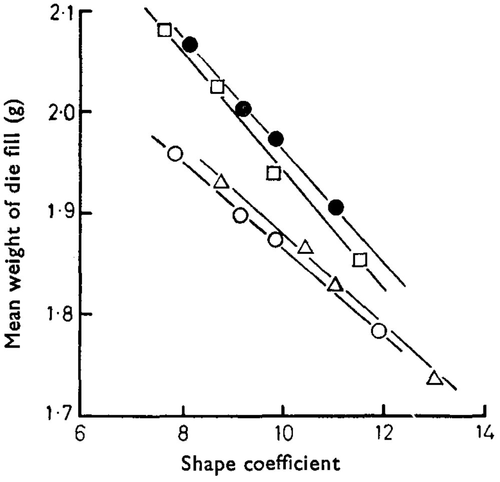

FIG. 2. The effect of shape coefficient on the mean weight of die fill for various particle sizes. ● 18–20 mesh, mean size 805 μm. □ 30–36 mesh, mean size 461 μm. △ 44–60 mesh, mean size 302 μm. ○ 60–85 mesh, mean size 213 μm.

Beyond a shape coefficient of 15, departure from linearity would be anticipated; extrapolation would otherwise predict zero weight in the die for very irregular particles.

The coefficient of variation of the die fill increases with particle size when shape is discounted: particles of mean diameter 805 μm have a coefficient of variation of 0·76, for those of 461 μm it is 0·48, for 302 μm it is 0·42 and for 213 μm it is 0·34. These mean sizes are from the sieve cuts 18–20, 30–36, 44–60 and 60–85 mesh respectively. For larger particles, where the mean diameter is 800 μm, there is only room for about 15 particles to fit across the width of the die. Thus the effect of the die wall, which is to introduce a region of variable, usually larger, voidage (Ridgway & Tarbuck, 1966) becomes paramount so that the variance is increased.

The effect of changing particle shape on the reproducibility of the weight fill of the die is shown in Fig. 3. Each line has but two points because of the large number of determinations which are needed to characterize a variance with any degree of precision. The trend is clear, however. Irregularity of particle shape at constant size causes the random variations of weight to become larger.

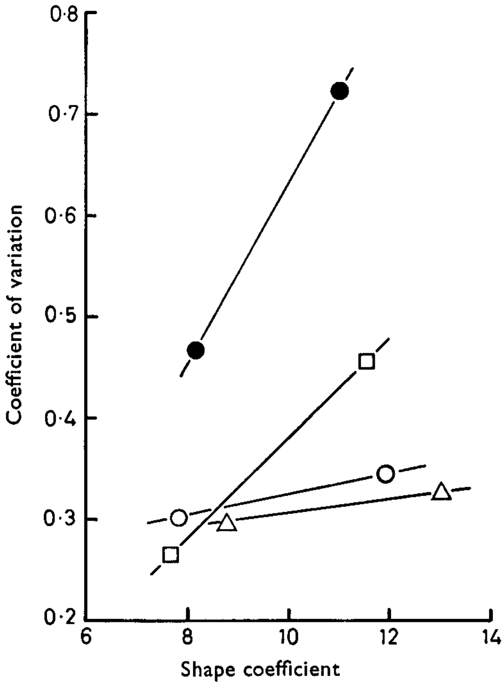

FIG. 3. The effect of shape coefficient on the coefficient of variation of the die fill. For key to points, see Fig. 2.

Finally, Fig. 4 shows how the mean weight of the die fill changes with increasing particle size, at constant shape factor. Here the points on the curves are interpolated to obtain values at rounded-off shape coefficients. At all shape coefficients, there is a maximum density which is achieved at a die diameter to particle size ratio of about 20. This is in accordance with the accepted practice of granulating to about 20 mesh for normal tablets. The maximum is flat so that no great change in mean weight will occur if the granule size is changed within reasonable limits. On the other hand, the

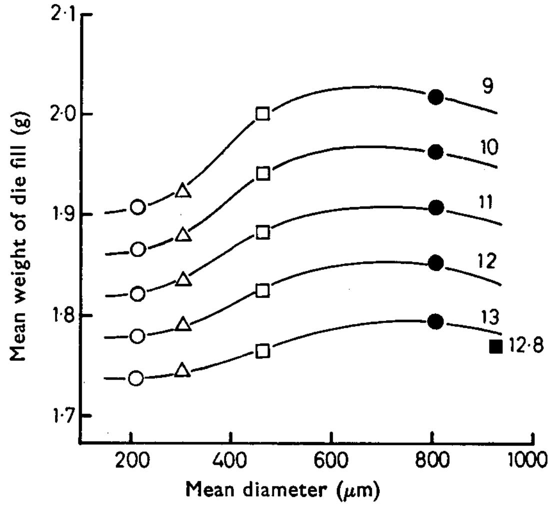

FIG. 4. The effect of particle size on the mean weight of material in the die for various shape coefficients. Key as for Fig. 2. The single point ■ represents restricted measurements made at a particle size of 925 μm (see text for details). Numbers represent values of the shape coefficient.

present work indicates that, other things being equal, a smaller granule will be better since the variation of weight will be less.

The reason for the maximum in the curves is not difficult to see. At small particle sizes, below about 200 μm, if the material will flow sufficiently well to enter the die it will have a constant bulk density. As the particle size increases, the energy or momentum obtained by each particle as it falls from the feed shoe into the die will increase. Macrae & Gray (1960) have shown that increasing the intensity of deposition of particles during pouring gives greater energy to each particle and allows rearrangement to occur in the surface layers of the bed as it builds up. The rearrangement allows increased packing density. This effect increases the bulk density of material in the die until a point is reached at which the particle size becomes appreciable compared with the size of the die cavity. This leads to restriction on the amount of rearrangement which can occur, and the restriction begins to be felt at a point where less than 10 or 12 particles can be fitted along the die diameter. The bulk density of the die fill then decreases.

The isolated point at the lower right side of Fig. 4 was obtained by making some measurements with 16/18 mesh sand, mean diameter 925 μm, with a shape coefficient of 12·84, the mean die fill being 1·77 g. This material was fine gravel rather than sand; some of the particles in it were agglomerates and were certainly not pure silica. However, the measurement does give some additional evidence for the general correctness of the trend of the lines in the graph.

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