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Brittle fracture propensity measurements on 'tablet-sized' cylindrical compacts

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The brittle fracture propensity test which was originally designed for large, square compacts with a central hole has been modified and extended to compacts of 'tablet-sized' dimensions. This allows a brittle fracture propensity (BFP) index to be measured at strain rates and conditions approaching those normally used in tableting. The BFP indices for microcrystalline cellulose, Tablettose and heavy magnesium carbonate were evaluated at punch velocities of 3·33 and 200 mm s^{-1} and found to be in good agreement with the results of previous workers.

Hiestand et al (1977) proposed the concept of a Brittle Fracture Propensity to quantify the tendency of materials to laminate during ejection. The method consisted of comparing the tensile strength of a 38·1 mm square compact containing a central hole of 1·09 mm diameter to that of the same sized compact without the hole and substituting the values into equation 1.

$$\mathrm{BFP} = 0{\cdot}5 \quad \left[\frac{\sigma_T}{\sigma_{To}} - 1\right] \qquad (1)$$

Where σ_{To} and σ_T are the tensile strengths of the square compacts with and without a central hole, respectively.

Hiestand et al (1977) considered the BFP factor to be the quantitation of stress relief by plastic deformation of the compact at the edge of the hole. If no stress relief occurs, the tensile strength should be approximately one-third of the tensile strength of a compact without a hole. However, if the stresses at the edge of the hole are relieved by plastic flow then no differences in the tensile strengths should be observed. Therefore a low value of the BFP index indicates the ability of a material to relieve localized stresses and a high value indicates a tendency of the material to laminate.

The method as described by Hiestand et al (1977) has obvious limitations in that the compacts need to be prepared using specialized punches and dies and a motorized hydraulic jack at conditions remote from those used in normal compaction. In the present study the technique has been extended to 15 mm diameter cylindrical compacts having a 1 mm diameter central hole and prepared using standard tableting conditions.

Materials and methods

Three materials expected to give a range of BFP values based on their compaction behaviour as previously reported (Roberts & Rowe 1985) were chosen for this study. These were microcrystalline cellulose (Avicel PH101-F.M.C. International, Food and Pharmaceutical Products, Ireland), spray-dried lactose (Tablettose, Meggle Michindustrie GmbH and COKG, West Germany) and heavy magnesium carbonate (Lohmann, West Germany).

Compression was carried out using the ICI High Speed Compression Simulator (Hunter et al 1976), fitted with specially designed, flat-faced, 'F type', tooling as shown in Fig. 1. The punches consisted of a 15 mm diameter lower punch with a 1 mm diameter central core rod allowing a maximum fill depth of 20 mm. The core rod was fixed flush into a hole in the lower punch and held in place by a grub screw on the side of the punch. To allow easier replacement in case of damage to the core rod, the hole passed through the length of the punch. The core rod was slightly pointed at the end to facilitate location into a similar sized hole in the upper punch.

The amount of material to give a 2·75 mm thick compact at zero porosity was calculated using the true densities of the materials, as determined previously (Roberts & Rowe 1985). A simple 'sawtooth' dis-

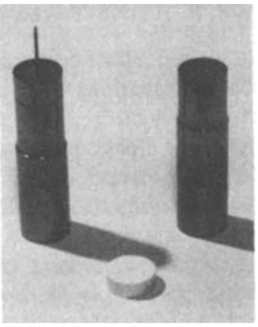

FIG. 1. Photograph of the specially designed 'F type' tooling and a typical compact with a central hole.

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* Correspondence.

placement/time profile was designed allowing the use of punch velocities of 3·33 and 200 mm s^{-1}, whilst keeping the ejection velocity constant at 15 mm s^{-1}. The die wall, punch faces and the central core rod were lubricated with a solution of 2% w/w magnesium stearate in carbon tetrachloride, facilitating both ejection of the compacts from the die and core rod removal, without the loss of material near the base of the core rod. A number of compacts were produced at various applied pressures to give a range of relative densities, for compacts with and without a central hole. The diametral breaking force of each of the compacts was evaluated using the CT40 strength tester (Engineering Systems, Nottingham) and converted to tensile strength using an equation suggested by Fell & Newton (1970).

Results and discussion

For the BFP index to be meaningful, pores in the compact must not act as sites of additional stress relief when a central hole is present. The only stress concentrator of significance which is capable of modifying the tensile strength of the compact must be the intentionally introduced hole. From an examination of Figs 2 and 3 it can be seen that for heavy magnesium carbonate compressed at a punch velocity of 3·33 mm s^{-1}, and to a lesser extent Tablettose compressed at both punch velocities, the BFP index shows some porosity-dependent behaviour. Hiestand & Smith (1984) found a similar increase in the BFP index with an increase in the relative density for sucrose and to a lesser extent spray-dried lactose. They did not question the validity of the index, but suggested that the BFP index should be measured at a single relative density and that a high relative density of 0·85–0·9 is preferable. A possible explanation of the porosity dependence of the BFP index can be ascertained from the work of Rossi (1968) who suggested that the modulus of a porous material can be significantly affected by changes in the shapes of pores, due to the stress concentration effects of these pores (2·0 for a spherical pore and 3·0 for a cylindrical pore) perpendicular to the axis of loading. Thus, as the porosity of the compact is reduced the shapes and orientation of the pores formed may vary, resulting in different stress relief interactions between the pores and the central hole. A further contributory factor may be cracks formed due to lamination of the compact during ejection. For compacts of heavy magnesium carbonate prepared at high relative densities, lamination occurred during ejection from the die. The effects of these cracks in the compact must clearly modify the value of σ_T thus giving an anomalous value of the BFP index. These effects probably account for the lack of dependence of the BFP index on the relative density for compacts of heavy magnesium carbonate prepared at the high punch velocity of 200 mm s^{-1} (Fig. 3), since lamination occurred at much lower relative densities.

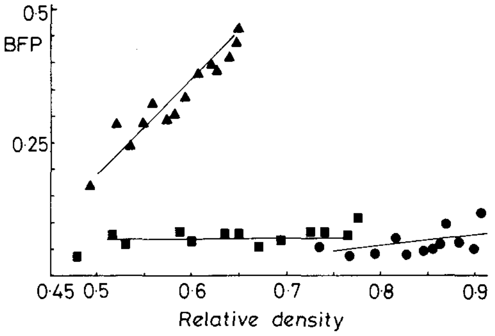

FIG. 2. The relation between brittle fracture propensity (BFP) and relative density for compacts produced at a punch velocity of 3·33 mm s^{-1} using the following materials: ▲, heavy magnesium carbonate; ●, Tablettose; ■, microcrystalline cellulose.

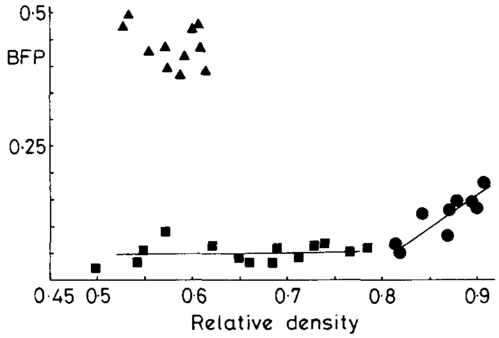

FIG. 3. The relation between brittle fracture propensity (BFP) and relative density for compacts produced at a punch velocity of 200 mm s^{-1} using the following materials: ▲, heavy magnesium carbonate; ●, Tablettose; ■, microcrystalline cellulose.

For comparison with results of other workers, BFP index values were calculated at stated relative densities as shown in Table 1. The values at a punch velocity of 200 mm s^{-1}, indicate the expected trend of increasing brittleness in the order microcrystalline cellulose > Tablettose > heavy magnesium carbonate. These also compare well with the values determined by Hiestand & Smith (1984) using square compacts (0·04 and 0·16 for Avicel PH102 and spray-dried lactose, respectively). However for a punch velocity of 3·33 mm s^{-1} the differences between microcrystalline cellulose and Tablettose (Table 1) were not as great. This may be due to differences in the modulus and stress distribution within the compacts, which would be expected to modify the brittleness of the specimens. Another interesting feature of the results is the increase in the BFP index with increasing punch velocity, particularly for the more brittle materials of Tablettose and heavy magnesium carbonate. This may be due to an increase in the modulus of the compact and thus provide an indication of the stress relieving ability of materials,

Table 1. The brittle fracture propensity index of microcrystalline cellulose, Tablettose and heavy magnesium carbonate at specified relative densities for punch velocities of 3·33 and 200 mm s^{-1}.

Material	Punch velocity mm s^{-1}	Relative density	BFP index
Microcrystalline cellulose	3·33	0·75	0·083
	200	0·75	0·055
Tablettose	3·33	0·90	0·073
	200	0·90	0·142
Heavy magnesium carbonate	3·33	0·60	0·359
	200	0·60*	0·487

* Compacts above a relative density of 0·6 had typical lamination type failure.

particularly considering that for microcrystalline cellulose there is little change in the BFP index with punch velocity, consistent with a good stress relieving material.

The close agreement of the values obtained here and those of earlier workers (Hiestand et al 1977, Hiestand & Smith 1984) is only to be expected because of the similarities in stress distribution patterns obtained using square and cylindrical shaped compacts (Stanley 1985). Furthermore, the ratio of stress in tension to compression at the centre of square compacts and cylindrical compacts are also similar—0·28 and 0·33, respectively (Berenbaum & Brodie 1959), thus identical stress concentration factors can be expected (Lipson & Juvinall 1963).

The only criticism of using 15 mm cylindrical compacts of tablet dimensions, is that in the analysis of the stress concentration factor it is based on a round hole in a semi infinite plate subjected to a uniform tensile stress. In this analysis the hole size must be very much less than the diameter of the plate. However from elasticity theory, Timoshenko & Goodier (1951) have evaluated the stress as a function of distance from the centre of the hole and it is evident from their analysis that the stress concentration around a hole is of a very localized character. For a cylindrical compact of the dimensions used in this study the stress concentration factor at the edge of the hole will be 3·0 while at four hole diameters from the centre of the compact the stress concentration factor reduces to 1·0082. It can also be calculated that the smallest sized compact that could be analysed using the BFP test is probably about 8 mm. Hence it is reasonable to assume that a compact of 15 mm will behave in an analogous manner to a square compact with a round hole and give similar values for the BFP index.

The method of calculating a brittle fracture propensity index developed by Hiestand et al (1977) based on square compacts has been successfully applied to compacts of tablet-sized dimensions. The method described has the obvious advantage in that tablets can be produced at strain rates approaching those normally used in tableting.

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