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The Material Tensile Strength of Convex-faced Aspirin Tablets

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Abstract—The material tensile strength of a range of convex-faced tablets, compacted under controlled conditions at pressures of 40 and 320 MPa from a size fraction of acetylsalicylic acid, has been assessed. The calculation of the tensile strength σ_t, from observed fracture loads obtained in diametral compression testing, is based on the equation derived by Pitt et al (1988), namely:

$$\sigma_t = \frac{10P}{\pi D^2}\left(2{\cdot}84\frac{t}{D} - 0{\cdot}126\frac{t}{W} + 3{\cdot}15\frac{W}{D} + 0{\cdot}01\right)^{-1}$$

where P is the fracture load, D is the tablet diameter, t is the overall tablet thickness and W is the central cylinder thickness. The strength of a tablet of a given shape compacted at 320 MPa was between two and four times greater than that of a similar tablet compacted at 40 MPa. For the thicker tablets ($W/D \geqslant 0{\cdot}2$) the material tensile strength was practically independent of shape. For the thinner tablets ($W/D = 0{\cdot}1$) the material tensile strength varied considerably with face-curvature, showing a maximum for each of the two compaction pressures at a D/R value of 0·67.

The literature contains a considerable amount of information on the compaction behaviour of numerous pharmaceutical powders. However, there are relatively few publications dealing with the use of punches with concave faces or bevelled edges—the most common forms of punches encountered in the pharmaceutical industry. Such shapes are used to improve surface finish and to avoid edge-chipping. The departure from the simple plane-faced cylindrical shape may present problems in the assessment of pressure/strength relationships and hence most fundamental compaction studies avoid the use of concave punches.

The surface hardness distribution of convex-faced tablets of aspirin has been investigated using a Brinell hardness tester (Aulton & Tebby 1975). The results showed that as the curvature of the punch increased there was a decrease in the mean Brinell hardness. A comparison of surface hardness distributions showed that, for "normal" (i.e. $D/R = 0{\cdot}63$: see later) concave punches, there was an almost uniform distribution of surface hardness across the tablet face, whereas tablets prepared with deeper concave punches had outer zones harder than the central region, and plane-faced tablets were harder in the centre than at the periphery. This indicates that there is probably a variation in force transmission over the punch face and a consequent variation in the density of the compacted material, the degree of variation depending upon the face curvature.

A similar phenomenon was observed when the pressure differences exerted at different positions within concave-faced punches during the compaction of chymotrypsin were measured (Filbry & Mielck 1985). The relative activity of the enzyme was quantitatively assessed and attributed to the effect of variations in compaction pressure. The results were interpreted in terms of a "curvature factor" obtained by dividing the radius of curvature of the punch face by the punch diameter. The range of pressure within the tablets varied as this curvature factor decreased from infinity (i.e. plane-faced tablets) to 0·63, and the value of the maximum pressure passed through a minimum at a curvature factor of unity.

Axial and radial powder movements during the tableting process were studied using various punch tip geometries by Sixsmith & McCluskey (1981). Different coloured powder layers, as used earlier by Train (1956), were used to assess the powder movement. Alteration of face-curvature did affect both radial and axial powder movement, the extent being dependent upon the relative curvature of the punch tip. The addition of lubricant decreased the relative axial movement but had little effect on radial movement. No quantitative assessment of density or powder movement was reported.

Whatever variations occur in the structure of the tablet, changes in face curvature, thickness and diameter, will influence the load at which the tablet fractures when subjected to diametral compression. It has been established by Newton et al (1972) that under this form of loading convex-faced tablets fracture in tension, i.e. break across the loaded diameter. Even so, without some means of calculating the material strength (i.e. the tensile fracture stress of the material which constitutues the tablet) from the fracture load, an assessment of which of the various tablet shapes has the greatest material strength cannot be made by fracture tests alone.

The material tensile strength, σ_t, of a plane-faced tablet, diameter D, thickness t, can be calculated from the fracture load P in diametral compression using the equation

$$\sigma_t = \frac{2P}{\pi D t} \quad (1)$$

This equation can be derived theoretically (Den Hartog 1952) and has been validated by extensive photoelastic work

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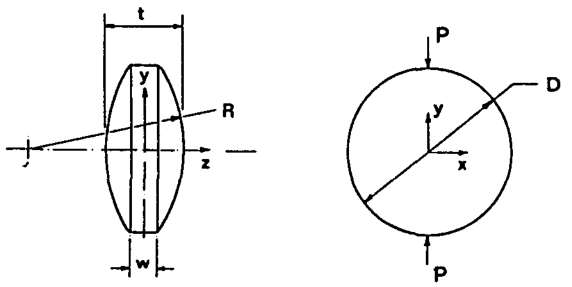

FIG. 1. Side and front elevations of convex-faced disc showing axes and symbols.

(Frocht 1948). A comparable theoretical solution for a tablet with symmetrical convex faces is not available, but Pitt et al (1988) have established an empirical solution based on fracture studies of a range of homogeneous brittle test specimens formed from gypsum. The resulting equation expresses the tensile strength of the material σ_t, in terms of the fracture load P, disc diameter D, overall thickness t, and central cylinder thickness W, as

$$\sigma_t=\frac{10P}{\pi D^2}\left(2{\cdot}84\frac{t}{D}-0{\cdot}126\frac{t}{W}+3{\cdot}15\frac{W}{D}+0{\cdot}01\right)^{-1} \qquad (2)$$

This equation was shown to be valid for convex-faced tablets with W/D in the range 0·1 to 0·3 and D/R ratios of 0 to 1·43, where R is the radius of curvature of the convex face (see Fig. 1).

In the work described in this paper, this equation is used in the determination of the effects of dimensions and shape on the material tensile strength of convex-shaped tablets compacted from aspirin crystals under carefully controlled conditions at two compaction pressure values. Implied assumptions in the application of equation (2) are that the different aspirin tablets are homogeneous on a scale which is small relative to the dimensions of the tablet (or more precisely sufficiently homogeneous to ensure that failure occurs at the point of maximum stress) and that the relevant failure criterion is the maximum principal stress criterion.

Materials and Methods

Several lots of crystals from a common batch of aspirin (acetylsalicylic acid) (Monsanto, Batch No. 4B611, Grade 7016) were sieved under identical conditions and the fraction 250–355 μm retained for use. These retained crystals were stored in amber glass jars with airtight seals at a temperature of approximately 20°C.

The punches used for compacting the sieved material were manufactured from KE960 steel, heat-treated to give a surface hardness within the range 570-620 VPN. All the punch tips were machined and polished to the same finish. The same die, internal diameter 12·50 mm, was used for all the work.

The pressure exerted upon the powder by a plane-faced punch is calculated by dividing the compaction force at the lower punch by the cross-sectional area of the punch. The pressure exerted by a concave-faced punch is not so readily characterized. If the powder behaved as a liquid then the punch would experience a uniform pressure acting normal to its surface. In practice this is not a realistic assumption; frictional forces are developed at the powder-die interface and the detailed force distribution over the punch face will not generally be known. Therefore, to facilitate comparison in these analyses between tablets of different face-curvature, the area used for the calculation of compaction pressure was that of the central cylindrical cross-section of the tablet. (In practice the difference between the area of the cylindrical cross-section and the area of the punch face is not great. The difference is 14% for the case of the greatest curvature.)

Tablets were compacted uniaxially on an Instron testing machine (Floor Model No. TT, Instron), modified to take a conventional "F" punch and die set. The lower compaction force was determined by means of a load cell (Instron GR) and recorded on a flat bed X-Y Recorder (Gould Model No. 6000). The load cell was electronically linked to the crosshead so that when a predetermined compaction force was attained on the lower punch, a relay was tripped to reverse the movement of the crosshead, thus withdrawing the punch from the die at the same speed as it was applied. In this way, the tablets were all compacted in a reproducible manner to the desired nominal pressure, with a coefficient of variation of pressure of approximately 0·5%. The two compaction pressure values used in this study were 40 and 320 MPa. The crosshead speed throughout was 5 mm min^{-1}.

Since the prime objective of the work described here was to assess the effects of changes in dimensions and shape upon the tensile strength of material compacted in the form of tablets, every care was taken to ensure that the dimensions of each set of tablets in a particular series covering a range of compaction pressures were kept constant throughout the range. The overall thickness of a tablet (t, see Fig. 1), will decrease for a given quantity of powder as the compaction pressure is increased. Hence, to keep the cylinder length, W (see Fig. 1), constant over the compaction pressure range, increasing quantities of powder had to be compacted to form the tablets. The exact quantities required were determined by trial and error. (As an example, for plane-faced tablets with W/D = 0·2, an extra 8% by weight of aspirin crystals was required over the pressure range 40 MPa to 320 MPa.)

Seven different face-curvatures (see Table 1) and three cylinder lengths (i.e. W/D = 0·1, 0·2 and 0·3) were examined at each of the two compaction pressures. The tablets were manufactured to within 5% of the required dimensions.

The tensile strength of tablets changes with time (Rees & Shotton 1970), particularly during the first hour after compression. This is attributed to stress relief of the crystals and interparticulate bonding. Kennerley (1980) had demon-

Table 1. Face curvature ratios.

Face curvature ratio D/R	Curvature description
0	Flat
0·25	Micro
0·50	Shallow
0·67	Normal
1·00	Unity
1·25	Deep
1·43	Coating

D = diameter of tablet (12·5 mm throughout)
R = radius of face curvature

strated that after 14 days stress relief is complete and tablet strength is independent of time. Hence, in this investigation, all the tablets were stored in sealed, airtight amber glass bottles for 14 days after compaction. The tablets were then fractured in diametral compression between hardened steel platens using a CT 40 tablet tester (Engineering Systems, Nottingham) at a platen movement rate of 1 mm min^{-1}. Ten samples of each tablet were tested. Results for the few tablets which did not fail diametrally in tension were excluded from subsequent calculations.

The mass and thickness of each tablet were recorded immediately before fracture. The dimensions and masses of some tablets were also noted 1 min after ejection from the die. It was concluded from these measurements that any changes in these properties during the 14-day storage period were less than ±0·001 g and ±0·01 mm, the respective sensitivity limits of the measuring devices.

Results and Discussion

Data are presented for tablets compacted at 40 and 320 MPa. A plot of mean fracture load versus face-curvature (Fig. 2) at both 40 and 320 MPa indicates clearly that, as either the cylinder length ratio (W/D) or face-curvature ratio (D/R) increases, then the fracture load increases. This is what would be anticipated as, in each case, the specimen is increasing in volume and mass. Coefficients of variation associated with the mean fracture load values were 10-15% or less except for some of the tablets compacted at 40 MPa (W/D=0·1, D/R=0 (20%); W/D=0·3, D/R=0 (23%); W/D=0·3, D/R=0·25 (18%)).

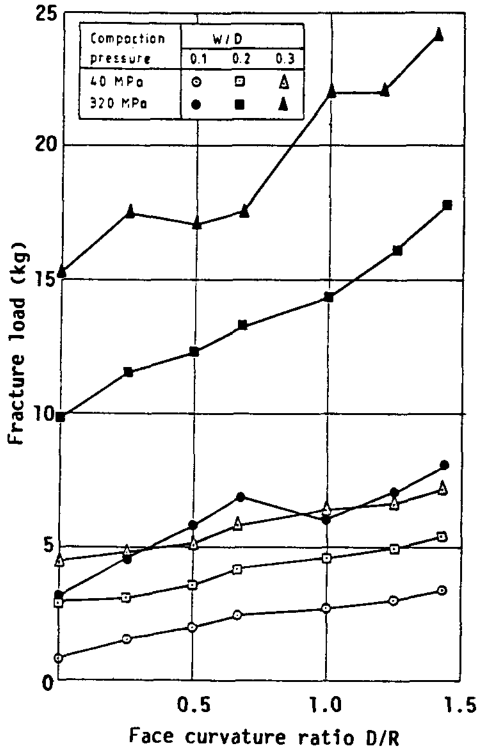

FIG. 2. Plot of mean fracture load versus face-curvature at both 40 and 320 MPa compaction pressure.

Table 2. Fracture load ratios after compaction at 320 and 40 MPa.

	W/D		
D/R	0·1	0·2	0·3
0	3·56	3·38	3·44
0·25	3·07	3·74	3·65
0·50	2·90	3·42	3·35
0·67	2·85	3·17	3·03
1·00	2·22	3·12	3·44
1·25	2·33	3·25	3·33
1·43	2·38	3·30	3·36

Table 3. Material tensile strength of aspirin tablets (MPa)

	Compaction pressure 40 MPa			Compaction pressure 320 MPa		
	W/D			W/D		
D/R	0·1	0·2	0·3	0·1	0·2	0·3
0	0·38	0·54	0·53	1·29	1·80	1·82
0·25	0·52	0·50	0·52	1·57	1·89	1·91
0·50	0·58	0·52	0·52	1·69	1·82	1·73
0·67	0·64	0·58	0·56	1·81	1·83	1·69
1·00	0·61	0·56	0·55	1·33	1·73	1·91
1·25	0·61	0·55	0·54	1·44	1·80	1·80
1·43	0·60	0·54	0·53	1·42	1·79	1·81

Fracture load ratios at the two compaction pressures for each shape tested are given in Table 2. It can be seen that the eight-fold increase in compaction pressure resulted in a considerable strength increase, varying from 2·22 times to 3·74 times. There was no clear systematic variation with increasing D/R values in the fracture load ratios for tablets with W/D$\geqslant$0·2, but for the W/D=0·1 tablets there was a well defined decreasing trend in the ratio from 3·56 to 2·38 as D/R increased from 0 to 1·43.

The W/D=0·1 tablets also stand out as distinctive when material tensile strengths are considered. Mean material tensile strength values (σ_t), derived from corresponding fracture loads using equation (2), are tabulated in Table 3. Recalling (Pitt et al 1988) that a tolerance band of the order of ± 10% is to be associated with equation (2), it can be seen that, for the two compaction pressures used, there are no significant material strength variations in tablets with W/D$\geqslant$0·2 as R/D increases. By contrast, however, tablets with W/D=0·1 show a clear maximum in the material tensile strength over this D/R range, and for both compaction pressures the maximum occurs with a curvature ratio of 0·67, the "normal" value.

These trends are clearly shown in Figs 3 and 4, which are plots of material tensile strength values normalized with respect to the value for the corresponding plane-faced (D/R=0) tablet, versus face-curvature ratio. For the 40 MPa compaction pressure (Fig. 3), the maximum material tensile strength occurring at the normal curvature value, with W/D=0·1, is 68% greater than that of the plane-faced tablet and it is noteworthy that this stronger condition persists, to a degree, for higher face curvatures at this cylinder thickness ratio. For the 320 MPa compaction pressure the maximum material tensile strength in the W/D=0·1 series is 43% greater than the plane-faced tablet value and this increase is lost almost entirely in the higher face-curvature tablets.

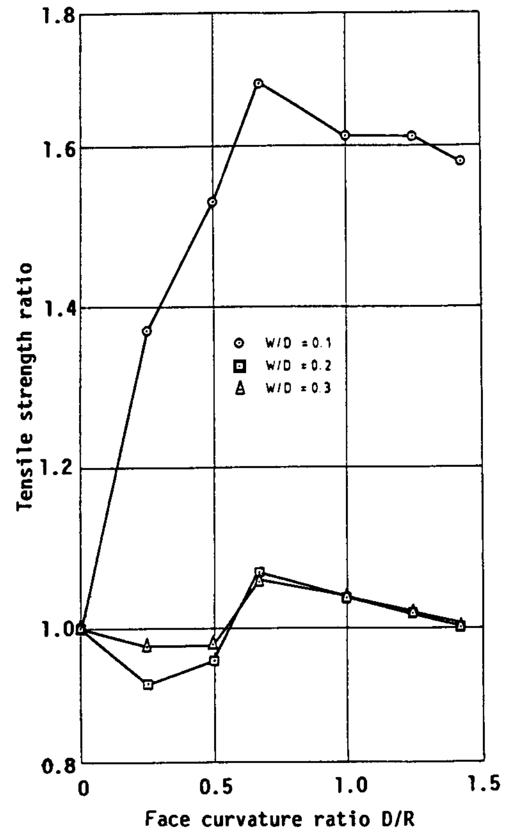

FIG. 3. Plot of material tensile strength values normalized with respect to the value for the corresponding plane-faced (D/R = 0) tablet, versus face curvature ratio for 40 MPa compaction pressure.

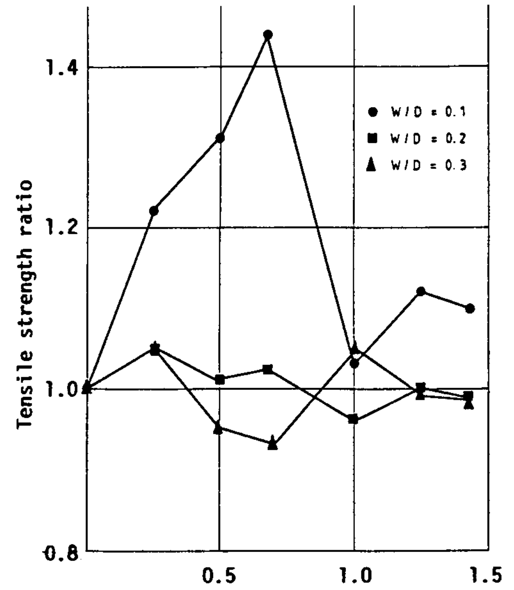

FIG. 4. Plot of material tensile strength values normalized with respect to the value for the corresponding plane-faced (D/R = 0) tablet, versus face curvature ratio for 320 MPa compaction pressure.

The relative independence of material tensile strength of face curvature for W/D values greater than, or equal to, 0·2 is probably associated with the greater volume of the central cylindrical part of these tablets compared with the volume of the curved end portions, and it would appear that the contribution of the former to the overall strength of the tablets dominates. For the thinner tablets (W/D = 0·1), the influence of changing punch curvature and die-wall friction on the powder flow characteristics in the compact and, consequently, on the material strength, appear to be much more significant. It might also be expected that the relative effects of these factors would be less the higher the compaction pressure. No simple explanation emerges for the marked contrast in the material tensile strength characteristics for W/D = 0·1 and D/R ≥ 0·67 at the two compaction pressures.

Conclusions

The work has clearly shown the important role of equation (2) in the assessment of the material tensile strength of convex-faced tablets.

Compaction at 320 MPa results in a very considerable strength increase compared with compaction at 40 MPa.

The material tensile strength of tablets with W/D ≥ 0·2 is practically independent of face curvature.

The material tensile strength of tablets with W/D = 0·1 attains a maximum value for a face curvature ratio (D/R) of 0·67, the conventional "normal" value.

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