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Some angular properties of magnesia and their relevance to material handling

T. M. JONES AND N. PILPEL

By measuring the angular characteristics of magnesia over a range of particle sizes from 30–3000 μ, it has been possible to determine the factors which influence the shapes of heaps, cones and wedges of the material, formed under a variety of experimental conditions. The relevance of the measurements to quality control, flowability, hopper and chute design and material handling is discussed.

WITH the increasing use of particulate solids in pharmaceuticals, interest has been stimulated in the angular properties of materials in granular and powdered form.

Several authors have outlined the methods available for measuring these properties (Zenz, 1957; Train, 1958; Brown, 1961; Pilpel, 1964) but the choice of a method for use in control investigations is often arbitrary. Consequently, the results obtained bear little relationship to the handling characteristics of the material concerned. Furthermore, little is known of the interrelation between the results obtained by the various methods.

In the present work an attempt has been made to compare the results of a number of these tests for one material over a range of sizes, the physical properties and flow characteristics of which are now established (Jones & Pilpel, 1966a, b). From these results it has been possible to evaluate the techniques critically and to note their application to handling characteristics.

Experimental

Determinations were carried out on close cut sieve fractions of free flowing magnesia, in the range 30 μ to 3000 μ. The physical properties of these fractions have been previously reported (Jones & Pilpel, 1966a).

ANGLES OF REPOSE

Natural angles of slip, θ_1, were measured by a fixed cone method using 50 g of powder and funnels with an angle of 45°; orifice diameters (1) 0·4 cm, (2) 0·7 cm; stem lengths 5·6 cm, terminating 3 cm above a horizontal surface (Train, 1958; Nash, Leiter, Johnson, Stender & Zeller, 1963; Kawai & Hasegawa, 1964). Static angles of repose, θ_2, were measured by the fixed base and cylinder method (Pilpel, 1964) using bases of diameter 3·69 and 9·00 cm with loads of 35 g and 400 g of powder respectively. Consolidated static repose angles, θ_3, were obtained by measuring a two dimensional drained heap in a rectangular box with a glass front (Jones & Pilpel, 1966b), the wedge length being 4·8 cm.

The angles were calculated from a knowledge of the heights, h, and the radii, r, of the heaps by the formula

$$\theta = \tan^{-1} h/r$$

From the Department of Pharmacy, Chelsea College of Science and Technology (University of London), Manresa Road, S.W.3.

where h and r were measured with a cathetometer. Between 6 and 12 replicates were determined for each sample, the results being expressed as mean values or as a range when the values were very varied.

In some instances it was convenient to measure the length of the sides of the cones, s, using dividers, readings being taken at four positions separated by 90° of arc. The angles in this case were calculated from

$$\theta = \cos^{-1} r/s$$

It was also possible to calculate the natural angle of slip from a knowledge of the mass, M, the bulk density, ρ_B of the sample, and the diameter of the cone base, d. The following formula was used:

$$\theta = \tan^{-1}\frac{24M}{\pi\rho_B d^3}$$

[This is derived from the fact that the volume of a cone $\equiv \frac{1}{3}$ the volume of a cylinder of the same height standing on the same base]. With this formula, difficulties in measuring the height of cones because of flattened apices or small values of h, can be eliminated.

ANGLES OF FRICTION

These were determined by measuring the angle from the horizontal at which shearing or slipping occurred in heaps or beds of material. Three methods were used:

(1) Tilting a heap, formed by the fixed cone method as above, and noting the angle of inclination, α_1, at which shearing occurred (Lowes & Perry, 1965). This yielded a dynamic internal shear angle.

(2) Preparing beds of material by dredging from a height of 5 cm onto a glass plate (cleaned with chromic acid), and weighing the quantity of material falling off at each angle of elevation (Krishna & Rao, 1963). The angle of sliding friction, α_2, was taken as the point of maximum inflexion in plots of weight versus angle. Measurements were made on beds several particles thick, α_{2_1}, and on beds one particle thick, α_{2_2}. The mean angle for single particles, α_{2_3}, was also noted.

(3) Rotating the material in a drum of diameter 13·6 cm, width 2·7 cm, at a speed of 2 or 4 rpm (Franklin & Johanson, 1955). Four angles were measured—the static angle for loosely packed, α_{3_1}, and compacted, α_{3_2}, samples, the surface kinetic angle, α_{3_3}, and the internal kinetic angle, α_{3_4}.

The results of the measurements on the different size fractions are summarised in Figs 1, 3 and 5.

Smooth curves have been drawn through experimental points although it does not necessarily follow that the angular characteristics are a unique function of particle size.

Discussion

The forces involved in the formation of heaps, cones and wedges of particles are mechanical, gravitational and interparticulate (frictional and cohesive) and attempts have been made to correlate the angles of the heaps with these fundamental forces (Dawes, 1952; Lowes & Perry, 1965).

Fig. 1 shows that the natural angle of slip, θ_1, remains appreciably constant over the range of particle sizes investigated. This method is essentially a measure of the packing characteristics and hence the shape and rugosity of the material. Since θ_1 is formed under kinetic conditions, it may be assumed that in this measurement the effect of interparticulate forces is largely overcome.

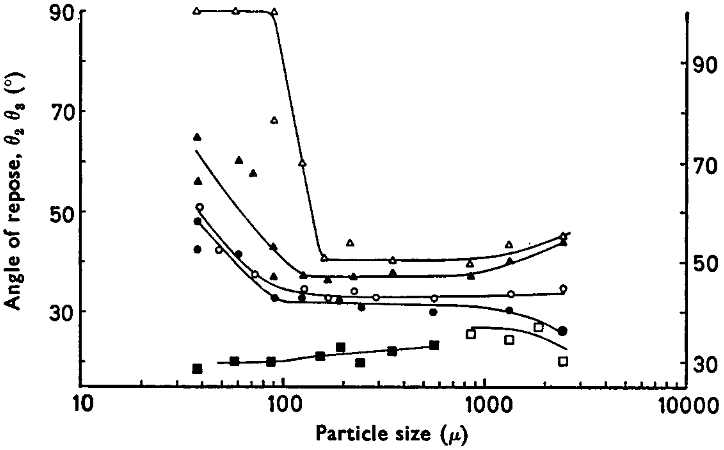

FIG. 1. The change in angle of repose with particle size. ■, θ_1 using funnel (1); □, θ_1 using funnel (2); ●, θ_2 (large base); ○, θ_2 (small base); ▲, θ_3 (loosely packed); △, θ_3 (consolidated).

The cones formed in the static methods are produced by the shearing forces in the mass surrounding them. At small particle sizes, the interparticulate forces oppose these shearing forces and consequently more material is held in the heap than would otherwise be the case. This may explain why the angles θ_2 and θ_3 show a distinct rise in value as the particle size decreases below 100 μ (Fig. 1).

The consolidated angles, θ_3, are even higher than the static angles of

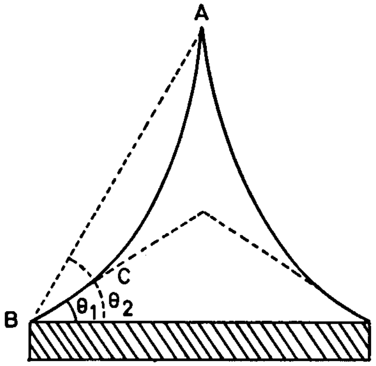

FIG. 2. Diagrammatic representation of false angle of repose.

repose, θ_2, since in the closer state of packing the forces between the particles are enhanced and can act more fully.

Heaps formed by material possessing strong interparticulate forces are often irregular (Craik, 1958) and seem to consist of a cone of angle θ_1 surmounted by a peak (Fig. 2). Similar observations have been reported by Dawes (1952). It is possible that these false peaks are produced by the weight of material enclosed by ABC (Fig. 2) exerting a shearing force greater than the force required to hold the material in position. The magnitude of such a shearing force is reflected in the values of α_1 (Fig. 3), the angle (about 10°) being small by comparison with the angle ABC (between 15° and 30° for $\theta_2 \simeq 45°$ and 60° respectively). The limiting angles at which monodispersed particles roll down slopes of randomly packed spheres of the same size are between 19·3° and 45° (Train, 1958). The upper limit (45°) is equivalent to the value of $\theta_1 + \alpha_1$, i.e. the angle above which false peaks are formed.

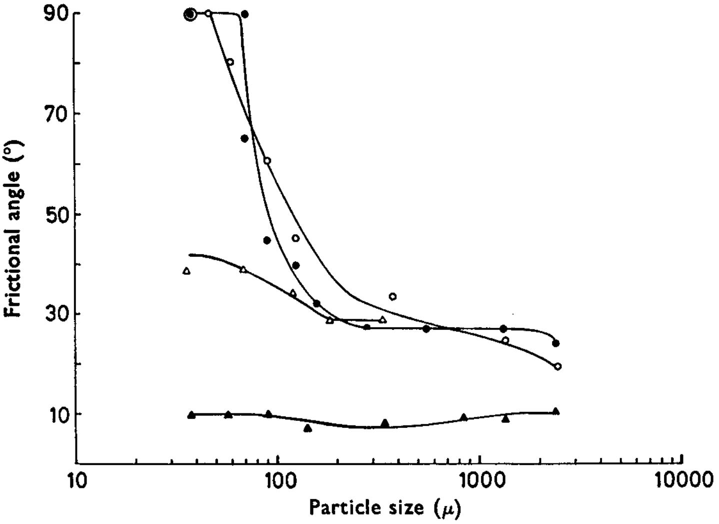

FIG. 3. Variation in frictional angles with particle size. ▲, α_1; △, α_{2_1}; ○, α_{2_3}; ●, α_{2_2}.

Since the false peak may be attributed to the influence of interparticulate and shearing forces, it may be possible to estimate their magnitude from a knowledge of the mass of material in the peak together with the influence of mechanical and gravitational forces on the sample. However, the height of the peak may be affected by disruptive shearing forces during formation and this would be a complicating factor.

Angles of friction, α_{2_1} and α_{2_2}, for beds of solids under dynamic conditions, also increase at small particle sizes (Fig. 3). The angle of friction α_{2_1} is a combination of the angle at which material slides over itself and over the supporting surface, whereas α_{2_2} is the apparent angle at which it slides on the glass only: it can be seen that $\alpha_{2_2} > \alpha_{2_1}$. In characterising material, it is often necessary to know both α_{2_1} and α_{2_2} since the former can be related to the internal frictional characteristics of beds and the latter to the adhesion of material to a given surface.

Beds of fine material, several particles thick, shear at α_{2_1} to produce beds one particle thick; thus α_{2_1} and α_{2_2} can both be determined in the same test (Fig. 3), two points of inflexion in the weight versus angle plots being observed.

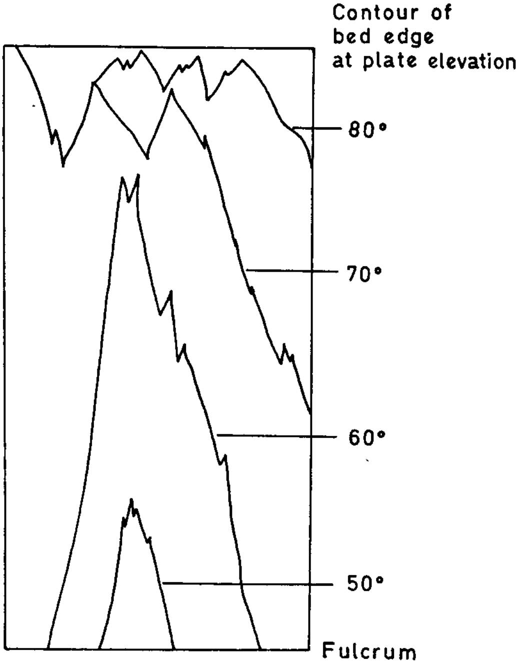

FIG. 4. Triangular shear pattern on tilting bed of single particles. 71μ diameter.

The values of α_{2_2} correspond closely to the mean angles of friction of individual particles α_{2_3} from a given sieve cut. In any bed of single particles there will be a distribution in the values of α_{2_3} due to slight differences in rugosity, shape and size. Thus on tilting, the particles having the lowest value will slide first. If the weight of these particles is sufficient, their downward motion will disturb the particles immediately below them and cause these to slide also. This kinetic disturbance spreads outwards and downwards producing a triangular shear pattern (Fig. 4). As tilting continues, other particles situated higher in the bed also cause propagating collisions with consequent triangular patterns. Similar conditions would arise in beds of mixed particle size or where aggregates have formed in monodispersed beds.

The static angles α_{3_1}, α_{3_2} vary similarly to θ_2, θ_3 (Fig. 5). α_{3_1}, α_{3_2} are the maximum angles which the surface subtends to the horizontal before movement occurs and this fits the broad definition of angle of repose as the angle of friction taken up by a granular solid about to slide upon itself (Fowler & Wyatt, 1960). The relationship between α_{3_1}, α_{33} and α_{3_4} fits the proposed correlation of Franklin & Johanson (1955); it is thus possible to estimate the internal kinetic angle from a knowledge of the static or surface kinetic angle for sieve fractions where the internal angle is difficult to measure.

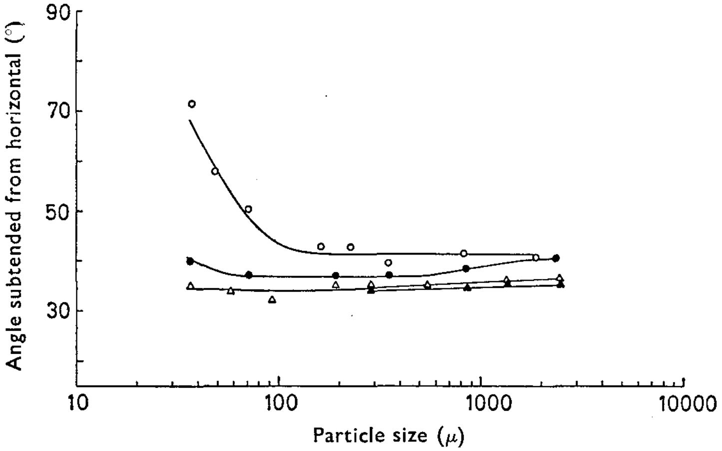

FIG. 5. Variation in angle of friction (α_3) with particle size, ●, α_{3_1}; ○, α_{3_2}; △, α_{3_3} ▲, α_{3_4}.

It can also be seen from Fig. 5 that the value of α_{3_3} does not vary appreciably with particle size. Thus under kinetic conditions the influence of interparticulate forces is outweighed by the kinetic energy of the particles. As the value of the surface kinetic angle is essentially similar to the natural angle of slip, θ_1, the original postulate that in measuring θ_1 interparticulate forces are overcome, appears to be justified.

Application of the above principles to material handling

CONTROL

Angles of repose can be successfully applied to control excessive moisture in powders and granulations (Wolf & Hohenleiten 1945; Craik & Miller, 1958; Fowler & Wyatt, 1960).

Since the presence of fine particles in granulations produces changes in the value of θ (Nelson, 1955; Craik, 1958; Pilpel, 1964) methods which do not cause segregation of material may be used as a control over excessive fines produced during handling.

The extent of segregation can be observed by forming a two-dimensional heap, from a wedge shaped hopper with a slotted outlet. Coarse material with a low angle of friction, α_{2_3}, rolls and slides towards the base (Fig. 6), whilst fine material possessing a high angle of friction remains in the central core. As the heap is built up, distinct striations are observed.

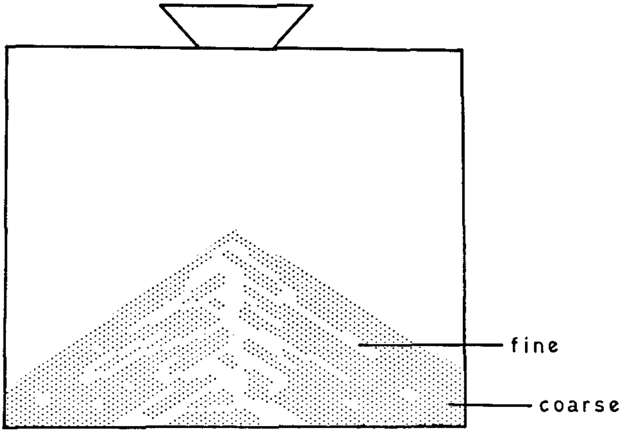

FIG. 6. Two dimensional heap of coarse and fine particles (schematic).

FLOWABILITY

An increase in the static angle of repose, θ_2 or θ_3, reflects a decrease in flow rate (Jones & Pilpel, 1966b).

In general if $\theta < 40°$ a material will flow easily through orifices and from hoppers. When the angle exceeds 50° flow takes place with difficulty and aggregation, "rat holing" or bridging may occur.

Because heaps with $\theta_2 > 40°$ often possess false peaks which may be disturbed during formation, it seems unwise to rely to any extent upon quantitative values in these instances, although qualitatively, dramatic variations in the angle of repose can be identified easily. The values of α_{3_1} however, are more reproducible since they apply to free surfaces. Consequently the values can be used directly and for comparisons between powders.

Clearly if the material is compacted by vibration or ramming, the angles θ_3 and α_{3_2} become relevant (Figs 1 and 5). These are higher than θ_2 or α_{3_1}, particularly at the small sizes. The problems of handling fine particles and of making them flow are often very difficult to overcome as a result of compaction.

Several authors have incorporated values for angle of repose or angle of friction in equations for calculating flow rate (Takahasi, 1935; Shirai, 1952; Zenz, 1962; Kawai & Hasegawa, 1964). From the present work and that of others (Train, 1958; Brown, 1961) it is obvious that these characteristics of a powder are not easily and uniquely definable. Although it may be advisable to include qualified values in flow data as a guide to material characteristics, it appears unwise to use them directly in quantitative flow equations.

HOPPER AND CHUTE DESIGN

The drained angle of repose, θ_3, implicitly defines the dead space in a horizontal based hopper after discharge. However, the static region during flow has a shear angle much greater than θ_3 (Brown & Richards, 1965) and designing hoppers with base angles $\equiv\theta_3$ does not necessarily give the best flow improvement.

The angles of friction α_1, α_2 and α_3 are useful in deciding the slope of a chute for conveying material. Caution should be observed in interpreting laboratory tests using constructional materials which differ from those used in the plant. Fowler & Chodziesner (1959) have developed an expression to perform such conversions.

FINE PARTICLE HANDLING

The methods used in these investigations are only of real value for relatively free flowing material since only these form good cones and heaps.

For fine particles $<50\,\mu$, static and dynamic methods produce heaps which do not possess well defined angular properties; angles of friction reach a limiting value of 90° and the free surfaces of rotating beds of fine particles are uneven. In these cases, the split plate method (Dawes, 1952; Shotton & Harb, 1966) and the Jenike Shear Cell (Ashton, Cheng, Farley & Valentin, 1965; Williams & Birks, 1965) have been successfully applied to studies on a variety of different materials.

In conclusion it remains to point out that the relationships between the various angles quoted in this paper are applicable only to the magnesia used. Further work on other materials is required to establish the generality of these relationships.

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