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The Effect of Machine Speed on the Consolidation of Four Directly Compressible Tablet Diluents

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Abstract—The reduction in porosity of a powder bed on compression was found to be a function of the velocity of the punch of the press. Substances which consolidated principally by fragmentation showed relatively little velocity dependence. However, the more important deformation was in a powder's consolidation mechanism, the greater the dependence on punch velocity and hence tablet press speed.

A frequently encountered phenomenon in tablet manufacture is that a formulation which produces tablets of acceptable quality on one tablet press may give rise to difficulties when transferred to another press or when the speed of the press is changed. It has been suggested by Jones (1981) that the time during which the particulate system is under a compressive load may be an important influence. Much of the work in this area has used contact times which are considerably longer than those encountered in normal tablet manufacture (e.g. David & Augsburger 1977; Rees & Rue 1978), and even when short contact times are used, the manner in which the punch moves may not be typical of that of a conventional press (Roberts & Rowe 1985).

In 1985, Armstrong & Blundell showed that the strength of tablets made from four directly compressible tablet diluents were, to a greater or lesser extent, dependent on the speed at which the tablets were produced. In all cases, an increase in speed led to a reduction in tablet strength. The aim of the present work was to discover why this should be.

Materials and Methods

Materials

Tablets were prepared from microcrystalline cellulose (Avicel PH102: K & K Greeff, London, UK), modified starch (Starex 1500; Colorcon, Orpington, UK), direct compression lactose (Fast-Flo lactose; Foremost, Baraboo, USA) and dicalcium phosphate dihydrate (Emcompress; Forum Chemicals, Reigate, UK).

Methods

A hand-operated Apex eccentric press was modified by fitting it with a 0·25HP electric motor, a flywheel weighing approximately 16 kg and a Kopp Variator so that machine speeds ranging from 0·33 to 2·67 rev s^{-1} could be obtained. This is equivalent to tablet production rates of 20 to 160 tablets min^{-1}. Flat-faced punches of diameter 12·5 mm were used. The upper punch was fitted with strain gauges and the distance between the faces of the upper and lower punches was measured by an LVDT. After amplification and conversion to digital form, transducer signals were stored and manipulated by a BBC Acorn computer. Full details are given elsewhere (Blundell 1986). Punch faces and the die wall were lubricated with a suspension of magnesium stearate in acetone.

Tablet crushing strength was measured on a CT40 strength tester (Engineering Systems, Nottingham) and the corresponding tensile strengths calculated according to the equation of Fell & Newton (1971). This equation corrects tensile strength for the porosity of the tablet and is of particular relevance in this case.

Results and Discussion

A number of workers, including Roberts & Rowe (1985) have shown that the consolidation mechanism of a powder may govern its response to changes in the speed of force application and the solids were chosen with this in mind. Dicalcium phosphate dihydrate has been shown to consolidate almost entirely by fragmentation (Duberg & Nystrom 1982), whereas modified starch undergoes plastic flow (David & Augsburger 1977). Microcrystalline cellulose and direct compression lactose consolidate by both mechanisms, though in the case of the former deformation predominates (David & Augsburger 1977). Lactose consolidates primarily by fragmentation, but directly compressible lactose contains some amorphous material which is capable of plastic flow (Vromans et al 1985).

Compression force was controlled by the weight of powder fed manually into the die, upper and lower punch settings being kept constant. This method permits a much greater reproducibility of applied force. It was noted that when compressing identical weights of the same powder, the force detected by the descending upper punch increased as the speed of the press was increased.

Fig. 1, as an example, shows the relationship between the weight of direct compression lactose in the die and the force detected by the punch in compressing that weight. For example, with 500 mg of direct compression lactose, forces of 10·7, 11·4 and 12·1 kN were detected when that weight was compressed at 0·33, 0·63 and 2·63 rev s^{-1}, respectively. Table 1 summarizes the forces detected via the punch when compressing given weights of the four substances at three compression speeds. The change of force varies with the substance involved, little difference being obtained with dicalcium phosphate dihydrate.

If the rate at which the load is applied exceeds the rate at

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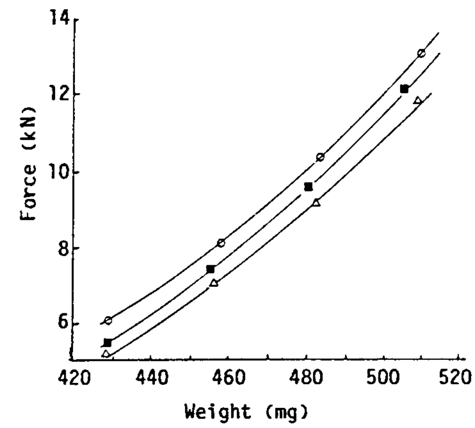

FIG. 1. Force-weight profiles of direct compression lactose compressed at three machine speeds (△, 0·33 rev s^{-1}; ■, 0·63 rev s^{-1}; ○, 2·63 rev s^{-1}).

which the powder can react to the force, then resistance to further densification will increase. Hence the porosity of the powder bed will be greater at any given force and tablet strength will be correspondingly reduced.

It is possible to measure the distance separating the punch faces, and so tablet thickness, and hence porosity, can be measured at any given force whilst the powder is being compressed. This has the advantage over conventional methods of measuring densification after tablet ejection from

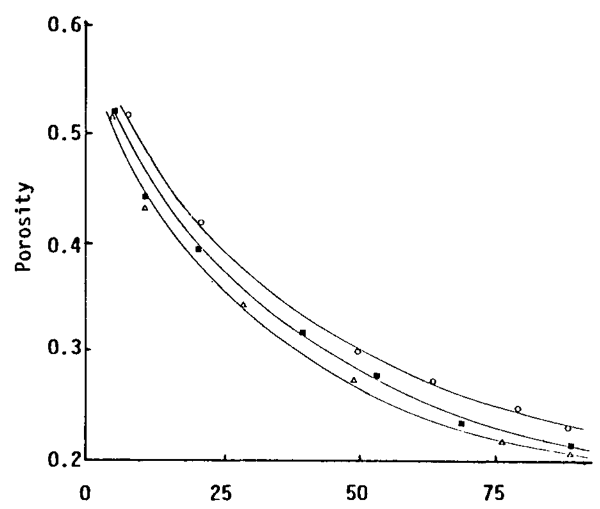

FIG. 2. The relationship between applied pressure and porosity for direct compression lactose at three machine speeds (Symbols as in Fig. 1).

the die in that the tablet has not had the opportunity to undergo elastic expansion.

Fig. 2 shows the relationship between porosity and pressure for the compression of direct compression lactose at three machine speeds. Each curve was constructed from between 100 and 200 points, but most of these have been omitted for reasons of clarity. At any given applied pressure, the porosity is higher at greater machine speeds, or alternatively, to achieve a given porosity, a higher pressure is needed.

Qualitatively similar results were obtained with the other three solids, though here too, quantitative differences were obtained.

Table 2 gives the pressures needed to achieve porosity fractions of 0·4 and 0·3 for all four solids. Thus the difference between these two pressures is the pressure needed to cause a reduction in porosity from 40 to 30%, and gives a measure of the compressibility of the solids.

Compressibility can also be estimated from the yield pressure of the solid. Porosity data were manipulated according to the Heckel equation (Heckel 1961) (Eqn 1).

$$\ln\frac{1}{1-D} = kP + A \qquad (1)$$

where $(1-D)$ is the porosity, P is pressure and k and A are both constants.

The yield pressure was calculated from the slope of the linear portions of the Heckel plots. In this case, linearity was assumed between pore fractions of 0·4 and 0·3, and the slope determined accordingly. The yield pressures are also given in Table 2.

In all cases, the pressures required to achieve given porosities increase as the machine speed is increased, as does the yield pressure. This confirms that the solid cannot react sufficiently quickly to the punch and an apparently more rigid body results.

The tensile strengths of tablets prepared over a range of compaction pressures were determined, and the strength-pressure profiles constructed. Interpolation at a pressure of 80MPa gives the strength data in Table 3.

Strength shows a reduction in all cases as speed is increased. However, it must be remembered that at different speeds, but at constant pressure, different porosities are obtained. The tensile strength can be corrected for porosity differences at 80MPa by reference to Fig. 2 and similar data which are available for the other solids. As the relationship between strength and porosity for each substance is known, it follows that the tensile strength can be calculated to take this into account. These data are also in Table 3. Differences between tensile strengths at the three speeds still exist, but are now much smaller. It is therefore likely that strength

Table 1. Forces detected on the punch face when compressing a given weight of solid at three compression speeds.

Solid	Weight (mg)	Maximum force (kN) detected at 0·33 rev s^{-1}	0·63 rev s^{-1}	2·63 rev s^{-1}
Microcrystalline cellulose	350	10·6	10·9	11·4
Modified starch	500	10·6	10·9	11·7
Lactose	500	10·7	11·4	12·1
Dicalcium phosphate	700	8·6	9·0	9·0

Table 2. Pressures required to give porosities of 40% (P_{40}) and 30% (P_{30}) of four solids compressed at three machine speeds, and yield pressures derived from the Heckel equation.

Machine speed (rev s^{-1})	Pressure (MPa)	Microcrystalline cellulose	Lactose	Modified starch	Dicalcium phosphate
0·33	P_{40}	34·0	17·8	34·8	14·6
	P_{30}	56·7	42·1	77·7	42·1
	P_{30}–P_{40}	22·7	24·3	42·9	27·5
	Yield	79·0	84·7	149·6	95·9
0·63	P_{40}	37·3	19·4	38·1	16·2
	P_{30}	61·6	45·4	82·6	47·8
	P_{30}–P_{40}	24·3	26·0	44·5	31·6
	Yield	84·7	90·2	155·2	110·1
2·63	P_{40}	43·7	22·7	45·4	17·8
	P_{30}	68·9	51·8	94·8	50·2
	P_{30}–P_{40}	25·2	29·1	49·4	32·4
	Yield	87·5	101·6	177·1	112·8

Table 3. Tablet tensile strengths (MPa) produced by a compressing force of 80 MPa, before and after correction for porosity changes.

Machine speed (rev s^{-1})	Microcrystalline cellulose	Lactose	Modified starch	Dicalcium phosphate
Before correction				
0·33	5·4	0·77	0·28	0·63
0·63	5·2	0·76	0·25	0·62
2·63	3·9	0·68	0·15	0·60
After correction				
0·33	5·4	0·77	0·28	0·63
0·63	5·3	0·77	0·27	0·62
2·63	5·4	0·79	0·24	0·64

differences brought about by a change in machine speed are fundamentally caused by the different porosities achieved during the compaction process.

The above changes in speed have been expressed in terms of machine rate. However, the solid responds to the speed at which the punch is actually moving, and the relationship between this and the machine rate is determined by the dimensions of certain components of the press. Armstrong et al (1983) derived an equation which describes the pattern of punch movement in an eccentric press, though it has recently been shown that, in practice, punch movement deviates from that predicted (Armstrong & Palfrey 1987). Punch velocity changes throughout the compaction process, and the speed at which the punch is travelling when it first touches the powder bed will further depend on the amount of powder contained in the die. However, from realistic assumptions regarding the height of the powder bed and the ultimate tablet thickness, punch velocities relating to the above work can be calculated. A punch attached to the Apex press rotating at 0·33 rev s^{-1} will have a velocity of about 40 mm s^{-1} when it first touches the powder, and this velocity will fall to zero at maximum punch penetration into the die. An increase in machine rate causes a proportional increase in punch velocity. Thus at 0·63 rev s^{-1}, the velocity at first contact is about 76 mm s^{-1} and at 2·63 rev s^{-1}, about 320 mm s^{-1}. Hence the changes in tablet strength and porosity reported in this communication would be expected to occur in any press showing a range of punch velocities of this order. Such presses include many rotary presses such as the Manesty B3B and Express, and the Kilian F1000 (Armstrong, in press).

A longer exposure to compression allows greater consolidation from whence the tablet derives its strength. This work provides further evidence that speed dependency is related to the consolidated mechanism of the solid. Fragmentation of Emcompress is rapidly achieved and prolonging exposure to the force has no further effect. Deformation is a more time-dependent process and so tablets made from substances which consolidate primarily by deformation benefit from increasing the time over which they are exposed to force by, for example, decreasing production rate.

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