J. Pharm. Pharmac., 1974, **26**, *Suppl.*, 11P–15P *Received January*, 31, 1974

Shearing and tensile tests on mixtures of pharmaceutical powders

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Shearing and tensile tests provide information about the mechanical properties of mixtures of fine pharmaceutical powders. They might be used for predicting their flow and compressional characteristics during handling and in the production of capsules and tablets.

Basic information about the flow and failure properties of dry powders is obtained by consolidating them to different packing densities and then carrying out shearing and tensile tests. Parameters are derived from the resulting yield loci which provide information on how the powders are likely to behave during handling and use (Jenike, 1961, 1964; Ashton, Cheng & others, 1965; Pilpel, 1971; Stainforth & Berry, 1973).

Many of the powders encountered in pharmacy and in the manufacture of cosmetics and detergents are mixtures. They consist either of one material in a range of sizes (e.g. milled drugs with a size range between about 2 and 50 μm), or of different materials (e.g. granulations for tablets and capsules which may contain up to half a dozen different ingredients in powder form). To date, there has been very little reported on the results of shearing and tensile tests on powder mixtures (Kočova & Pilpel, 1973 a, b).

Recently, we have suggested that the same tests that are now made on single powders might also provide useful information about the properties of powder mixtures.

The purpose of the present paper is to summarize some of our recent findings on model mixtures of powders and indicate how they might be applied for predicting the properties of some mixtures that are used in the pharmaceutical and allied industries.

Tensile strength parameters

On the basis of a theory developed by Cheng (1968), one can express the tensile strength of a mixture of two powders, designated A and B, by the equation (Kočova & Pilpel, 1973 a):

$$T = h\left(t_0 - \frac{\bar{d}}{3}\left[\frac{\rho}{\rho_0} - 1\right]\right)\frac{a\,b\,c\,\rho}{2\bar{v}\,\rho_s}M\left[\bar{s}_A y^2 + 2\bar{s}_{AB}y(1-y) + \bar{s}_B(1-y)^2\right] \quad (1)$$

and the tensile strength of a mixture of three powders, designated X, Z and W by:

$$T = h\left(t_0 - \frac{\bar{d}}{3}\left[\frac{\rho}{\rho_0} - 1\right]\right)\frac{a\,b\,c\,\rho}{2\bar{v}\,\rho_s}M\left\{\bar{s}_X x^2 + \bar{s}_Z z^2 + \bar{s}_W\left[1-(x+z)\right]^2 + 2\,xz\bar{s}_{XZ} + 2x\bar{s}_{XW}\left[1-(x+z)\right] + 2z\bar{s}_{ZW}\left[1-(x+z)\right]\right\} \quad .. \quad .. \quad (2)$$

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The symbols are defined later.

The tensile strength depends on the "range", t_0, and on the so called "strength", Σ_0, of the force h (which is a function of t) between neighbouring particles, and it has been shown that for several powders and powder mixtures one can write

$$\frac{t_0^3}{\Sigma_0} h = \Phi\left(\frac{t}{t_0}\right) \qquad \ldots \quad \ldots \quad \ldots \quad (3)$$

where $\Phi\left(\frac{t}{t_0}\right)$ is the same function for all the materials.

The value of Σ_0 for any powder or mixture of powders can be compared with that of any arbitrarily selected "standard powder" by assigning to the latter a value of $\Sigma_0 = 1$ and then employing the relationship

$$\frac{\Sigma_{0A}}{\Sigma_{0B}} = \frac{t_{0A}^3 h_A}{t_{0B}^3 h_B} \qquad \ldots \quad \ldots \quad \ldots \quad (4)$$

The detailed procedure for deriving Σ_0, t_0 etc. is more fully described elsewhere (Cheng, 1968; Kočova & Pilpel, 1973a).

We have shown that for a variety of binary and ternary mixtures the values of tensile strength predicted by means of equations 1 and 2 are in good agreement with the values obtained in actual tensile tests.

Table 1 shows the agreement for a series of binary mixtures, each at one particular packing density; Table 2 shows the agreement for a ternary mixture at different packing densities. (The code numbers of the mixtures, enabling them to be identified, are the same as in our previous publications, Kočova & Pilpel, 1973 a, b).

Table 1. *Comparison between the calculated and the observed values of the tensile strengths for binary mixtures.*

Mixture code* No.	Packing density ρ/ρ_s	T(predicted) g cm^{-2}	T(observed) g cm^{-2}	Error %
M.1	0·355	0·83	0·62	+25
M.2	0·283	1·77	1·44	+18
M.3	0·225	1·46	1·62	—11
M.4	0·259	0·99	1·22	—22
M.7	0·240	1·55	1·21	+22
M.11	0·192	1·22	1·48	—21
M.12	0·179	1·29	1·67	—29
M.13	0·222	1·46	1·42	+2·7
M.16	0·420	1·15	1·11	+3·5

Table 2. *Comparison between the calculated and the observed values of the tensile strengths for ternary mixtures.*

Particle density, ρ_s, g cm^{-3}	Packing density, ρ/ρ_s	$\frac{\rho_o}{\rho_s}$	ρ_o, g cm^{-3}	t_o, μm	Σ_o (relative to $CaCO_3$)	T (predicted) g cm^{-2}	T (observed) g cm^{-2}	Error %
1·525	0·715	0·66	1·007	2·05	30·2	3·47	3·16	+ 8·9
	0·731					3·76	4·21	—17·5
	0·741					5·02	5·26	— 4·8
	0·851					17·80	13·42	+24·8

Shear strength parameters

Detailed studies (York & Pilpel, 1972; Kočova & Pilpel, 1973 a,b; Walton, 1973) have led us to think that the angle of internal friction, Δ, is a valuable parameter for comparing the shearing properties of both "simple" and "complex" single powders and mixtures of powders (Williams & Birks, 1967).

In all cases, except those in which allowance has to be made for the effects of particle shape (Walton, 1973), Δ is related to the particle size by the expression

$$\tan \Delta = D - K \log \bar{d} \qquad (5)$$

For mixtures, Δ also depends on the weight composition of the mixture and this is illustrated for some representative binary mixtures in Fig. 1. However, there does not at present appear to be any predictable connection between the shapes of these curves and the properties of the individual components in the mixtures.

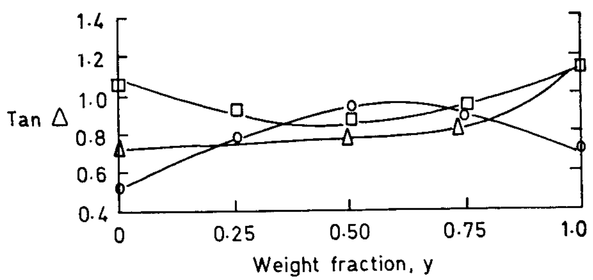

FIG. 1. Tan Δ versus weight fraction, y, in binary mixtures. (□) Samples L1, C2, M13, M14, M15. (○) Samples C6, L7, M16, M17, M18. (Δ) Samples L1, U6, M10, M11, M12.

Equation 5 shows that D is the value of tan Δ when the (mean) particle size, $\bar{d}$ = 1 μm and if one plots the values of D against log t_0, one obtains a straight line, as shown in Fig. 2. This result supports a previous suggestion (Cheng, Farley & Valentin, 1968) that there may be a connection (which depends on the lattice structure of a powder and the surface geometry of its particles) between the interparticle forces in tension, h, and those in shear, g.

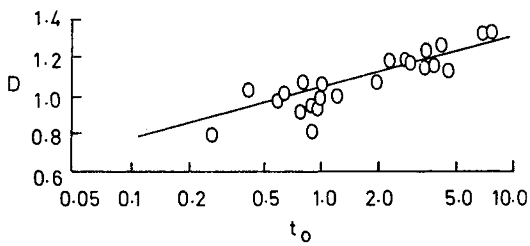

FIG. 2. Plot of D versus log t_0.

The parameters Σ_0, t_0 and Δ vary significantly from one powder to another and depend on the chemical nature and particle size and shape of the components (Walton, 1973). The values alter as the powders are mixed together in different proportions by weight, due to the changes that occur in the interaction forces between the particles.

Table 3. *Calcium carbonate mixtures.*

Code No. of powder	C6	M1	M2	M3	C3
Σ_0	5·2	13·1	24·9	25·0	5·2
t_0	1·3	4·3	3·6	2·8	1·3
Δ	0·50	0·93	0·91	1·0	0·95

Table 3 illustrates the effects on the parameters of mixing one size fraction of calcium carbonate with a different size fraction of the same material in various proportions.

Table 4 illustrates the effects of mixing one size fraction of calcium carbonate with a different size fraction of lactose in various proportions.

Table 4. *Calcium carbonate lactose mixtures.*

Code No. of powder	C2	M13	M14	M15	L1
Σ_0	1·0	0·18	0·36	0·52	15·8
t_0	1·0	0·64	0·83	1·0	2·2
Δ	1·05	0·95	0·89	0·91	1·15

* Identification of powder systems from Kočova & Pilpel (1973).

	Single materials		Binary mixtures		
	Particle diameter, $\bar{d}$ (μm)	Code No.	Components of mixture	Weight fraction of first component	Code No. of mixture
Calcium carbonate	1·52	C.1	C.6 + C.3	0·75	M.1
	3·75	C.2	C.6 + C.3	0·50	M.2
	9·80	C.3	C.6 + C.3	0·25	M.3
	13·05	C.4	L.6 + L.1	0·75	M.4
	25·0	C.6	L.6 + L.1	0·25	M.6
	0·25	U.1	U.6 + U.1	0·75	M.7
Ultramarine	1·90	U.4	U.6 + L.1	0·50	M.11
	1·89	U.5	U.6 + L.1	0·75	M.12
	3·88	U.6	C.2 + L.1	0·25	M.13
			C.2 + L.1	0·50	M.14
			C.2 + L.1	0·75	M.15
Lactose	4·14	L.1	C.6 + L.7	0·25	M.16
	17·74	L.6	C.6 + L.7	0·50	M.17
	30·2	L.7	C.6 + L.7	0.75	M.18

The parameters Σ_0 and t_0, being derived from tensile measurements, might be expected to relate to the compressional properties of powders and to the strengths of the compacts produced during a tableting process. But further experiments, involving the preparation of tablets, will be needed to test this suggestion.

The parameter Δ, on the other hand, is more likely to relate to the flow properties of powders and might thus be useful for predicting their behaviour in transportation, handling and mixing.

The following examples may be used to illustrate some of the potentialities of the parameter Δ:

When as little as 0·5% w/w of B.P. magnesium stearate was mixed with micronized griseofulvin, the latter's angle of internal friction decreased from 40° to 38° showing that the magnesium stearate was functioning as a glidant.

When 50% w/w of B.P. maize starch and 1·5% w/w of B.P. magnesium stearate were mixed with two different grades of B.P. lactose, the angle of internal friction of the coarse mixture was 9° and of the fine mixture was 13°. This showed that, as expected, the former had significantly better flow properties than the latter.

These results indicate that shearing and tensile tests might provide useful information on the compression and flow properties of mixtures of powders for use in pharmaceutical and other applications.

List of Symbols.
a, b, constant ratios; A, designation of powder component; B, designation of powder component; c, co-ordination number; $\bar{d}$, mean particle diameter μm; D, value of tan Δ when $\bar{d} = 1$; g, interparticle force in shear; h, interparticle force in tension; K, constant; M, index of mixing; $\bar{s}$, mean surface area of particles; t, particle surface separation μm; t_0, "range" of interparticle force μm; T, tensile strength g cm^{-2}; $\bar{v}$, mean volume of particles; W, designation of powder component; x, weight fraction of powder component X; y, weight fraction of powder component B; z, weight fraction of powder component Z; Σ_0, "strength parameter" of interparticle force; ρ, bulk density of powder g cm^{-3}; ρ_0, packing density of powder when $T = 0$; ρ_B, particle density of powder g cm^{-3}; Δ, angle of internal friction; Φ, functional relationship.

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