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Characterization of the sphericity of particles by the one plane critical stability

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Abstract—A method has been developed to characterize the roundness of particles in terms of the angle necessary to tilt a plane such that the particles would roll, the 'one plane critical stability'. The method is based on determination of the centre of gravity of the particle from a digitized image of the coordinates of its outline and computing the angle necessary to incline a plane such that the centre of gravity moves outside the boundary of the particle. The method is particularly applicable to differentiate between the various shapes formed during the production of spherical granules by extrusion/spheronization.

The assignment of a size characteristic to a particle is a function of the method used to measure the particular dimension involved and a series of techniques is available to describe particle size (Allen 1981). The choice of which technique to apply to size characterization in industrial particle technology is closely associated with the ultimate use of the particles, allowing critical dimensions to be described. The characterization of particle shape is a related problem. However, because of its complexity of definition and interaction with size description, shape is a less well determinable characteristic. Reviews of shape characterization clearly show that no single method provides an accurate all-embracing definition of shape, e.g. Beddow et al (1976). It is important for industrial application that, as is the case with size, the method of describing shape must have a practical relevance to in-use situation. Such problems arise with the evaluation of the pharmaceutical process of producing spherical granules by extrusion/spheronization.

Extrusion/spheronization is now an accepted method for the preparation of dense spherical pellets—spheroids. Although a patent application for the process was initially filed in 1964, it was not until 1970 with the publication of two review articles (Reynolds 1970; Conine & Hadley 1970) that the process became widely known. However, little fundamental work on the process has been undertaken, due, in part, to the lack of methods available to characterize and quantify the various stages of the process. A major problem in this respect has been the quantification of the shape changes that occur during spheronization. Indirect methods, such as tapped density and drag diameters are too insensitive to measure subtle differences in shape, and are poor methods for differentiating between size and shape change effects. The alternative, observation methods,—direct measurement (Herdan 1960; Heywood 1947); shape comparison (Crayton 1974) or rotating vector methods (Beddow et al 1977) were developed to characterize the indentation of irregular particles and, as such, they are either unsuitable for detecting changes in smooth holographs, or too complex and painstaking to measure on the quantity of granules that are required to characterize a batch of spheroids. This paper proposes a new approach to shape characterization, particularly applicable to assessment of the spherical form of particles.

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Theory

An object resting on a plane will move if that plane is tilted until it reaches an angle at which the centre of gravity of a particle profile is moved outside the particle. The angle to which the plane must be raised before that particle will move will be defined as the 'one plane critical stability' (OPCS (Fig. 1)). If a

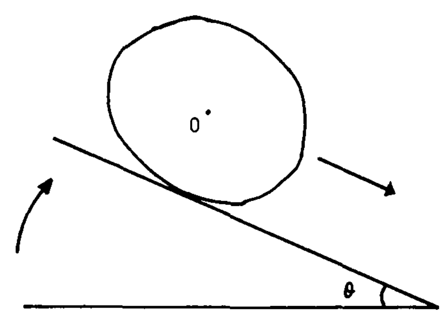

FIG. 1. The angle θ of a plane must be raised through to make a particle move from its critical stability 0 = centre of gravity.

particle is observed in its plane of maximum stability, e.g. for an ellipse, the worst possible case is always considered lying along the longest plane. The two dimensional image has a total area, A, with N points on the boundary, dividing the image into equal area segments. The base of a typical triangular segment is the line joining the points u(j), u(j + 1) which is assumed to be a straight line (Fig. 2). Point u(j) is a point on the complex plane with

$$u(j) = x(j) + iy(j). \quad (1)$$

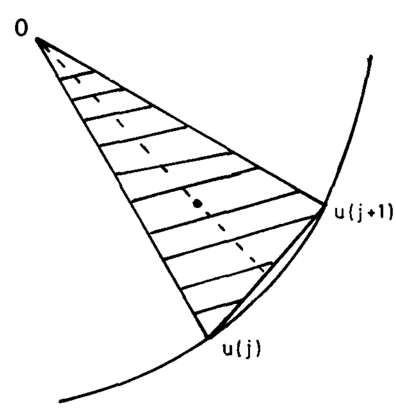

FIG. 2. One equal area segment of particle profile, 0 = centre of gravity.

$$\bullet = \frac{u(j) + u(j+1)}{3}$$

If the N points (u(j)) around the boundary of the outline have a mean of zero, i.e. $\bar{u} = 0$

$$\text{or} \sum_{J=0}^{N-1} x(j) = 0 \qquad (2)$$

$$\text{and} \sum_{J=0}^{N-1} y(j) = 0 \qquad (3)$$

and if each triangle between the origin and adjacent point u(j) and u(j + 1) has an equal area, then the centre of area is at the origin. This can be proved as follows:
The location of the centre of area of a triangle lies two-thirds of the way along the line joining the origin to the centre of the opposite side (true for all triangles). Therefore—

$$u(\text{c. of g. of } \Delta j) = \frac{2}{3}\frac{u(j) + u(j+1)}{2} \qquad (4)$$

$$= \frac{(u(j) + u(j+1)}{3}$$

As there are N such triangles of equal area, the centre of area of one object is given by

$$u(\text{c. of g.}) = \frac{1}{N}\sum_{J=0}^{N-1}\frac{(u(j) + u(j+1)}{3} \qquad (5)$$

$$= \frac{2}{3}\bar{u}$$

but $\bar{u}$, the mean of all (j) is zero (at the origin) therefore u(c. of g.) = 0, i.e. at the origin.

As the granule rolls down a plane inclined at θ from the horizontal, from a position where point u(j) is in contact to a point where u(j + 1) is in contact (Fig. 3), the greatest possible increase in the centre of gravity is from

|u(j)|—maximum height at the centre of gravity with point u(j) in contact (hj)

$$\text{to} \quad \underset{\substack{\text{new weight}\\ \text{of c. of g.}\\ \text{(uj)}}}{|u(j+1)|} - \underset{\substack{\text{fall of incline when rolling}\\ \text{down}}}{|u(j) - u(j+1)|\sin\theta(= hj + 1)} \qquad (6)$$

if hj + 1 > hj granule will not roll

& if hj + 1 = hj granule will just roll

the granule will roll for all points

u(j) if

$$\sin\theta > \left|\frac{(u(j+1)| - ||u(j)|}{u(j) - u(j+1)}\right| \qquad (7)$$

critical angle is given by

$$\theta = \arcsin \max\left(\left|\frac{u(j+1)| - |u(j)|}{u(j) - u(j+1)}\right|\right) \qquad (8)$$

N.B. $|u(j)| = ((x(j))^2 + (y(j))^2)^{\frac{1}{2}}$

The more difficult it is theoretically for the particle outline to roll, the greater the angle at inclination of the plane must be. The angle θ therefore gives a direct measure of the critical stability of the granule—the more elliptical the particle, the more stable it is, therefore the greater the angle.

Methods

The objects to be examined were placed on a microscope slide and viewed via a Camera-lucida side arm of a microscope (Vickers M75) so that the image could be traced on a

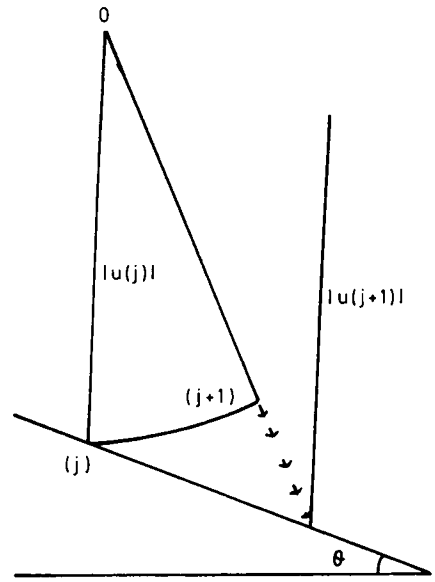

FIG. 3. Movement of the centre of gravity of particle 0 = centre of gravity.

computer-linked digitising tablet (Apple II). The outline of the objects was traced with a light pen and the digitized signal fed onto a floppy disc. (Groups of data for 23 particles could be stored on each disc.) The information was processed by computer to calculate the value of the OPCS, based on equation 8, dividing each particle into 32 equal area segments. The digitized information from the graphics tablet was used to determine the (perimeter)^2/area and could also be processed to determine the ellipticity and circulatory factors described by Proffitt (1982). Tapped bulk densities were determined by the method described in B. S. 1460/1967. Representative samples of a batch of granules were obtained with a spinning riffler (Microscal Ltd.).

Results and discussion

An evaluation of the number of granules which are required to provide a representative sample was undertaken collecting data on one, two, three, four and five sides of the disc, which represent 23, 46, 69, 92, 115 particles. The results in Table 1 illustrate that reproducible values for OPCS were obtained and that there was no significant difference between the largest and smallest mean value for the OPCS as assessed by a t-test. To standardize the procedure, therefore, data were

Table 1. The mean OPCS of granules from the same batch.

No. of granules per sample	Mean OPCS (degrees)	s.d.
23	53·4	8·6
46	53·6	9·8
69	53·4	10·0
92	52·2	10·0
115	51·7	10·2

collected on two sides of a single floppy disc, representing 46 particles. This provided an isolation of the data for each particle and appears to provide a value which was representative of a sample and was adopted throughout all further investigations.

To evaluate the usefulness of the OPCS as a characterization of the process, three typical shapes were separated from the granules by stopping the spheronization process at different times. The typical shapes observed are the cylinders of the early stages which occur as the extrudate is chopped, dumb-bells which are found as the cylinders are compressed along the axis and spheroids which are the product of a successful process. The results for shape characterization by (a) (perimeter)^2/area, (b) ellipticity (Proffitt 1982), (c) circularity, (Proffitt 1982) and (d) OPCS are given in Table 2.

Table 2. Shape factors of some typical granules formed during spheronization.

Shape factor	Shape	Mean	s.d. (σ)
P^2/A	Cylinders	15·314	0·958
	Dumb-bells	14·110	0·694
	Spheres	13·420	0·235
Ellipticity	Cylinders	0·9992	0·001
	Dumb-bells	0·9988	0·001
	Spheres	1·0000	0·000
Circularity	Cylinders	0·9550	0·016
	Dumb-bells	0·9863	0·009
	Spheres	0·9980	0·001
OPCS	Cylinders	41·918	10·041
	Dumb-bells	33·030	7·853
	Spheres	17·147	3·382

These clearly demonstrate the ability of the value OPCS to differentiate between the various stages of the process whereas the other three methods are relatively insensitive.

An equally clear demonstration of the insensitivity of tapped bulk density to monitor the process is shown in Table 3, where samples of granules taken after various residence times of the spheronization process are assessed.

The value of OPCS therefore provides a relatively simple and effective quantification of shape of particles observed during the spheronization process and, as such, allows the characterization of process variables and provides a possible quality control procedure.

Table 3. The change in tap density and OPCS with residence time.

Residence time (min)	Change in tap density (% drop)	OPCS (degrees)
1	0·3	54·61
2	0·2	35·65
5	0·2	22·63
10	0	23·73
15	0	19·56
20	0	20·82

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