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TENSILE AND SHEAR TESTING OF SOME PHARMACEUTICAL POWDERS

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ABSTRACT

Twelve pharmaceutical diluents have been characterized by determination of particle size distribution, tensile strength and the shear cell parameters cohesiveness (C), flow factor (FF) and the effective angle of friction (δ). Nine materials possessed a size distribution approximating log-normal. Avicel and the starch samples exhibited highest cohesiveness and lowest flow factors. The correlation between C and FF was good, but T was unrelated to either. Values of $^C/T$ varied from 0.19 to 3.16. It is suggested that in many cases, estimates of T may not fit the yield locus at the corresponding bulk density because of different modes of consolidation in the tensile tester and shear cell.

Avicel was shown to be a simple powder, whereas StarX and Celutab exhibited some complex behaviour. Estimates of the shear index, n, as a measure of flowability for Avicel, StarX and Celutab were not in line with their flow factors.

INTRODUCTION

Tensile and shear testing of powders, originally applied to hopper and bin design (1-3), has recently been extended to prediction of flowability during processing (4-10). In the manufacture of tablets and capsules it is essential that diluents selected have good flow properties, otherwise high weight and dose variation will occur. From a shear cell fundamental measurements of the cohesiveness (c), effective angle of friction (δ) and flow factor (FF) for a powder can be obtained (2). Few measurements of such parameters or of tensile strength (T) have been carried out on pharmaceutical diluents as a means of gaining an index of their flowability.

Various authors have attempted to relate tensile strength (T) values obtained from a tensile tester to C values determined using the shear cell. Some workers noted that $^{C}/T$ for a number of bulk powders was approximately 2(11, 12). If this were so, tensile strength measurements would provide a very simple method of characterising a powder. However, other authors (13) have shown that $^{C}/T$ varies between 1.1 and 4, and Hiestand and Peot (14) observed that values of tensile strength for aspirin and sitosterols were much too large to be compatible with the use of T as an end-point of the shear yield locus.

In this paper, measurements of tensile strength and shear cell parameters for twelve pharmaceutical diluents are reported and compared. Problems in determination of these values are discussed, together with their possible application in pharmaceutical manufacturing.

EXPERIMENTAL MATERIALS

The powders, their manufacturers and some properties are listed in Table 1.

METHODS

Particle Densities

These were determined by liquid displacement using a specific gravity bottle and dioxane at 25°. The dioxane was presaturated with the particular material. Values are given in Table 1.

Particle Size Distributions

These were determined using an Endecott sieve shaker and sieves in the particle size range 90 - 710µm and an Alpine Air-Jet Sieve System in the range 10 - 90µm.

TABLE 1

Characteristics of Twelve Diluents

Diluent	Mnfr	Particle Density	Geometric Mean Particle Size Microns	Geometric Standard Deviation	% Moisture at 60% R.H.
Avicel PH101 (Microcrystalline cellulose)	F.M.C. (U.S.A.)	1.60	35	2.1	4.64
Cornflour (Starch)	Fielders (Aust.)	1.49	52	1.8	9.70
StarX 1500 (Starch)	Staley (U.S.A.)	1.49	32	2.1	9.41
Mobile Starch	Fielders (Aust.)	1.49	30	2.0	11.00
Lactose (Spray-dried)	Wyndale (N.Z.)	1.54	116	2.0	0.14
Lactose(Coarse Crystalline)	D.M.V. (Holland)	1.54	Fig. 1	Fig. 1	0.30
Lactose(Fine Crystalline)	D.M.V. (Holland)	1.54	Fig. 1	Fig. 1	0.40
Dipac(Sucrose/ Dextrin)	Amstar (U.S.A.)	1.52	283	1.3	0.40
Amerfond (Sucrose)	Amstar (U.S.A.)	1.53	70	1.8	0.72
Sugartab (Sucrose + ?)	Mendell (U.S.A.)	1.53	600	2.4	1.42
Celutab (Dextrose/maltose)	Mendell (U.S.A.)	1.41	282	1.4	9.31
Encompress (Dicalcium phosphate dihydrate)	Mendell (U.S.A.)	2.34	Fig. 1	Fig. 1	4.08

Tensile Strength Measurement

The apparatus used was the Warren-Spring Ajax tensile tester (15). Measurements were made after consolidating the powders to different bulk densities, using a normal load and a twisting action. The tensile strength reported is the mean of several determinations. New samples of powder were used for each measurement at each bulk density.

Shear Cell Studies

The shear cell used was of the Jenike (1, 2) type. Yield loci were determined for each of the powders, using a normal consolidating stress of 50 gcm^{-2}. In addition, for Avicel, Celutab and StarX, yield loci were determined using a number of normal consolidating stresses ranging from 30 to 75 gcm^{-2}. From each yield locus, values of C, δ and FF were obtained by the method of Jenike (2).

RESULTS AND DISCUSSION

The particle size distributions of nine of the materials approximated very closely to a log-normal distribution. Values of the geometric mean diameter and the geometric standard deviation are included in Table 1. The size distributions of Encompress and the coarse and fine crystalline lactose samples could not be approximated to log-normal, probably because of deliberate truncation by the manufacturers. Log-probability plots of their size distributions are shown in Fig. 1.

It is apparent that Avicel and the three starch samples have the lowest mean particle sizes and also wide distributions. It might be expected that their flowability would be relatively poor. The particle size range for optimum flow of many materials often occurs between 200-300µm. For this reason it is likely that Dipac, Celutab and Encompress would exhibit good flow properties. The size distributions of these materials is also quite narrow. Sugartab has a very high geometric mean particle size of 600um. Such a coarse material may flow rather poorly, especially

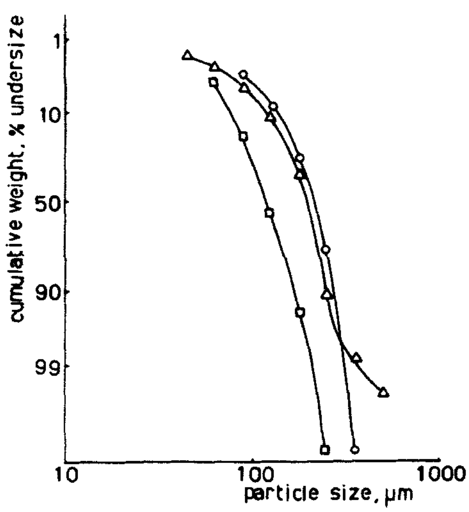

FIG. 1: Log-probability Plots for Encompress, Δ, Lactose (Coarse Crystalline), O, and Lactose (Fine Crystalline), □.

through fine orifices, because of mechanical interlocking of particles.

There are numerous modes of representation of the variation of tensile strength, T, with bulk density, ρ_B in the literature. Most frequently log T is plotted against ρ_B(12), log ρ_B(15), packing density ρ_B/ρ_p (16-18), or log packing density (11). There is no 'a priori' reason to expect linearity from such relationships, and only the log-log plots are frequently linear, probably because of the insensitive nature of this type of plot. In this work, log T is plotted against porosity, ε, given by $1-\rho_B/\rho_p$ (Figs. 2 and 3).

Porosity is a useful parameter for comparative purposes and it is apparent that for most materials, the plots show good linearity. Such a plot is useful to screen materials for suitability in tabletting. A good tablet vehicle would exhibit a low tensile strength at normal porosities, but tensile strength should increase dramatically as the powder is compacted. Avicel exhibits the highest slope, while StarX exhibits the lowest.

Fig. 4 compares the yield loci for StarX and Celutab at a normal consolidating stress of 50 gcm^{-2}. The tensile strength of each material is included. The intercepts on the shear stress

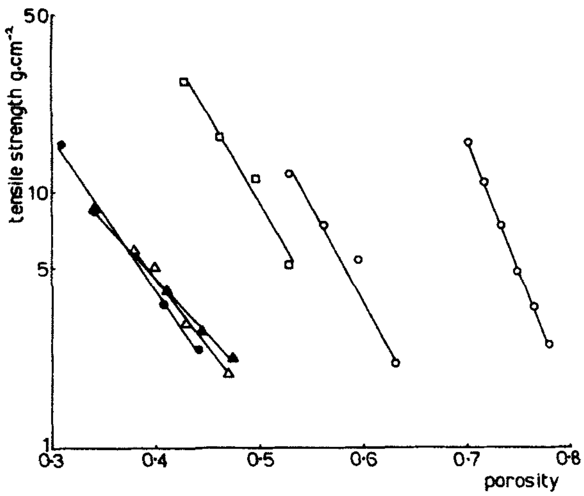

FIG. 2: Plot of Log Tensile Strength Versus Porosity for Avicel, O, Cornflour, O, Mobile Starch, □, Amerfond, ●, Sugartab, ▲, and Celutab, △

axis represent the cohesiveness of material, C, and it is apparent that the cohesiveness of StarX is approximately twice that of Celutab. Similarly the unconfined yield strength, f_c indicated by the intersection of the first Mohrs semi-circle with the normal stress (σ) axis is greater for StarX than Celutab. At higher normal stress values, the yield loci converge and the major consolidating pressure, σ_1, indicated by the intersection of a second Mohr's circle, is similar for both materials. The effective yield locus is the line drawn from the origin

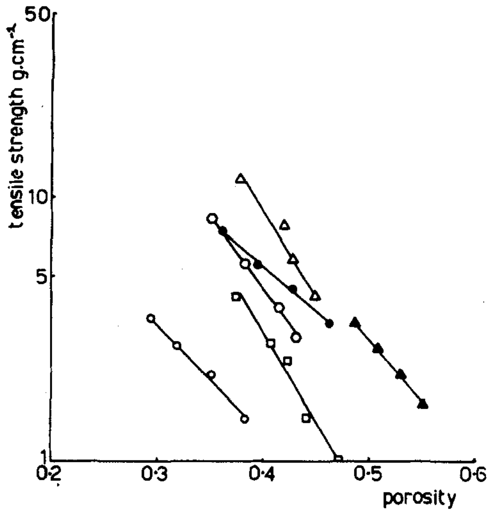

FIG. 3: Plot of Log Tensile Strength Versus Porosity for StarX, ●, Lactose (Spray-dried), O, Lactose (Coarse Crystalline), O, Lactose (Fine Crystalline), Δ, Dipac, □, and Encompress, ▲.

tangential to the second Mohr's circle and can be described by the effective angle of friction, δ. The values of δ for Celutab and StarX are obviously very similar.

Jenike, Elsey and Wolley (19) derived a parameter known as the flow factor (FF) given by $\sigma 1/f$ and suggested its use as an index of flowability. York (8) has suggested that $1/FF$ or fc/σ_1 may be a more suitable measure of flowability as it is in fact the unconfined yield strength per unit major consolidating pressure.

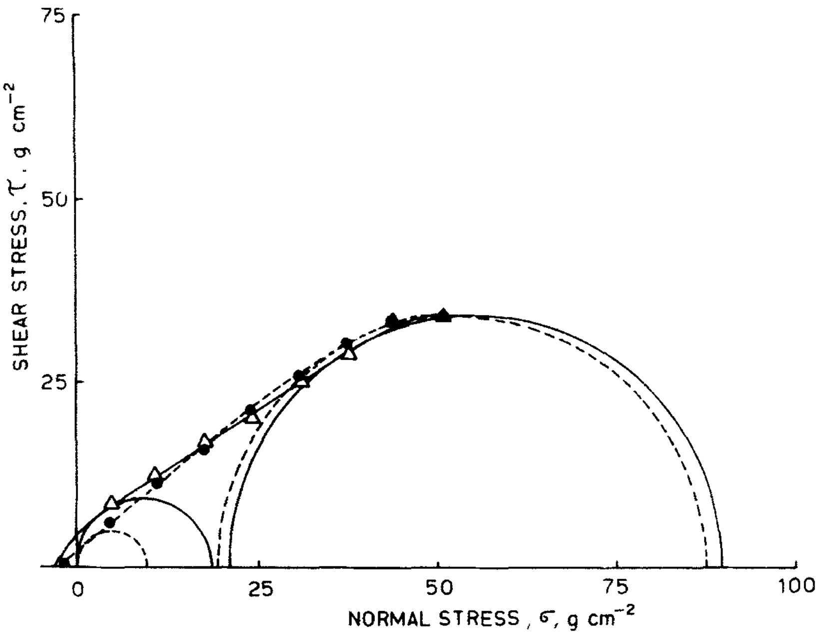

FIG. 4: Yield Loci of StarX, △, and Celutab, O, at a Normal Consolidating Stress of 50 gcm^{-2}.

Celutab has a $^{1}/FF$ value of 0.10 suggesting it is more free-flowing than StarX, with a $^{1}/FF$ value of 0.21. This can be verified in practice by measurement of flow rates through orifices or hoppers.

There is a strong relationship between cohesiveness, C, and the reciprocal of flow factor. Using linear regression a correlation coefficient of 0.97 was obtained. This is better than might be expected because there are errors involved in extrapolating the yield locus back to zero normal stress to determine C. However, there is little if any correlation between tensile strength, T, and C, or FF. As stated previously, some authors (11, 12) have suggested that $^C/T$ is a constant for a number of powders, often approximately equal to 2. For the twelve materials studied here, $^C/T$ was found to vary between 0.19 and 3.16 (Table 2). If the value of $^C/T$ is below 1, then it is difficult to argue that the measured value of tensile strength is a true end-point of the yield locus. This is what Hiestand and Peot (14) observed with aspirin and sitosterols. One possible explanation for an apparently high value of T for a coarse material is that under the effects of consolidation, some size reduction occurs. This possibility was tested for Celutab and the coarse crystalline lactose by sieve analysis before and after consolidation to the bulk density shown in Table 2. The results as presented in Fig. 5 show that very little size reduction actually occurred. The probable explanation is that, although a similar bulk density can be achieved in both the shear and tensile testers, the method of consolidation used in the two systems may not produce the same strength in the material (20).

The bulk density reported in Table 2 is that achieved in the shear cell, using a normal consolidating stress of 50 gcm^{-2}. The tensile

Table 2 presents tensile and shear data for the twelve materials studied

TABLE 2

Tensile and Shear Data for Twelve Diluents

Diluent	Bulk Density gml^{-1}	T gcm^{-2}	C gcm^{-2}	C/T	δ^{o}	FF	1/FF
Avicel PH101	0.41	5.58	7.03	1.26	46.7	3.8	0.26
Cornflour	0.57	3.38	3.19	0.94	38.1	6.7	0.15
StarX 1500	0.72	2.40	5.17	2.40	38.0	4.8	0.21
Mobile Starch	0.69	4.30	6.39	1.49	43.1	3.4	0.29
Lactose(spray dried)	0.91	1.01	3.19	3.16	43.1	6.8	0.15
Lactose(coarse crystalline)	0.88	2.93	1.34	0.46	38.8	18.5	0.05
Lactose(fine crystalline)	0.87	6.60	1.28	0.19	35.4	20.1	0.05
Dipac	0.82	1.22	2.04	1.67	42.7	12.2	0.08
Amerfond	0.83	2.11	4.79	2.27	48.7	4.5	0.22
Sugartab	0.80	2.25	4.15	1.84	52.4	4.4	0.23
Celutab	0.74	1.81	2.04	1.13	39.4	9.7	0.10
Encompress	1.01	1.37	1.60	1.17	35.5	14.8	0.07

strength values are interpolated from Figs. 2 and 3 at the corresponding porosity. The values for the flow factor (FF) are generally as would be expected from particle size information. The finer materials, Avicel and the three starches, exhibit much lower

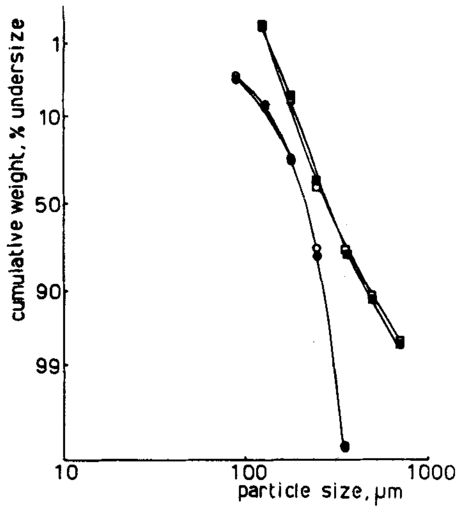

FIG. 5: Log-probability Plots for Coarse Crystalline Lactose (Circles) and Celutab (Squares) before Consolidation (Open Points) and After Consolidation (Shaded Points) in the Tensile Tester

values than the moderately coarse diluents. Cornflour appears more free flowing than the other two starches, as it has a higher geometric mean particle size and a narrower size distribution. Based on the values of the flow factors, the crystalline lactose powders and Encompress appear to be the most free-flowing. The very coarse material, Sugartab, exhibits a very low FF value.

The magnitude of the effective angle of friction δ, is a measure of the difficulty in maintaining steady state flow (19). It is interesting to note that the starch diluents exhibit quite low δ values. This suggests that although large orifices are necessary to initiate flow (based on low FF and high C values) once flow is initiated it will be quite uniform.

Ideally, the shear properties of pharmaceutical powders should be measured at the consolidating stress, corresponding to that in the hopper of a tablet machine or capsule filling machine. However, it is difficult to estimate such stresses and they would vary with the bulk density and frictional properties of the powder. It is much more convenient to subject each powder to the same normal consolidating stress to determine a flow factor. Some powders, however, have been shown to demonstrate "complex" behaviour in that parameters such as FF and δ are a function of the degree of consolidation (21, 22). This is in contrast to "simple" powders where these values are relatively independent of degree of consolidation. Therefore, it was considered important to establish whether the shear cell parameters of the powders in this study varied with normal consolidating stress.

Table 3 presents values of C, δ and FF for three representative powders, Avicel, StarX and Celutab, at a number of normal consolidating stresses.

Avicel appears to be a simple powder in that FF and δ remain relatively constant. StarX and Celutab do show some

TABLE 3

Variation of Shear Cell Parameters of Avicel, StarX and Celutab with Normal Consolidating Stress

Powder	Normal Consolidating Stress gcm^{-2}	C gcm^{-2}	δ °	FF
Avicel	24	8.47	53	3.3
	30	8.47	50	3.7
	37	10.69	51	3.7
	50	14.34	51	3.8
StarX	30	5.48	39	5.3
	37	11.47	42	3.3
	43	11.99	40	3.4
	50	10.56	48	4.8
Celutab	30	1.96	40	15.0
	37	2.35	36	12.8
	43	3.13	38	11.9
	50	4.17	39	9.7
	75	5.21	38	13.8

complex behaviour which has been observed previously with coarse granular materials (1, 23). Over the range of consolidating stress covered, however, the variation is not very great and the interpretation of the FF values would be the same at any stress. That is, Celutab would flow much more readily than the two finer materials.

A number of authors (3, 22) have fitted yield loci to the well-known Warren Spring equation.

$$\left(\frac{\Upsilon}{c}\right)^n = \frac{\sigma + T}{T} \qquad (1)$$

The parameter, n, is termed the shear index and its magnitude has been used as an index of flowability (3, 4). For very free-flowing materials, n approaches 1, whilst for cohesive materials, the value approaches 2. Yield loci for Avicel, StarX and Celutab at a normal consolidating stress of 50 gcm^{-2} were fitted to the logarithmic form of Eq. 1.

$$n \quad \log \frac{\Upsilon}{c} = \log \frac{\sigma + T}{T} \qquad (2)$$

by linear aggression. The values of the shear index obtained were Avicel 1.08, StarX 1.40 and Celutab 1.15, the correlation coefficients being 0.99 in each case. These values therefore, would suggest that Avicel is more free-flowing than the other two powders, in contrast to the order suggested by the flow factors. It is apparent from Eq. 1 that the value of n obtained would be highly dependent on the estimate of tensile strength, T, whereas estimation of FF does not necessitate knowledge of T. As discussed previously, if the modes of consolidation in the shear cell and tensile tester are different, then this could cause cause apparent errors in T and an anomalous answer for n.

It is planned to examine the usefulness of shear cell parameters, such as the flow factor, in predicting the flowability of a number of powders on tablet machinery.

REFERENCES

1. Jenike, A.W., Bulletin 108, Utah Engng. Expt. Station University of Utah, Utah. (1961).
2. Jenike, A.W., Bulletin 123, Utah Engng. Expt. Station, University of Utah, Utah. (1964).
3. Ashton, M.D., Cheng, D.C.H., Farley, R. and Valentin, F.H.H., Rheol., Acta., 4, 206. (1965).
4. Pilpel, N. Endeavour, XXVIII, No. 104, 73 (1969).
5. Pilpel, N. in "Advances in Pharmaceutical Sciences", 3, Bean, H.S., Beckett, A.H. and Carless, J.E. (Eds.) Academic Press, London and New York.
6. Pilpel, N. and Walton, C.A., J. Pharm. Pharmac., 26, Suppl., 1P (1974).
7. Kocova, S. and Pilpel, N., Powder Tech., 8, 33 (1973).
8. York, P., Powder Tech., 11, 197 (1975).
9. Stainforth, P.T. and Berry, R.E.R., Powder Tech., 8, 243 (1973).
10. Marshall, K. and Sixsmith, D., J. Pharm. Pharmac., 28, 770 (1976).

11. Farley, R. and Valentin, F.H.H., Powder Tech., 1, 344 (1968).

12. Cheng, D.C.-H., Farley, R. and Valentin, F.H.H., Paper to the Tripartite Meeting of Chemical Engineers, Montreal (1968).

13. Stainforth, P.T., Ashley, R C. and Morley, J.N.B., Powder Tech., 4, 250 (1970).

14. Hiestand, E.N. and Peot, C.B., J. Pharm. Sci., 63, 605 (1974).

15. Ashton, M.D., Farley, R. and Valentin, F.H.H., J. Sci. Inst., 41, 763 (1964).

16. Kocova, S. and Pilpel, N., Powder Tech., 7, 51 (1973).

17. Eaves, T. and Jones, T.M., J. Pharm. Sci., 61, 256 (1972).

18. Eaves, T. and Jones, T.M., J. Pharm. Sci., 61, 342, (1972).

19. Jenike, A. W., Elsey, P.J. and Wolley, R.H., Proceedings A.S.T.M., 60, 1168 (1960).

20. Jenike, A. W., Rheol, Acta., 4, 206 (1965).

21. Cheng, D.C.H. and Farley, R., Powder Tech., 2, 131 (1968).

22. Williams, J.C. and Birks, A.H., Powder Tech., 1, 199 (1967).

23. Jenike, A.W. and Johanson, J.R., Storage and Flow of Solids, Jenike and Johanson Inc., No. SFS-3 (1967).