

The effect of punch velocity on the compaction of a variety of materials

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The effect of punch velocity over the range 0.033–400 mms⁻¹ on the compaction of a variety of materials has been studied using constants derived from the Heckel equation as criteria to describe their behaviour. For materials known to deform plastically, e.g. maize starch and polymeric materials, there was an increase in the yield pressure with punch velocity attributable to a change either from ductile to brittle behaviour or a reduction in the amount of plastic deformation due to the time dependent nature of plastic flow. For materials known to consolidate by fragmentation, e.g. magnesium and calcium carbonates, there was no change in yield pressure with increasing punch velocity. The data has been analysed in terms of the strain rate sensitivity of the materials calculated from their yield pressure at low and high punch velocities.

The importance of the time-dependent deformation of pharmaceutical materials has been recognized for sometime now. Several investigators, in particular Shlanta & Milosovich (1964) have used stress relaxation measurements to examine the plastic flow of materials at constant strain. More recently David & Augsburg (1977) analysed stress relaxation data based on the Maxwell model of viscoelastic behaviour in order to quantify the plastic deformation rates of some direct compression materials.

Since plastic deformation is a time dependent phenomenon, it has long been recognized that compression rate will effect the extent to which this occurs. Several investigators (Baba & Nagufuji 1965; Seitz & Flessland 1965) have shown a decrease in the breaking strength of compacts with an increase in the machine rate. Other authors interpreted these trends as a decrease in plastic deformation due either to a reduction in the time available for stress relaxation and thus bond formation, or a change to a fragmentation type mechanism (Hiestand et al 1977; Ritter & Sucker 1980). Rees (1980) emphasized that the strength of a perfectly elastic-brittle particle shows no rate dependence, whereas for a viscoelastic particle capable of plastic deformation its strength may be increased by several orders of magnitude at high strain rates. For example David & Augsburg (1977) examined various direct compression materials at compression cycle durations of 0.09 and 10 s. They found no increase in compact strength for the relatively brittle materials Fast-Flo lactose and Dipac sugar, in contrast to the plastically deforming

materials microcrystalline cellulose (Avicel PH101) and modified starch (Sta-Rx 1500). These results confirm the importance of plastic deformation in creating strong bonds within a compact. Rees & Rue (1978) have examined the effect of changes of relative density utilizing the Heckel equation (1961a, b) on the 'contact time', which Jones (1977) described as the total time that a detectable force is applied to the die contents during the compression and decompression event. Tablet density increased with an increase in 'contact time' for Avicel PH101 and Sta-Rx, due to prolongation of the time available for plastic deformation. However Emcompress, a brittle material, showed no changes in density with 'contact time'.

The strain rate sensitivity of materials has not been investigated to any great extent in the literature. However, recently, Barton (1978) investigated the effect of strain rate on the degree of volume reduction of sodium chloride, potassium bromide and Dipac sugar and found that larger volume reductions occurred at the lower rates. Al-Hassani & Es-Saheb (1984) compared several materials at a range of strain rates from 10⁻³–10⁴ s⁻¹ and found that at 40% axial strain, the axial compression pressure increased significantly above 10² s⁻¹, which is above the range of strain rates encountered on most compression machines. They attributed the increase to be as a consequence of a change in deformation mechanism from ductile to brittle behaviour.

The purpose of the present study is to investigate the effect of punch velocity on the yield stress as determined by Hersey & Rees (1972) using an equation derived by Heckel (1961a, b) and thus

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Table 1. Bulk, tap and true densities for the materials used in this study.

Material	Description	Manufacturer	Bulk density g cm ⁻³	Tap density g cm ⁻³	True density g cm ⁻³
Heavy magnesium carbonate	3MgCO ₃ Mg(OH) ₂ ·4H ₂ O	Lohmann, West Germany	0.484	0.622	2.187
Calcium phosphate	Ca ₃ (PO ₄) ₂	Albright and Wilson Ltd	0.386	0.614	3.103
Calcium carbonate	CaCO ₃	J and E Sturge Chemicals Ltd	0.341	0.587	2.827
Paracetamol		Thornton and Ross Ltd	0.218	0.379	1.275
Paracetamol D.C.	Spray dried with 4% hydrolyzed gelatin	Graesser Salicylates Ltd	0.378	0.498	1.288
Lactose B.P.	α-Monohydrate	Serolac Dairy Crest	0.394	0.687	1.541
Anhydrous lactose USP	β-anhydrous granulated	Humko Sheffield Chemical Co	0.543	0.752	1.545
Tabletose	α-Monohydrate spray-dried	Meggle Milchindustrie GMBH	0.529	0.669	1.529
Avicel PH101	Microcrystalline cellulose	FMC Corporation	0.276	0.385	1.534
Sodium chloride	NaCl Analar grade	B.D.H. Chemicals Ltd	1.159	1.288	2.130
Mannitol	D-Mannitol	C.C.A. (Biochem), Holland	0.595	0.752	1.475
Maize starch		Laing National Ltd	0.583	0.723	1.475
Corvic R46/88	Co-polymer: 85% vinyl chloride, 15% vinyl acetate	ICI Plastics Division	0.776	0.839	1.376

evaluate the strain rate sensitivity of some commonly used pharmaceutical materials.

MATERIALS AND METHODS

Materials

The materials examined in this study (Table 1) were chosen because they are among the more commonly used excipients. The materials were used as received from the respective manufacturers. True densities were determined using an air comparison pycnometer (Beckmann Model-930), five determinations for each material were carried out and the mean calculated. Bulk density measurements were performed by pouring a quantity of powder into a 250 cm³ glass cylinder, tilted to an angle of 45°. Tap density was determined using the same cylinder mounted on a Jolting volumeter, all readings taken at a limiting volume (approximately 1000 taps).

Compression

Compression was carried out using the ICI High Speed Compression Simulator (Hunter et al 1976) fitted with 10 mm flat faced punches. Uniaxial compression was provided by using a simple 'saw-tooth' displacement/time profile for the top punch, the lower punch remaining stationary. Punch velocities chosen for the experiment ranged from 0.033–400 mms⁻¹. These speeds encompass the range of velocities encountered during research using physical testing machines (0.05–5 cm min⁻¹), single punch machines (50–150 mms⁻¹) and during production using rotary machines (100–400 mms⁻¹). To allow direct comparison of all the materials, the

amount of material required to give a 3.5 mm thick compact at zero porosity was calculated from the true densities. Measurements of displacement and force were performed to an accuracy of ±10 μm and ±0.025 kN respectively. The elastic deformation to the punches and other parts of the simulator were evaluated to allow true displacements to be determined. The displacements and forces were monitored throughout the compression cycle with readings being captured and fed through a 4-channel (A to D) converter to a PET microcomputer (CBM 3032). The actual number of points captured during each compression cycle was 1000 points; however over the actual compression event only 100 to 600 points could be captured, depending on the material to be compressed and punch velocity.

For Avicel PH101 at the slowest velocity, ten compressions were performed for two batches to check reproducibility of the system and method. For the other materials three compressions were carried out at each punch velocity. The die was cleaned with acetone after each compression and lubricated with a solution of 2% w/w stearic acid in carbon tetrachloride. Powder was accurately weighed out and poured into the die cavity, ensuring uniform distribution of material.

When the data from one compaction event had been processed, details of the corrected tablet thickness and upper and lower forces were stored on floppy disc for later processing.

Manipulation of the data

In the present study the Heckel equation (Heckel 1961a, b) was used to analyse the relationship

between relative density measured during compaction and the applied pressure.

$$\ln [1/(1 - D)] = KP + A \quad (1)$$

The relative density D is given by the ratio between the apparent density of the compact at pressure P and the true density of the powder. K is the slope of the straight line portion, the reciprocal of which is referred to as the mean yield pressure P_y . From the value of A the intercept, the relative density D_A , can be calculated using equation (2)

$$D_A = 1 - e^{-A} \quad (2)$$

Although there is no fundamental basis for the prediction or inference of compaction behaviour from D_A , it has been evaluated for comparative purposes. The value of D_0 was obtained from the relative density of the powder bed at the point when a measurable force is applied. The straight line portion was determined mathematically using suitable software which calculated the double derivative of the plot to give an evaluation of the pressure where constant slope started and ended. It was not possible to use the above method for the calculation of the yield pressures, of calcium carbonate, heavy magnesium carbonate and calcium phosphate, because of the curvilinear nature of the Heckel plots. This confirms the findings of Armstrong & Morton (1979) for the compaction of magnesium carbonate. Reproducible values of the constants were obtained by comparing the correlation coefficients obtained from the regression analysis, for a range of selected pressures at one particular speed. The pressure ranges which gave the 'best fit' for the three materials were 150–350 MPa-calcium carbonate, 75–200 MPa-heavy magnesium carbonate and 160–300 MPa-calcium phosphate. These ranges were used in the subsequent analysis of the Heckel plots for the evaluation of the change of P_y with punch velocity.

RESULTS AND DISCUSSION

Two batches of Avicel PH101 were chosen to investigate the accuracy of the method. For a punch velocity of 0.033 mms^{-1} the constants P_y , D_A and D_0 with their respective \pm standard deviations were for Batch 1: $68.9 \pm 2.786 \text{ MPa}$, 0.507 ± 0.0096 , 0.264 ± 0.0026 and for Batch 2: $68.2 \pm 1.481 \text{ MPa}$, 0.516 ± 0.0042 , 0.262 ± 0.0046 . The low values obtained for both batches reflect the confidence in the experimental procedure and the accuracy in the method of analysis of the results.

The shapes of the Heckel plot can give a good indication of the type of mechanism occurring during compression. Typical plots are shown in Figs 1, 2,

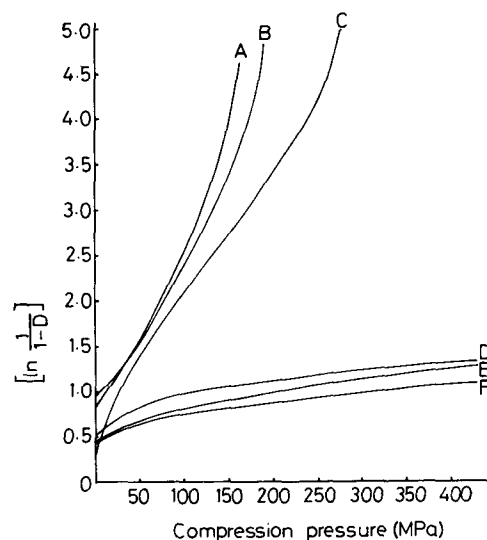


Fig. 1. Heckel plots at 3.33 mms^{-1} for: (A) maize starch, (B) Corvic R46/88, (C) Avicel PH101, (D) calcium carbonate, (E) heavy magnesium carbonate, (F) calcium phosphate.

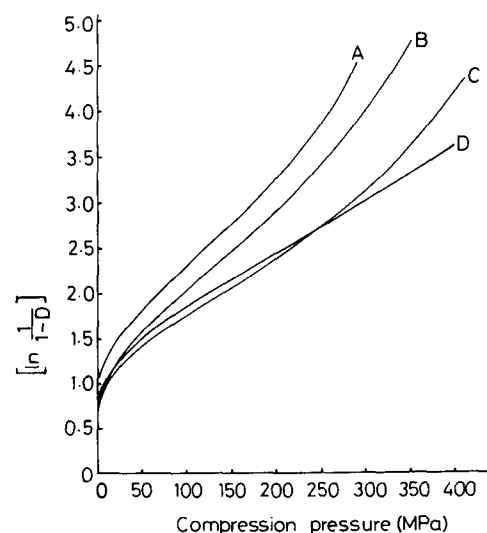


Fig. 2. Heckel plots at 3.33 mms^{-1} for: (A) paracetamol drug, (B) paracetamol D.C., (C) anhydrous lactose, (D) Tablettose.

and 3 for a punch velocity of 3.33 mms^{-1} . The most striking aspect of the plots is the relative incompressibility of the inorganic minerals, calcium phosphate, heavy magnesium carbonate and calcium carbonate with apparent porosities at 400 MPa of 34.3, 28.3 and 26.5% respectively (cf. lactose 2.3%). The high apparent porosities are as a result of a mechanism of deformation involving fragmentation

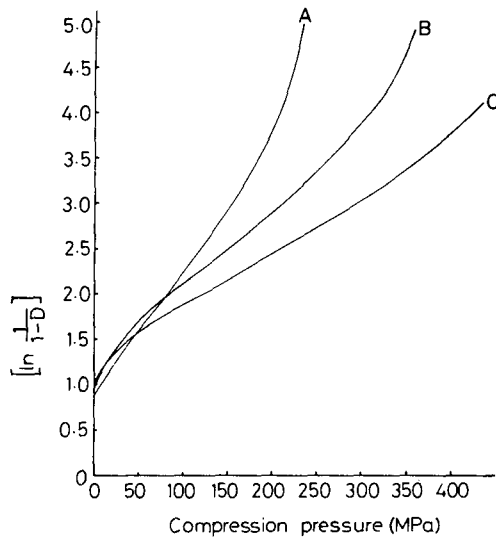


FIG. 3. Heckel plots at 3.33 mms^{-1} for: (A) sodium chloride, (B) mannitol, (C) crystalline lactose.

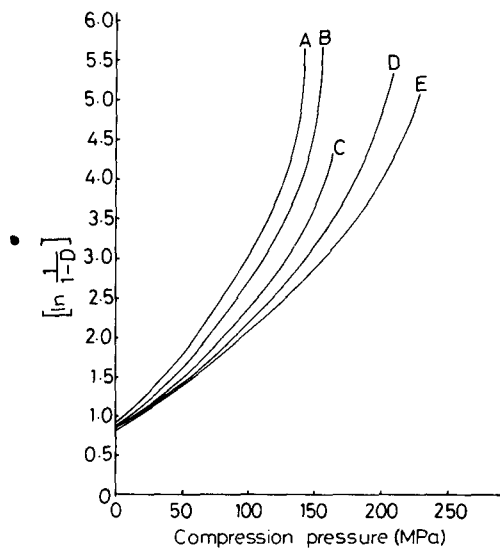


FIG. 4. Heckel plots for maize starch at the following punch velocities: (A) 0.33 , (B) 1.00 , (C) 3.33 , (D) 33.33 , (E) 166.67 mms^{-1} .

and bonding by interlocking at particle contacts. This is quite likely because the high inherent particle hardness of these inorganic minerals would tend to oppose a plastic deformation mechanism. Armstrong & Haines-Nutt (1970) reported this mechanism to be probable for the compaction of magnesium carbonate at pressures up to 140 MPa, based on measurements of particle size distributions of disintegrated tablets produced at a range of pressures.

Consolidation by a fragmentation mechanism would result in an increase in the dislocation density of the crystals, thus strain hardening effects could become important particularly in the latter stages of densification. This resistance to densification has been reported by Seelig & Wulff (1946) for the compaction of metals and probably accounts for the flat portion in the Heckel plots.

In contrast, maize starch and Corvic R46/88 show a large reduction in volume with pressure, apparent porosities at 150 MPa being 2.25 and 3.09% respectively (cf. lactose 11.51%). The plots also show considerable curvature in the opposite direction to the other materials, indicative of a mechanism involving considerable reluctance to deformation in the initial stages of compaction (0–50 MPa), as the elastic deformation of the particles is overcome. Between 50–100 MPa yielding of the material takes place as indicated by the linear portion in the plot. Such a mechanism has been proposed by Palmer & Rowe (1974), based on photomicrographs. They showed that the spherical shape of the particles is still discernible at 50 MPa applied pressure, whereas at 220 MPa the particles consist of regular polyhedra, suggesting considerable squashing of contact points. Paronen & Juslin (1983) showed similar particulate structures with increasing pressure for maize starch. The permanent deformation of both these materials when the stress is removed is limited due to their high elastic recoveries. In the present study the axial recovery during decompression was approximately 10.0% for Corvic R46/88 and 7.1% for maize starch, compared with only 2.6% for calcium carbonate. Above 100 MPa considerable curvature of the plots occurs, this indicates an increase in deformation. However, examination of the Heckel plots of some of the other materials also show this curvature to be present. This is most likely to be as a consequence of the limitations of the Heckel equation. As the density of the material under load approaches the true density of the powder then $\ln(1/(1-D))$ approaches infinity and rapid curvature of the plot is thus seen to occur.

Sodium chloride shows some similarities to Corvic R46/88 and maize starch, in the shape of the Heckel plot at pressures above 150 MPa. However resistance to deformation in the initial phase does not occur, probably due to a feature of the cubic shaped nature of the particles. To achieve close-packing, considerable slippage and rearrangement of the crystals would occur before the onset of plastic deformation. Photomicrographs presented by Down (1983) over the pressure range 0–35 MPa have shown

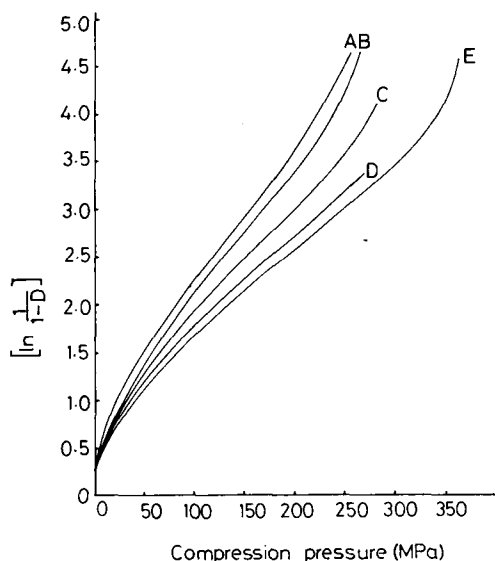


Fig. 5. Heckel plot for Avicel PH101 at the following punch velocities: (A) 0.33, (B) 3.33, (C) 20.00, (D) 66.67, (E) 300 mms^{-1} .

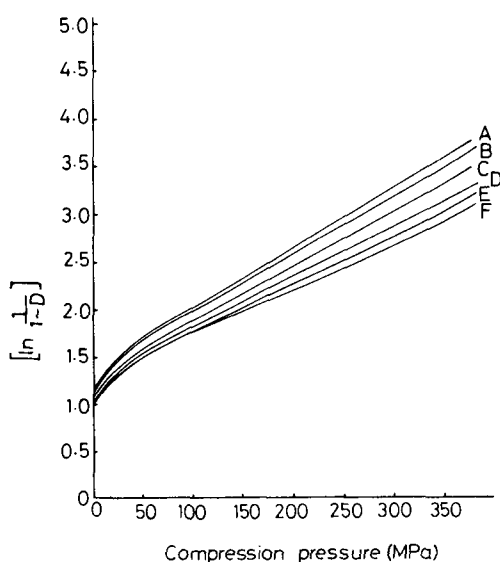


Fig. 6. Heckel plots for crystalline lactose at the punch velocities: (A) 0.033, (B) 0.33, (C) 3.33, (D) 33.33, (E) 166.67, (F) 390 mms^{-1} .

considerable movement of the crystals. Bonding is thought to occur by localized brittle fracture at the interface between crystals followed by extensive plastic flow. The disappearance of cracks and flow of material into indentations on the surface of crystals has provided evidence for such a deformation mechanism (Down 1983).

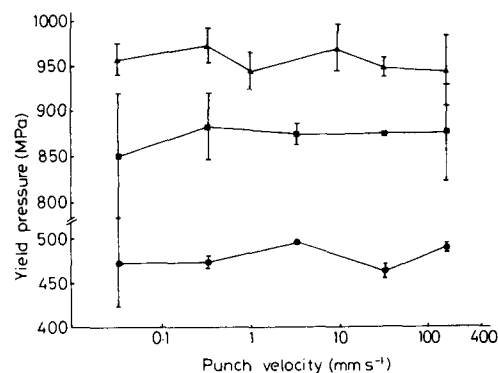


Fig. 7. Yield pressure versus punch velocity for: ▲, calcium phosphate; ■, calcium carbonate; ●, heavy magnesium carbonate.

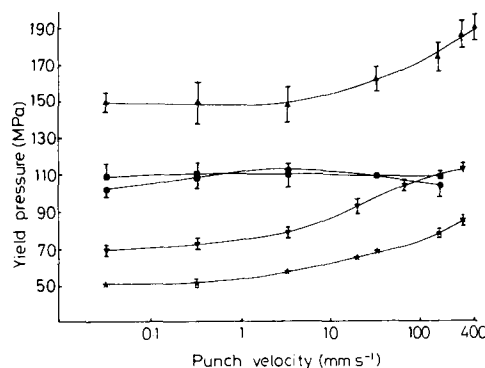


Fig. 8. Yield pressure versus punch velocity for: ▲, anhydrous lactose; ■, paracetamol D.C.; ●, paracetamol drug; ▼, Avicel PH101; ★, Corvic R46/88.

The mechanism for the compaction of Avicel is well documented. Reir & Shangraw (1966), Lamberston & Raynor (1976) reported that extensive plastic flow occurs during compression due to numerous slip-planes and dislocations present in its structure. David & Augsburger (1977) confirmed this by measurements on its stress relaxation behaviour. From the present study Avicel PH101 was found to consolidate rapidly with increasing pressure as compared with the other excipients; this is discernible in view of the steepness of the gradient.

The Heckel plots for maize starch, Avicel PH101 and crystalline lactose at various punch velocities are presented in Figs 4, 5 and 6 respectively. Two trends are immediately obvious from the plots. Firstly the slope decreases with increasing punch velocity and secondly the curves move down the y-axis, indicating a decrease in densification. These effects will tend to increase the yield pressure P_y and decrease the value of D_A , evaluated from the intercept. The effect of

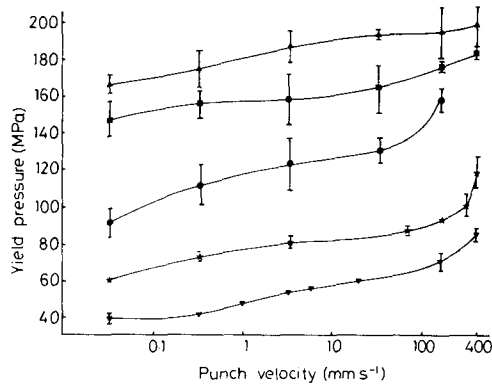


FIG. 9. Yield pressure versus punch velocity for: ▲, crystalline lactose; ■, Tablettose; ●, mannitol; ★, sodium chloride; ▼, maize starch.

punch velocity on the yield pressure P_y of the material is shown in Figs 7, 8 and 9.

The inorganic minerals, calcium carbonate, calcium phosphate and heavy magnesium carbonate show no change in the yield pressure with punch velocity, as would be expected for materials which consolidate by fragmentation. Rees & Rue (1978) found a similar effect for the comparison of the Heckel plots of dicalcium phosphate at two 'contact times'. High values of the yield pressure P_y , are also indicative of materials that consolidate by brittle fracture.

An interesting feature of the graphs of yield pressure versus punch velocity is the little difference between the two grades of paracetamol. Paracetamol D.C. consists of spherical particles composed of an outer shell of 4% hydrolysed gelatin and an inner core of drug crystals, similar to those prepared by Seager et al (1982). During compression this structure is easily broken down and deformation proceeds as if the material was pure paracetamol as indicated by the Heckel plots. The main difference between the two grades of paracetamol is the amount of elastic recovery that occurs during decompression. For a punch velocity of 3.33 mms^{-1} the axial recoveries of the materials were 9.2 and 5.8% for paracetamol drug and paracetamol D.C. respectively. The other feature of the compression characteristics of these two materials is that a fragmentation mechanism is dominant as indicated by the insignificant change in P_y with punch velocity. However conflicting evidence has been reported (Doelker & Shotten 1977) suggesting that they both show some degree of plastic flow and brittle behaviour (Humbert-Droz et al 1983). Thus, the process may be one of mixed behaviour and this certainly may be

Table 2. Values of the SRS (strain rate sensitivity) of the materials.

Material	SRS %
Calcium phosphate	—
Calcium carbonate	—
Heavy magnesium carbonate	—
Paracetamol D.C.	1.8
Paracetamol drug	10.6
Lactose	16.2
Tablettose	19.2
Anhydrous lactose	20.3
Avicel PH101	38.9
Sodium chloride	39.9
Mannitol	46.4
Maize starch	49.3
Corvic	54.1

the case as is evident from the value of the yield pressure of approximately 110 MPa.

The mechanism of compaction of lactose has been reported to be by fragmentation (Hersey et al 1973; Cole et al 1975). Yield pressures for the three lactoses, anhydrous, spray-dried and crystalline at a punch velocity of 3.33 mms^{-1} were 149, 156 and 175 MPa respectively. This indicates that the crystalline lactose is considerably more brittle than the processed grades. This may be due to the more angular nature of conventional crystalline lactose as compared with the aggregated collection of smaller crystals of the direct compression lactoses (Shangraw et al 1981). The graphs of yield pressure versus punch velocity indicates that there is some strain rate dependence. This suggests that a certain amount of plastic deformation is occurring at the lower punch velocities. For anhydrous lactose (Rees & Rue 1978) found differences in the Heckel plots at two 'contact times', thus supporting this hypothesis. However, in a similar experiment performed on spray-dried lactose, McKenna & McCafferty (1981) suggested that brittle fracture was occurring. Comparisons between the present study and both these works is not strictly applicable, as densities were measured on ejected compacts and the 'contact time', as applied includes the additional component, dwell time, which may be the factor contributing to the differences in densification behaviour.

Most of the materials show increasing resistance to deformation with punch velocity, especially at the higher speeds. These changes occur over the range of punch velocities associated with commercial high-speed rotary compressors.

The mechanism for an increase in the yield pressure with punch velocity could be one of a reduction in the amount of plastic deformation, due to the time dependent nature of plastic flow and thus

Table 3. Values of D_O and D_A at varying punch velocities.

Materials	Punch velocity mms^{-1}													
	0.033		0.33		3.33		33.33		166.67		300		390	
	D_O	D_A	D_O	D_A	D_O	D_A	D_O	D_A	D_O	D_A	D_O	D_A	D_O	D_A
Calcium phosphate	0.357	0.509	0.355	0.507	0.327	0.492	0.325	0.488	0.324	0.483	—	—	0.295	0.481
Heavy magnesium carbonate	0.368	0.468	0.369	0.467	0.358	0.454	0.361	0.458	0.357	0.463	—	—	—	—
Calcium carbonate	0.405	0.597	0.409	0.601	0.401	0.588	0.364	0.581	0.350	0.577	—	—	—	—
Anhydrous lactose	0.592	0.697	0.594	0.691	0.549	0.667	0.547	0.669	0.524	0.650	0.533	0.664	0.498	0.663
Lactose	0.671	0.758	0.658	0.755	0.639	0.727	0.617	0.720	0.608	0.711	—	—	0.606	0.715
Tabletose	0.622	0.747	0.611	0.738	0.549	0.711	0.536	0.704	0.522	0.716	—	—	0.516	0.700
Paracetamol D.C.	0.589	0.724	0.575	0.714	0.514	0.695	0.512	0.689	0.482	0.670	—	—	—	—
Mannitol	0.672	0.763	0.656	0.756	0.592	0.727	0.585	0.708	0.582	0.721	—	—	—	—
Avicel PH101	0.264	0.507	0.260	0.572	0.263	0.543	0.262	0.520	—	—	0.265	0.538	—	—
Paracetamol drug	0.694	0.775	0.693	0.781	0.648	0.765	0.641	0.757	0.623	0.718	—	—	—	—
Sodium chloride	0.633	0.636	0.625	0.660	0.588	0.622	0.590	0.605	0.580	0.610	0.586	0.621	0.578	0.635
Corvic	0.614	0.569	0.616	0.537	0.601	0.489	0.602	0.514	0.600	0.525	0.599	0.539	—	—
Maize starch	0.586	0.467	0.584	0.439	0.559	0.444	0.564	0.494	0.551	0.473	—	—	0.575	0.559

Table 4. Differences in the values of the two constants D_O and D_A between the punch velocities 0.033 and 166.67 mms^{-1} .

Material	Difference in D_O	Difference in D_A
Avicel PH101	0.001	0.034
Heavy magnesium carbonate	0.011	0.005
Corvic	0.014	0.044
Calcium phosphate	0.033	0.026
Maize starch	0.035	-0.006
Sodium chloride	0.050	0.026
Calcium carbonate	0.055	0.020
Lactose	0.063	0.047
Anhydrous lactose	0.068	0.047
Paracetamol	0.071	0.057
Mannitol	0.090	0.042
Tabletose	0.100	0.031
Paracetamol D.C.	0.107	0.054

bond formation and/or an increase in brittle behaviour. For the plastic materials Avicel PH101, maize starch and Corvic R46/88, the former mechanism is more likely. Rees (1980) suggested that particle strength increases by several orders of magnitude at very high rates of loading resulting in an increased particulate resistance to deformation. This will result in high stress concentrations at interparticulate contacts and reduction in these stresses is achieved by fragmentation of particles. For the remainder of the materials, changes from ductile to brittle behaviour probably accounts for the increases in yield pressure.

Due to the curved nature of the plots of yield pressure with punch velocity, it is not feasible to determine the strain rate sensitivity (SRS) from the slope. However, using the equation below, some indication of the SRS can be evaluated and is presented in Table 2.

$$\text{SRS} = \frac{P_{y2} - P_{y1}}{P_{y2}} \times 100$$

Where P_{y1} = the yield pressure at 0.033 mms^{-1} , and P_{y2} = the yield pressure at 300 mms^{-1} .

Calculation of the SRS for the inorganic minerals was not possible, due to the variations in the graph (Fig. 7), but they are included for comparison purposes in their expected positions. The materials which are more strain rate sensitive are those materials which plastically deform, as would be expected from the above discussion. This order compares well with that presented by Rees & Rue (1978), who found that tablet density increased due to prolonging the time available for deformation in the rank order: modified starch > microfine cellulose > anhydrous lactose > sodium chloride > dicalcium phosphate.

For all the materials, D_O and D_A tend to decrease with punch velocity as shown in Table 3. The decrease of D_O with punch velocity is due to a decrease in the rearrangement and particle slippage phase of densification of the powder bed. This could be due to an increase in the frictional and adhesive forces between particles opposing the rearrangement process. At higher punch velocities, the effects of air escape from the powder bed, due to disturbance of such packing processes may have a secondary effect. The value of the applied pressure required to effect particle slippage and thereby rearrangement, will be related to the surface structure, size and shape of the particles.

The difference between the value of D_O at 0.033 and 166.67 mms^{-1} is presented in Table 4. This gives some indication of the mechanisms involved. For paracetamol D.C., Tabletose and mannitol there is a large change in D_O with punch velocity, indicative of a particle fracture mechanism during the rearrangement phase.

As particle size decreases so the number of contact points per unit area increases resulting in higher

interparticulate cohesive forces which would tend to oppose dense packing conditions leading to a decrease in D_O . York (1978) found that as particle size of lactose decreased, D_O decreased, thus supporting the present study.

A small change in D_O with punch velocity is indicative of spherical or regular shaped particles. This is probably the case for maize starch, Corvic R46/88 and to a certain extent sodium chloride, because regular shaped particles pack densely, as indicated by the little difference between bulk and tap densities (Table 1). Therefore punch velocity will have little effect on the rearrangement processes involved during the initial densification. The inorganic minerals also show little change in D_O with punch velocity as is evident from Table 4. Initial rearrangement will result in a packing structure of high porosity due to the ability of the irregular shaped surface of the particles to interlock. Due to this packing structure, any further imposed load will be dissipated, reducing stress concentrations and thus filling of intraparticle voids by particle breakage will be reduced. Calcium carbonate has also been shown to exhibit high cohesive and frictional forces (Kokova & Pilpel 1971) and this may also contribute to a resistance to particle slippage and rearrangement.

The difference in D_A values calculated at the two punch velocities are presented in Table 4 and no trends are evident. This was the expected result because D_A is not related to any fundamental physical process of compaction or material property and is only a mathematical constant in the Heckel equation.

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