Tensile fracture characteristics of double convex-faced cylindrical powder compacts

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Doubly-convex cylindrical tablets compacted uniaxially from different particle-size fractions of "Di-Pac-Sugar" powder, have been fractured under diametral loading conditions employing the standard diametral compression test. The ratio of cylinder length to diameter (*W/D*) ranged from 0.476–1.06; the ratio of cylinder diameter to radius of curvature of the tablet faces (*D/R*) was varied from 0.0–1.184. An equation based on geometrical volume equivalence conditions, of a doubly-convex cylinder to that of a plane-faced cylinder, relating the tensile strength of the "material" to the fracture load and dimensions of doubly-convex cylindrical specimens, has been developed. This equation is valid for any compacted cylindrical tablet. Using this equation, it was possible to assess the tensile strength of doubly-convex tablets, and a shape factor has been defined. Also, the predicted fracture loads obtained by this equation are found to compare well with those values determined by utilizing a "stress factor" based on the photoelastic stress analysis. For higher ratios of *W/D* and smaller diameter values, the variation in the results calculated is found to be less than 5%. Meanwhile, for higher values of *W/D*, the geometrical end effect is reduced and the tensile fracture stress tends to become similar to that of a plane-faced specimen.

1. Introduction

In the pharmaceutical industry, various methods and techniques are in use [1] for testing the mechanical properties and quality of the final compact, such as the tablet strength, hardness, toughness, etc. However, one of the most widely used techniques for testing the tensile strength of pharmaceutical tablets is the "diametral compression test". This test was originally devised by the Brazilian engineers Carneiro and Barcellos [2] for testing the strength of concrete. It became known as the "Brazilian disc test" and is commonly used in the determination of the tensile fracture stress of brittle materials. Basically, this test involves subjecting a simple plane-faced cylindrical disc specimen to two diametrically opposed loads, uniformly distributed along generators of the disc.

An analytical solution for the elastic stresses developed in the plane-faced disc specimen by this form of loading is available [3,4]. The maximum tensile stress, σ_{tf}, which acts across the loaded diameter is uniform and proportional to the applied load is given by

$$\sigma_{tf} = 2P/(\pi D T) \quad (1)$$

where P is the applied load, D the diameter and T the thickness of the compact respectively, as shown in Fig. 1.

In the last few decades, extensive data have been obtained by various investigators performing diametral compression tests on flat cylindrical tablets, employing either conventional or actual industrial testing machines [5–7]. However, many important aspects which relate to the final compact mechanical properties and which, to date, have not been systematically investigated, are the basic fundamentals of the mechanics involved in the porous powder compacts of different geometries. Also, the influence of rates and forms of loading on the mechanical properties as well as the failure modes and characteristics of the tablet formed finally. As a result of the works published by Amidon *et al.* [8], and later by Es-Saheb *et al.* [9] on pharmaceutical powder compaction, and the more recent static and dynamic investigations on the pharmaceutical compacts by Es-Saheb [10, 11]; it has become increasingly evident that the whole "stress–strain history" of compaction (i.e. the loading–unloading cycle), and the compact material and geometry, as well as the rate and form of load application during the compaction process and testing of the final compact, have very significant effects on the final static and dynamic strength, failure modes and characteristics of compacts.

In the diametral compression test, a knowledge of the resulting stress distribution is essential if the fracture load is to be quantitatively interpreted in terms of the material fracture stress. Generally, very little is known of the stresses developed in both plane and non-plane-faced cylindrical bodies under diametral

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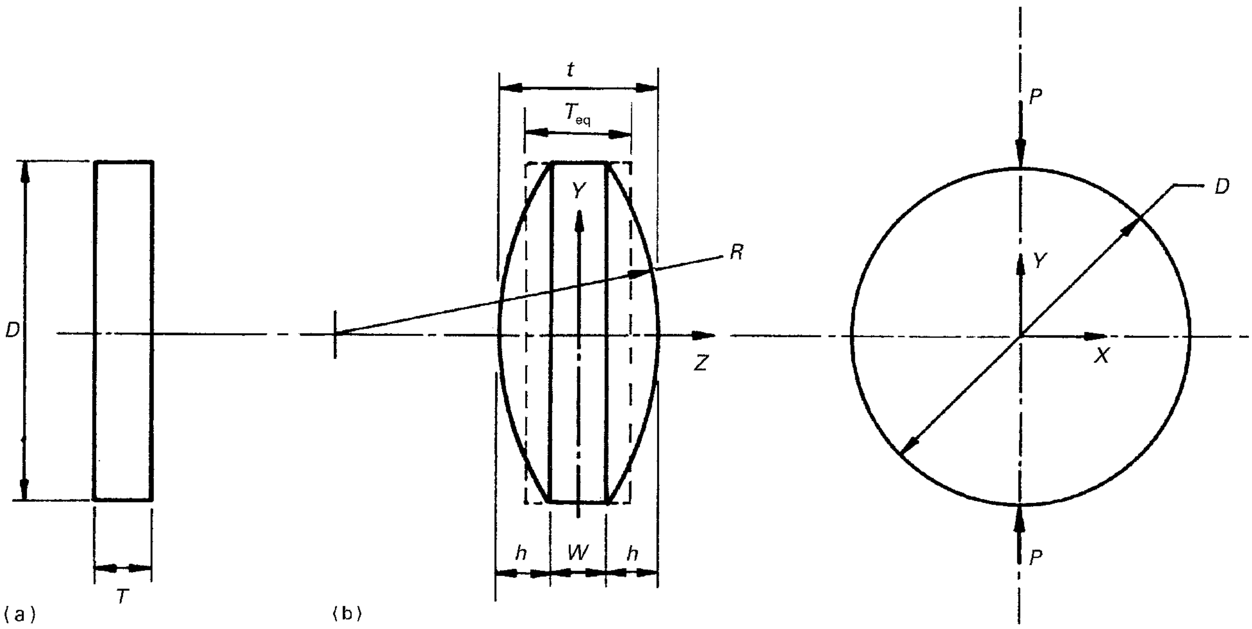

Figure 1 Schematic drawing showing the coordinates axes and symbols of (a) a plane-faced cylindrical specimen, and (b) a doubly-convex cylindrical specimen.

compression. Amongst the few reported attempts on plane-faced pharmaceutical tablets is the investigation of the stress in grooved tablets under diametral compression, described by Newton *et al.* [12].

The interpretation of fracture-load data from non-plane faced cylindrical tablets, (see Fig. 1), for which the stress distribution is unknown, is extremely difficult. However, Pitt *et al.* [13], in order to assess the strength characteristics of doubly-convex-faced cylindrical discs, regarded the basic problem as one of stress analysis, i.e. what are the stresses induced in the specimen under the action of diametral loading conditions? To address such a complicated problem, they first turned to the photoelastic stress analysis method. They managed to give complete assessment of the stress distribution in a range of convex-faced cylindrical discs subjected to diametral compression. Also, they succeeded in determining, what they called a "stress factor", I_{max}, for a certain range of doubly-convex faced cylindrical discs. The geometrical ranges they investigated were $0.06 \leqslant W/D \leqslant 0.3$ and $0.0 \leqslant D/R \leqslant 1.43$, see Fig. 1. Then, these authors showed that the maximum tensile stress, σ_t, in a convex-faced disc subjected to diametral compression could be calculated from the expression

$$\sigma_t = (2P/\pi DW)I_{max} \tag{2}$$

where P is the load, I_{max} is a stress factor, D the diameter of the disc and W is the cylindrical portion length. In a recent work by Pitt *et al.* [14] on double-convex cylindrical gypsum discs (covering the same geometrical ranges used in the previous work [13]), Equation 2 was used for the determination of the material tensile strength from the fracture load values. Also, the authors developed an empirical equation relating the material tensile strength, σ_f, to the fracture load, P_c, and dimensions of a doubly-convex disc, given as

$$\sigma_f = (10P_c)/(\pi D^2 F) \tag{3}$$

where D is the specimen diameter, and F is a factor dependent upon the cylinder length ratio and face-curvature ratio of the specimen and can be determined directly as the ratio of fracture loads [14] or from the expression

$$F = 2.84t/D - 0.126t/W + 3.15W/D + 0.01 \tag{4}$$

It is important to state here that Equations 1–4 are limited in their application and only valid under certain conditions. They are all based on the assumptions of linear elastic homogeneous and isotropic material conditions, which do not exist in all powdered compacts, particularly pharmaceutical tablets. Meanwhile, Equations 3 and 4 are both applicable to discs which fail in tension when loaded diametrically and only valid for the materials and geometrical ranges covered in the investigation, particularly the factor F. These issues will be scrutinized and discussed in more detail in the following sections.

The main objective of the work described in this paper was to determine the effects of variations in face curvature, R, and thickness, W, as well as powder particle sizes (180, 212, 250, 355, and 710 μm) on the stresses developed in actual convex-faced cylindrical tablets made of sugar (i.e. Di-Pac-Sugar) powder subjected to two-diametrically opposed forces uniformly distributed along a "generator" of the cylindrical portion of the tablet. An analysis of the fracture-load data, is also described. The investigation covers a wide range of geometries, that produced a successful resilient tablet (i.e. the ranges covered are $0.47 \leqslant W/D \leqslant 1.06$ and $0.0 \leqslant D/R \leqslant 1.184$). A general assessment of the failure characteristics, including a discussion relating the material tensile strength to the fracture load and the dimensions of a doubly-convex cylindrical tablet as well as the particle size, is presented.

2. Experimental procedure
2.1. Test materials
The tabletting characteristics of the chosen test material "sugar" are well known. It is specially formulated for direct compaction without granulation, and hence referred to in the text by the trade name "Di-Pac-Sugar". It is one of the most commonly used excipients for tabletting in the pharmaceutical industry. In this work, the Di-Pac-Sugar was in a form of powder (co-crystallization of 97% sucrose and 3% dextrins, manufactured by Amstar Corporation, New York), with a bulk density of 1580 $\mathrm{kg\,m^{-3}}$ (using an air-comparison pycnometer, Beckman model 930). The particle-size analysis and distribution of this powder employing the sieve analysis test are presented in Table I.

2.2. Specimen details and preparation
In general, for a given diameter, D, the doubly-convex-faced cylinders (see Fig. 1), can be characterized in terms of two independent variables; the cylinder length, W and the face radius curvature, R. These variables, however, can be expressed in dimensionless forms as D/R and W/D. The plane-faced disc, the sphere and the infinitely long cylinder present some special cases in which $D/R = 0, D/R = 2$ and $W/D = 0$, and $W/D \to \infty$, respectively. The analytical solution of such cases for the linear elastic isotropic homogeneous conditions are available in the literature [3, 4, 15]. The overall thickness of the specimen, t, is readily obtained from the expression, $t = W + 2h$ (see Fig. 1). This can be rewritten as $t = W + 2R - (4R^2 - D^2)^{1/2}$ or in a normalized form with respect to the diameter, as

$$t/D = W/D + 2R/D - [4(R/D)^2 - 1]^{1/2} \quad (5)$$

In preparing the samples, the main consideration was to generate wide ranges of these variables (i.e. W/D and D/R), including the ratios encountered in the pharmaceutical industries. Consequently, to investigate the effects of the various geometries, it is ideal to prepare two sets of specimens; one with constant W/D and varying D/R and the other with constant D/R and varying W/D. Unfortunately, because our tablets were prepared from Di-Pac-Sugar powder by the uniaxial compaction technique, the ranges of geometrical dimensions planned could not be totally achieved. The main reasons which hinder these objectives are: (i) the well-known friction effects engendered during the compaction process which resulted in non-uniform stress distribution and, consequently, non-homogeneous final compacts; and (ii) the powder material "capping pressure" constraints, widely known in the tabletting industry [16–19]. However, to overcome some of these constraints, and to achieve reasonable working ranges of the variables W/D and D/R, five powder size fractions of Di-Pac-Sugar and six standard circular sets of punches and dies of various geometries, were used to produce the required tablet samples.

The sets of punches and dies used are made of high-grade steel by Manesty Company Ltd, UK. Three sets consist of flat-faced punches of 9.5, 7.93 and 6.35 mm diameter, while the other three sets are of similar diameters but with punches of concave faces. These punches were used to produce the required specimens of both geometries: doubly-convex and plane-faced cylindrical tablets of the various dimensional ratios. The dimensions of the punches (i.e. both concave and flat) used together with the successful limited compact ranges and compression ratios of both sample geometries (i.e. doubly-convex and plane-faced tablets) are displayed in Tables II and III for concave and flat sets, respectively.

TABLE I Particle-size distribution of Di-Pac-Sugar powder

	Aperture (μm)									
	1000	710	355	250	212	180	125	90	63	< 63
Wt%	0.0	10.4	35.2	15.3	12.4	17.0	4.1	2.2	1.6	1.8

TABLE II The dimensions of the concave punches, and the double-convex cylindrical tablet geometries and compression ranges investigated

Punch set	Punch dimensions (mm)			Tablet geometries and compression ratios			
	D	R	h	W (mm)	t (mm)	t/W	W/D
1	9.50	11.78	1	6.55	8.55	1.31	0.689
				7.00	9.00	1.29	0.737
				7.50	9.50	1.27	0.789
				9.50	11.50	1.21	1.000
2	7.93	6.70	1.3	3.74	6.34	1.70	0.472
				5.00	7.60	1.52	0.631
				7.48	10.08	1.35	0.943
3	6.35	8.70	0.6	5.15	6.35	1.23	0.811
				5.78	6.98	1.21	0.910
				6.42	7.62	1.19	1.011
				6.73	7.93	1.18	1.060

TABLE III The dimensions and compression ratios of the flat punches and tablets investigated

Punch set	Punch diameter D(mm)	Flat tablet thickness T(mm)	compression ratio T/D
1	9.5	4.75	0.500
		7.00	0.737
		8.55	0.900
		9.50	1.000
2	7.93	4.75	0.598
		5.83	0.735
		6.34	0.799
3	6.35	4.75	0.748
		5.20	0.819
		6.35	1.000
		6.98	1.099

Using the six sets of punches and dies, a total of more than 660 samples from all particle-size fractions of Di-Pac-Sugar (180, 212, 250, 355 and 710 μm), were prepared on the Instron machine (Model 1197) at constant uniaxial compression speed of 5 mm min^{-1} for later testing. For each powder–punch combination conditions and each ratio case of W/D, T/D and D/R, six tablets were made to ensure consistency of the results. However to clarify the nature of the variation of the fracture loads with the ratios of T/D and W/D, few extra ratios were added to the investigated ranges by preparing and testing two additional series of specimens employing, only, the larger punch sets (of 9.5 mm diameter). The extra ratios included were $T/D = 0.6$ and 0.8 and $W/D = 0.554, 0.563, 0.794$, and 0.821, respectively.

2.3. The fracture tests

All the formed plane-faced and doubly-convex cylindrical specimens were fractured between parallel hardened steel platens, under the action of diametral loading conditions, on the Instron machine at constant compression speed of 0.2 mm min^{-1}. The fracture load in each case is recorded. For each case of powder–punch combination and each compression ratio, a mean fracture load value of six determinations was calculated. However, in all cases, the variation observed was less than 6%. Then the average fracture loads calculated for all the tablets were plotted on common axis with the powder particle size for the various ratios of T/D and W/D and are displayed in Figs 2 and 3, respectively. Meanwhile, Fig. 4 shows typical variations of the mean fracture load for the plane-faced specimens with T/D ratios and doubly-convex specimens with W/D ratios for the different values of D/R and a particle size of 355 μm.

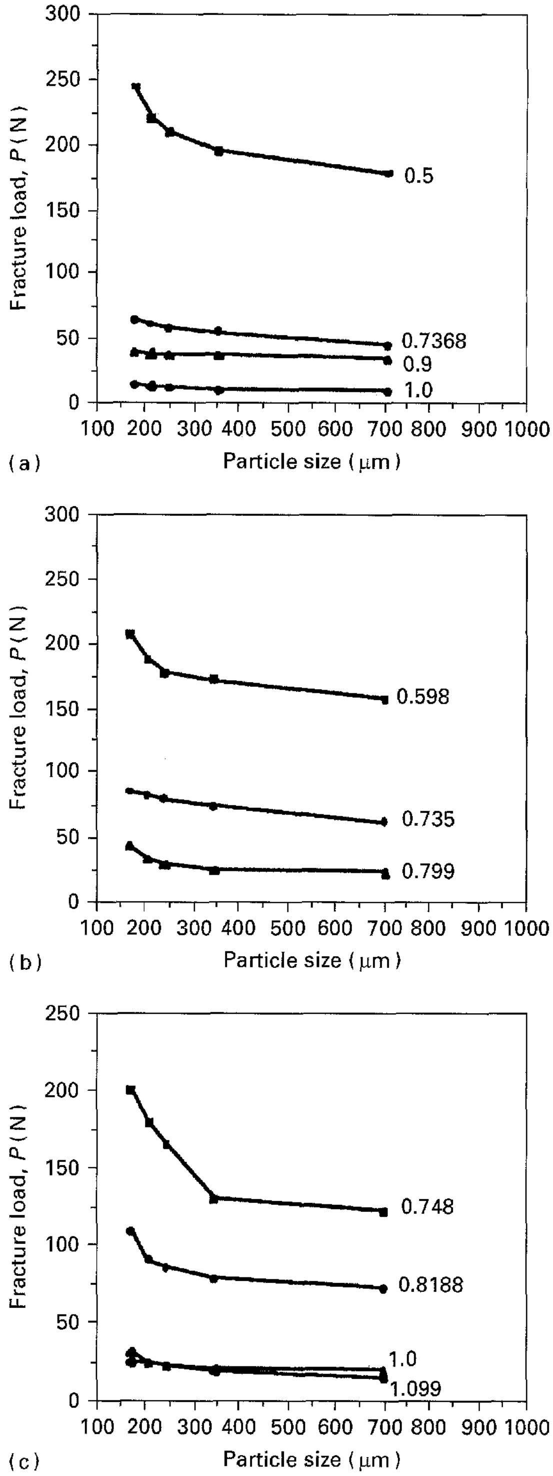

Figure 2 The mean fracture loads, P, obtained for the tablets made at the different ratios shown of T/D of the various particle sizes using the punch sets of (a) $D = 9.5$ mm, (b) $D = 7.93$ mm, and (c) $D = 6.35$ mm.

3. Results, analysis and data manipulation

3.1. The tensile strength of plane-faced cylindrical compacts

The mean tensile strengths of plane-faced cylinders (i.e. flat samples with $D/R = 0$) of differing cylinder lengths (T/D), were calculated from Equation 1 using the respective mean fracture load, P. These results are shown in Fig. 5 for all tested tablets. The results show that the determined tensile strengths vary, indicating that the plane stress assumption is not valid over this thickness range.

3.2. The tensile strength of doubly-convex cylindrical compacts

The doubly-convex cylindrical specimens consume relatively more complex geometries than those of

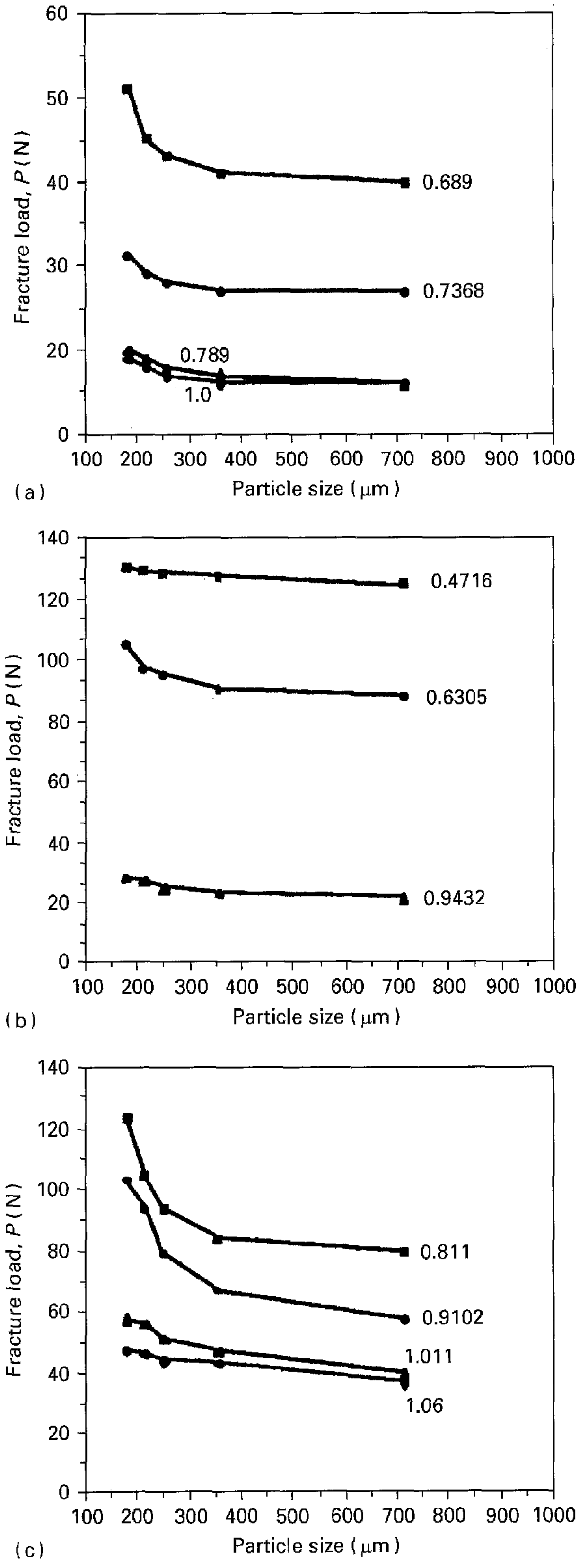

Figure 3 The mean fracture loads, P_c, obtained for the tablets made at the different ratios shown of W/D of the various particle sizes using the punch sets of (a) $D = 9.5$ mm, (b) $D = 7.93$ mm and (c) $D = 6.35$ mm.

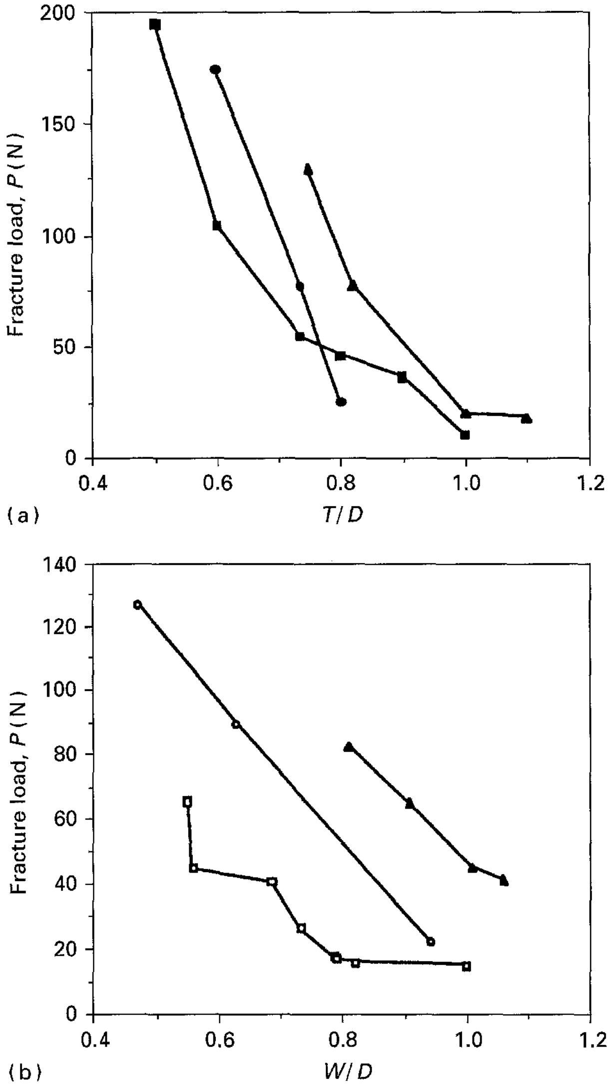

Figure 4 (a) Typical variation of the mean maximum fracture load for the plane-faced specimens, P, with T/D ratios for D values of (■), 9.5 mm, (●) 7.93 mm and (▲) 6.35 mm and particle size of 355 μm. (b) Typical variation of the mean maximum fracture load for double-convex specimens, P_c, with W/D ratios for D/R values of (□) 0.806, (○) 1.184 and (▲) 0.73. and particle size of 355 μm.

plane-faced samples. Consequently, and as reported earlier, it is problematic to calculate the tensile fracture stress from the fracture load data for such compacts (e.g. many pharmaceutical tablets), for which, in the first place, the stress distribution is unknown. Unfortunately, the adoption of Equation 2 to determine the fracture tensile strength of our powdered porous tablets, was found to be impossible. This is mainly due to (i) the fact that it was not possible to determine directly the values of the "stress factor", I_{max}, for our samples from the limited range recorded by Pitt *et al.* [13, 14], (ii) the non-linear nature of I_{max} which restricts extrapolation, and (iii) the obvious non-homogeneous porous nature of our samples compared with those elastic isotropic samples of photoelasticity and the relatively homogeneous moulded gypsum samples used in Pitt *et al.*'s investigations. Thus the photoelastic results are limited in correlating experimentally determined stresses with observed fracture loads in geometrically similar porous compact discs. However, the photoelastic data offered some scope for interpolation and extrapolation, but this scope is limited and it was not possible to apply it to our compacts.

At this stage, and in the wake of what is stated above; it was thought that the only main difference between the doubly-convex and plane-faced specimens was in geometry. This difference, however, was

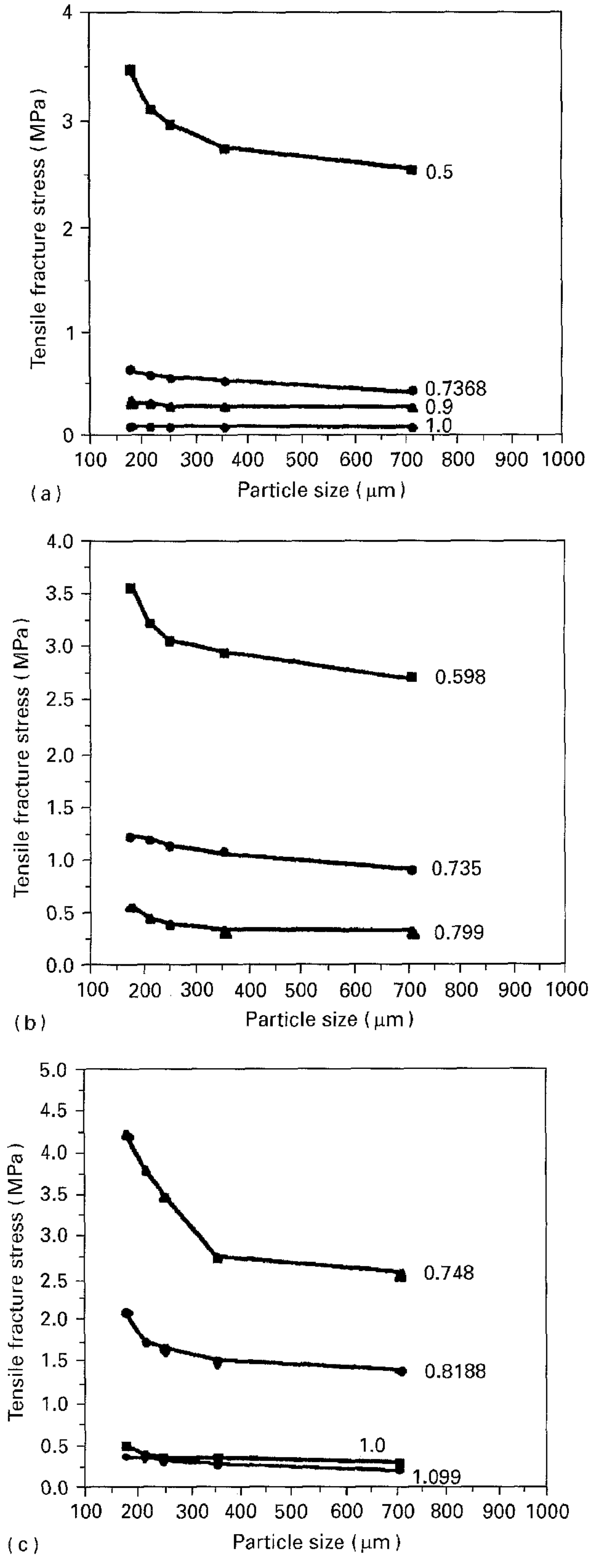

Figure 5 The variation of the maximum tensile fracture stress, σ_{tf}, for the plane-faced specimens of different T/D ratios, as shown, with the particle size using the punch sets with (a) $D = 9.5$ mm, (b) $D = 7.93$ mm and (c) $D = 6.35$ mm.

reflected in the different tensile strength expressions given by Equations 1 and 2. Thus, to avoid materials effects and most of the cumbersome and time-consuming procedures involved in the photoelastic stress analysis, it was suggested that the "volume equivalence" concept of the different sample geometries be utilized. Also, for convenience, in calculating the max-

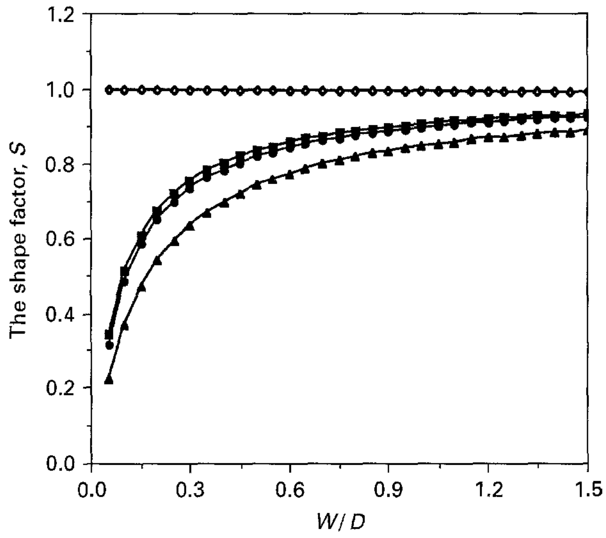

Figure 6 The variation of the shape factor, S, with W/D ratios for D/R values of (◇) 0, (■) 0.73, (●) 0.806, (▲) 1.184.

imum tensile fracture stress in our doubly-convex tablets, it was thought prudent to keep the formula form relatively similar to that of Equation 2, and given as

$$\sigma_{fc} = [(2P_c)/\pi D W]S \qquad (6)$$

where σ_{fc} is the maximum tensile fracture stress for the convex case, P_c the maximum compressive fracture load, D the compact diameter, W the length of the cylindrical portion and S the "shape factor". This factor, as reported above, is calculated on the basis of equating the doubly-convex cylindrical specimen volume, V_{convex}, to an equivalent plane-faced cylinder specimen volume, V_{eq}, see Fig. 1, given as

$$2\{(\pi/3)h^2(3R - h)\} + (\pi/4)D^2 W = (\pi/4)D^2 T_{eq} \qquad (7)$$

which can be reduced to give $T_{eq} = (8h^2/D^2)(R - h/3) + W$, or can be rewritten as

$$T_{eq} = WK \qquad (8)$$

where K is a geometry equivalence factor given as

$$K = (8h^2/WD^2)(R - h/3) + 1 \qquad (9)$$

the reciprocal of which is the shape factor, S, i.e.

$$S = 1/K \qquad (10)$$

The factor S as function of W/D for the different values of D/R used in this investigation is displayed in Fig. 6. It is obvious that for a plane-faced case (i.e. flat cylindrical specimens, where $h = 0.0$ and $R \to \infty$) both of the factors K and S are equal to 1. Therefore, $T = T_{eq} = W$, consequently Equations 1 and 6 become identical.

The mean tensile strength, σ_{fc}, of doubly-convex cylinders (i.e. double-convex specimens of different D/R ratios), of differing cylindrical lengths (i.e. different W/D ratios), are calculated from Equation 6 using the respective mean maximum fracture load, P_c, and the corresponding values of the specimens thickness, W, and the shape factor, S, respectively. These results are shown in Fig. 7 for all double-convex specimens. Again, the results show that the determined tensile strengths are varying in a relatively non-linear manner.

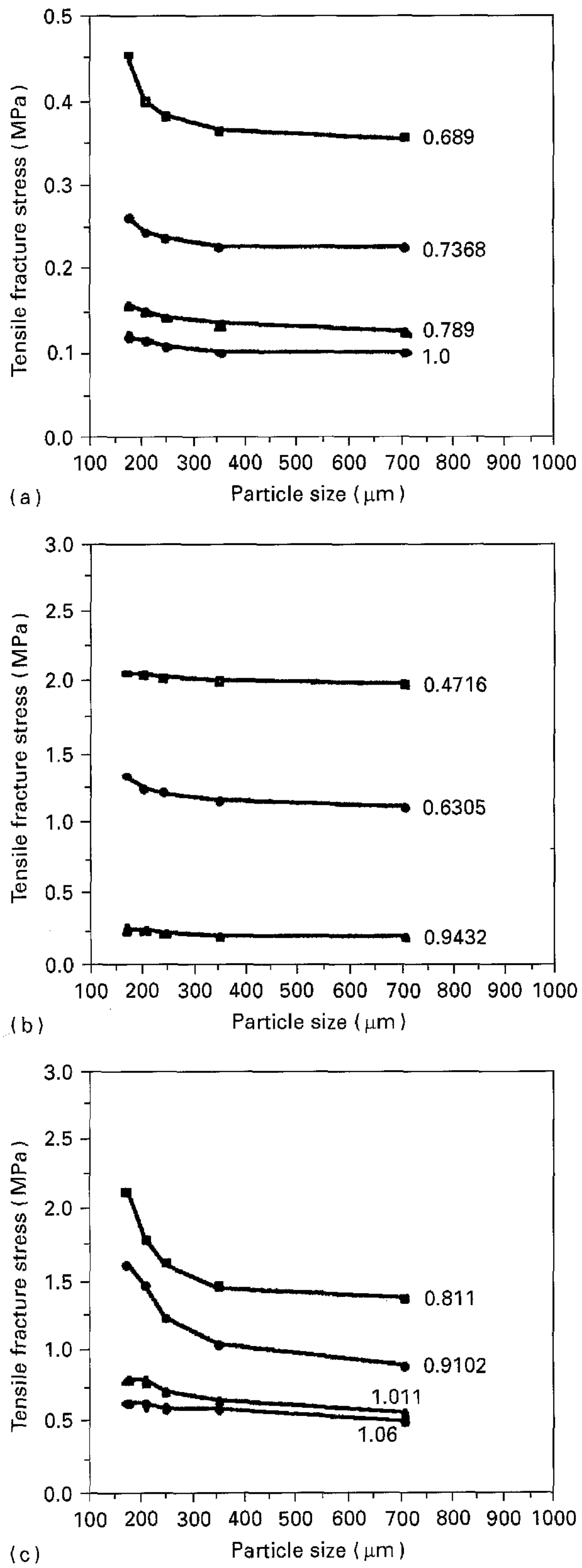

Figure 7 The variation of the maximum tensile fracture stress, σ_{tc}, for the doubly-convex specimens of different W/D ratios as shown, with the particle size using the punch sets with (a) $D = 9.5$ mm, (b) $D = 7.93$ mm and (c) $D = 6.35$ mm.

This emphasizes the non-homogeneous nature of these compacts and, consequently, the non-uniform stress distribution across the samples.

3.3. The fracture-load prediction of doubly-convex cylindrical specimens

In order to determine quantitatively the fracture load of doubly-convex cylindrical discs, it is necessary to determine the tensile strength of the material used. Unfortunately, by examining Figs 5 and 7, the results show that the determined tensile strengths, for all tablets, vary non-linearly. This indicates that there is no such value as true tensile strength of these porous compacts.

In spite of this, because Equation 6 is based only on geometrical considerations, it is thought that a correlation exists between this equation and Equation 1. Consequently, by scrutinizing the experimental data obtained and conducting preliminary calculations, some exact doubly-convex specimen cases and their corresponding equivalent flat specimens were identified. Therefore, for the equivalent flat specimen, the tensile strength (i.e. $\sigma_{tf} = \sigma_{tf.eq}$) is calculated employing Equation 1. But, as stated earlier, for flat tablet cases (i.e. where $S = 1$, thus $T = T_{eq} = W$), Equations 1 and 6 become identical, and consequently $\sigma_{tc} = \sigma_{tf} = \sigma_{tf.eq}$. Thus assuming this equivalent tensile strength to present the tensile strength of the compact material (for the specific conditions of W/D and equivalent T/D), and using the corresponding values of D, W and S of the equivalent doubly-convex tablet, the predicted fracture load, $P_{c.pre}$, could be calculated from Equation 6, which becomes

$$P_{c.pre} = (\sigma_{tf.eq}\pi D W)/(2S) \qquad (11)$$

The measured fracture load, P_c, and the predicted values, $P_{c.pre}$, of a few typical doubly-convex specimens together with their managed corresponding equivalent flat samples (i.e. P) and their tensile stress values (i.e. $\sigma_{tf.eq}$) for the various particle size fractions are shown in Table IV.

4. Discussion

4.1. The prepared sample limitations imposed by friction

In our case, a single-ended pressing technique was employed in preparing the specimens. It is well known that during the compaction process a non-uniform stress distribution results due to the effect of friction as well as the non-homogeneous nature of the powders [16–19]. This, eventually, is accompanied by an uneven density distribution throughout the compact, where the density tends to decrease with increasing distance from the pressing punch face. This is manifested in all of our samples by the considerable variation in the values of fracture loads and stresses, as displayed in Figs 2–5 and 7.

Thus to produce sound compacts it is necessary to optimize the frictional effects throughout the process [20]. This includes the reduction of the friction between the punches and powder, die walls and powder, and the inter-particle friction. Also, the powder column height is of a great importance in order to obtain a resilient defect-free final compact in addition to the selection of a relatively optimal compression force form and speed. The punch geometry also alters the friction conditions during the compaction process and thus has a considerable effect on the mechanical properties of the final compact.

TABLE IV The measured and predicted fracture loads, P_c, and $P_{c.pre}$, of typical doubly-convex specimens

Particle size (μm)	Flat samples		Double-convex samples		Variation (%)
	Fracture load, P (N)	Fracture stress[a], $\sigma_{tf,\ eq}$, (MPa)	Fracture load		
			Measured, P_c (N)	Predicted[b], $P_{c.pre}$ (N)	
$D/R = 0.73$ ($D = 6.35$ mm, $S = 1.1179$, $T/D = 0.8188$ and $W/D = 0.811$)					
710	73	1.407	79	80.80	2.3
355	78	1.504	83	86.37	4.1
250	85	1.639	93	94.12	1.2
212	90	1.735	103	99.63	3.3
180	108	2.082	122	119.56	2.0
$D/R = 0.806$ ($D = 9.5$ mm, $S = 1.155$, $T/D = 0.900$ and $W/D = 0.689$)					
710	35	0.274	40	34.91	12.7
355	37	0.290	41	32.72	20.2
250	37	0.290	43	32.72	23.9
212	38	0.298	45	33.62	25.3
180	40	0.314	51	35.42	30.5
$D/R = 1.184$ ($D = 7.93$ mm, $S = 1.269$, $T/D = 0.735$ and $W/D = 0.6305$)					
710	65	0.895	88	70.74	19.6
355	77	1.060	90	83.78	6.9
250	82	1.129	95	89.23	6.1
212	86	1.184	97	93.58	3.5
180	88	1.212	105	95.79	8.8

[a] Calculated using Equation 1.
[b] Calculated using Equation 11.

It should be remembered that testing is only carried out over limited ranges (i.e. limited compression ranges of T/D and W/D), where there is a fairly good chance of forming sound tablets; this is indicated clearly in Tables II and III. Therefore, a large number of preliminary tests were conducted to determine the ranges which would produce a "perfect" coherent compact for a specific powder, punch and die combination. At large ratios of T/D and W/D the produced tablets are found to be weak and tended to "crample" (i.e. loose compacts). Meanwhile, at smaller ratios, high compression pressures were attained and very hard compacts were obtained with a tendency to laminate and show end "capping" during compaction and on ejection from the die. This high pressure is known in the pharmaceutical tabletting industry as the "capping pressure". It varys with the different compaction conditions and from one powder to another. However, inspite of these difficulties, reasonable working ranges of height to diameter ratios are managed for the different conditions and powder, die and punch combinations. These ranges of both flat and double-convex Di-Pac-Sugar compacts are shown in Tables II and III, respectively. This explains some of the rooted difficulties in the compaction process which are faced in preparing the actual test tablets, and the failure in achieving the planned "impossible" ideal wide compression ratios of T/D and W/D.

4.2. Powder bonding and strength

The mechanisms governing powder bonding are well documented in the literature [1, 9, 17–19]. All, however, illustrate that increased compression pressure is required to form strong compacts, but this action exacerbates the problems associated with decompression, which can lead to lamination of the formed compact. Generally, the amount of bonding, (which is mainly due to the amount of plastic deformation involved in the process), and the tensile strength (fracture load) of the tablets is a function of the bonding created by the compaction pressure. As the axial pressure increases, so does amount of plastic deformation, and hence the bonding. This could be seen from Figs 2–5 and 7 where the tensile strength is seen to increase as the height to diameter ratio (i.e. T/D and W/D) is decreased for both cases of flat and double-convex tablets, respectively.

Also it is noticed that the maximum fracture loads, see Figs 2–4, and stresses, see Figs 5 and 7, of the compacts increase as the powder particle sizes are decreased in a non-linear manner for the different ratios of T/D and W/D, respectively. This can be attributed to the fact that for smaller particle sizes, higher initial backings (i.e. high tap densities) are achieved. Consequently, this tends to offer higher resistance to compaction and hence higher axial pressures are required. This, in turn, will result in higher densification and more plastic deformation and hence higher tensile strength.

Furthermore, from Figs 5 and 7, it is always observed that the tensile strength of the doubly-convex compacts is greater than that of the equivalent flat ones. This indicates that more plastic deformation is involved during the compaction of the double convex tablets. Also, the dominance of the various mechanisms of deformation and bonding during compaction and the mechanical properties of the material are important in determining the final compact tensile strength as well as the shape and mode of failure involved in the diametral compression test [8, 21].

4.3. The tensile strength of porous cylindrical tablets

By examining the values of the fracture tensile strength, σ_{tf}, of the flat compacts calculated using Equation 1 and displayed in Fig. 5, it is obvious that these values change considerably for the different ratios of T/D and various powder particle-size fractions. This is also clearly manifested in the non-linear pattern shown in Fig. 4a. This reflects the non-homogeneously changing nature of these porous compacts, which suggests that the tested compacts can be regarded from the mechanical point of view as if they are made of "different materials". Consequently, this indicates that, there is no such value as true tensile strength, but the values obtained for a particular set of experimental conditions are true for those conditions.

As far as the "Brazilian disc test" is concerned, the non-homogeneous porous nature of the tablets resulted in the non-linear stress distribution patterns, particularly at the advanced stages of the test. At these stages, the contact areas at the loading regions tend to increase and change the loading conditions, and hence the stress nature and distribution patterns (i.e. some non-linear compressive stresses are produced) [4]. It is important that the specimen fractures only in tension and not as a result of the compressive or shear stresses.

It is also important to mention that the mechanical properties of the specimen and load platens determine the stress distribution within the specimen. In our samples it was observed that, at higher ratios of T/D and W/D, the specimen is soft enough for there to be a spreading of the load at the contact points due to flattening of the specimen. This will prevent ideal line loading and reduce the shear and compressive stresses and allow failure to be initiated in tension. Meanwhile, at the lower ratios of T/D and W/D, the specimen tends to show a high modulus of elasticity and, consequently, ideal line loading may be approached causing failure to be initiated due to compression or shear stresses.

Furthermore, it was noticed in most tablets that, even if tensile failure is ensured, the brittle nature of these materials resulted in a variable value for the breaking load of nominally identical tablets. Although the variation observed in our investigation is relatively small (i.e. $\sim 6\%$), nevertheless, it agrees well with that given by Newton and Stanley [22]. Therefore, the assumption of a plane-stress condition, especially for these porous structures, is not valid. This would also, explain, some of the variations and difficiencies observed in the work of Pitt *et al.* [14], including their assumption of plane-stress conditions over the investigated geometrical ranges, and the use of a "fixed value" for the tensile strength of the material (i.e. the fracture stress of the flat sample with $T/D = 0.2$) to predict the fracture loads of the cast doubly-convex gypsum discs.

It is thus concluded that the use of one value for the fracture tensile stress to present the tensile strength of the "porous tablet" is somewhat erroneous. Thus, it is believed that the use of the different equivalent tensile strength (i.e. $\sigma_{tf.eq}$) values, corresponding to the specific doubly-convex condition, in predicting the tablet fracture load in this investigation is more accurate, see Table IV.

From Fig. 6 it is clear that as the ratio of W/D increases, the shape factor, S, tends to approach the unit value (i.e. $S = 1.0$), which corresponds to the plane-faced condition. This indicates that the end geometrical effects are diminishing as the W/D ratio increases for the different values of D/R, particularly for the smaller diameter, D, values. Thus the measured fracture load values for all the cases of W/D and T/D tends to give equal values at these high ratios, as shown in Fig. 4. This suggests that at higher ratios of W/D, (e.g. in our case $W/D > 1.5$), the geometrical end effects could be ignored and all specimens in this case could be treated as plane-faced. Consequently, in this range (i.e. $W/D > 1.5$), Equation 1 could be applied directly (with relatively small acceptable error), to calculate the tensile fracture stress in a double-convex specimen.

From Table IV, it is observed that as the tablet diameter, D, increases, the predicted fracture loads, $P_{c.pre}$, from the values of the tensile stress, $\sigma_{tf.eq}$, of the equivalent plane-faced specimen, are found to vary considerably from the measured values (e.g. at $D = 9.5$ mm the variation was 20%–30% compared to less than 5% at $D = 6.35$ mm). This large variation taking place at large diameter values, could be due to the higher "caps" volume contribution (i.e. the volume of the convex parts of the specimen) to the total volume of the tablet. This results in more complex geometries and hence sophisticated non-linear stress distributions. However, for small values of tablet diameter (e.g. $D = 6.35$ mm) the predicted fracture load variation is found to decrease (less than 5%). This suggests that the prediction of the fracture load using Equation 11 would be comparable with that of Pitt *et al.* [14], which was based on photoelastic stress analysis. For smaller tablet diameters, Equation 11 is found to give closer results and less scatter in the values calculated. Generally, Equations 6 and 11 are easy to apply and give comparatively accurate results. Also, being derived on geometrical bases, they tend to be less susceptible to materials properties and conditions.

From Figs 5 and 7, the fracture strengths σ_{tf} and σ_{tc} of all the tablets are found to be considerably lower than the average yield stress, σ_y, of the Di-Pac-Sugar powder itself as derived from the Heckel equation employed in previous investigations by Es-Saheb [11] and Es-Saheb *et al.* [9]. Also, the actual tensile strength of the Di-Pac-Sugar compacts, reported recently by Es-Saheb [21] in an investigation utilizing a dynamic testing approach, was found to be higher than those observed in this study. This loss of strength would arise generally from a combination of the following factors: (i) reduced quantity of powder, due directly to the presence of porosity (this is the major effect); (ii) the ratio of the cross-sectional area of the welds between particles to overall cross-sectional area is less than the ratio of the volume of "material" to the overall volume; and (iii) the notch effect of the porosity produces local high stress concentrations at the welds between particles. These factors contribute towards a lower strength than would be expected from a strictly linear relationship between porosity and strength. These factors, however, are heavily affected by the speed of compaction and the manufacturing method [9, 11, 21].

Finally, the development of some means of reliably predicting material tensile fracture stress from the fracture loads of "porous disc tablets" over a wide range of face curvatures and cylinder lengths is required. However, as discussed above, this proved to be a challenging task. The community working in the mechanics field, in general, and in powder mechanics as well as porous structures, has long accepted the enormous difficulties in achieving a fundamental understanding of the actual mechanics involved in the compaction process and the fracture mechanics of the porous powder compacts (i.e. tablets). Particularly, in the absence of reliable constitutive relations in these fields which would assist in the understanding of these phenomena and revealing some of the mysteries of this science.

5. Conclusion

The success of the final compact requires specific powder–die combinations and friction conditions which depend mainly on the punch and die sizes and geometries as well as the powder particle size and shape conditions.

The fracture loads of a variety of doubly-convex-faced Di-Pac-Sugar tablets formed, have been determined employing the diametral compression test. The relationship between the fracture load and the particle size for all tablets, of the different values of T/D and W/D ratios, is found to be non-linear.

The equations used to calculate the fracture stresses are applicable to specimens which fail in tension when loaded diametrically. An equation based on geometrical volume equivalence of a doubly-convex tablet to that of a flat-faced cylindrical tablet is derived. This equation is valid for any compacted brittle materials. Employing this equation, it was possible to assess the tensile strength of doubly-convex tablets, and a shape factor has been defined. The prediction of fracture load from the tensile strength of porous compacts is not easy, where there is no such value as true tensile strength for a porous compact. However, the predicted fracture loads determined by this equation compare well with those based on photoelastic stress analysis by Pitt *et al.* For high ratios of W/D and lower values of diameter, D, the variation in the results calculated was found to be less than 5%. Also, for higher values of W/D the geometrical end effects decrease and the tensile fracture stress tends to become similar to that of plane-faced specimens. The tensile strength of the convex tablet is always found to be greater than that of the equivalent corresponding flat tablet. Finally, for a constant W/D the shape factor, S, tends to decrease with increasing face curvature.

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Received 18 February 1994
and accepted 17 March 1995