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An evaluation of the techniques employed to investigate powder compaction behaviour

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Summary

Employing 4 model powders, Avicel, Dipac, mannitol and paracetamol and a single-punch tablet machine, the most common techniques used to investigate powder compaction behaviour are compared and assessed. The use of tablet tensile strength rather than crushing force is found to bias the interpretation of lower punch work (LPW_1)/hardness profiles when compacts of very different thicknesses are produced. Radial versus axial pressure cycles, maximum die wall pressure and lower punch work on recompression (LPW_2) are demonstrated to be insensitive comparative measurements of compaction behaviour. Further, the latter suffers from considerable interpretive difficulties. Stress relaxation measurements are employed to verify data obtained from residual die wall pressure determinations and Heckel plots. Thus, the use of LPW_1/crushing force profiles, residual die wall pressure versus axial pressure and elastic recovery versus LPW_1 plots are simple and efficient techniques that assist in defining powder compaction behaviour.

Introduction

With the current widespread use of instrumented tablet machines, considerable information exists in the literature on the compaction behaviour of numerous pharmaceutical powders. However, the use of many varied, and often unsubstantiated, techniques has led to a great deal of confusion and discrepancy in the literature.

Therefore the aims of this investigation are to outline the most commonly used procedures for assessing powder compaction behaviour; to experimentally verify the validity and applicability of each technique; and to critically compare each technique.

Of the various procedures apparent in the literature, the following techniques appear to be the most commonly used in assessing various aspects of powder behaviour during the compaction process.

Energy of compaction

Since powders with different packing characteristics and different elastic and plastic deformational properties will absorb varying amounts of energy during compaction for equally applied pressures, it may be more useful to correlate energy of compaction, rather than applied pressure, with tablet characteristics. However, energy of compaction has not been defined consistently in the literature. Fig. 1 demonstrates typical force displacement graphs for a first compaction and a recompaction. In this instance, force transmitted to the lower punch is employed as it eliminates the inclusion of energy expended against die wall friction. Areas OAC (Fig. 1a) and ODF (Fig. 1b) are the work done on the lower punch in the first and second compaction, defined by LPW_1 and LPW_2, respectively. It is apparent that part of LPW_1 is recoverable as the compact does work on the ascending punch, area ABC (Fig. 1a). However, since the compact does not elastically recover at the same rate as the ascending top punch, area ABC is only part of the elastic energy recovery. If the assumption is made that the tablet has fully recovered elastically prior to recompression and that solely elastic deformation occurs on this recompression step, LPW_2 is a measure of the elastic energy recovered by the ascending top punch in the first compaction (de Blaey and Polderman, 1970, 1971; de Blaey et al., 1971). LPW_2 may thus be employed as a measure of the extent of elastic deformation undergone by a powder during compaction. Recognition must, however, be made of the fact that the above assumptions may not be strictly applicable in the practical sense. As seen in Fig. 1b, area ODE could be due to incomplete elastic recovery or further plastic deformation or both.

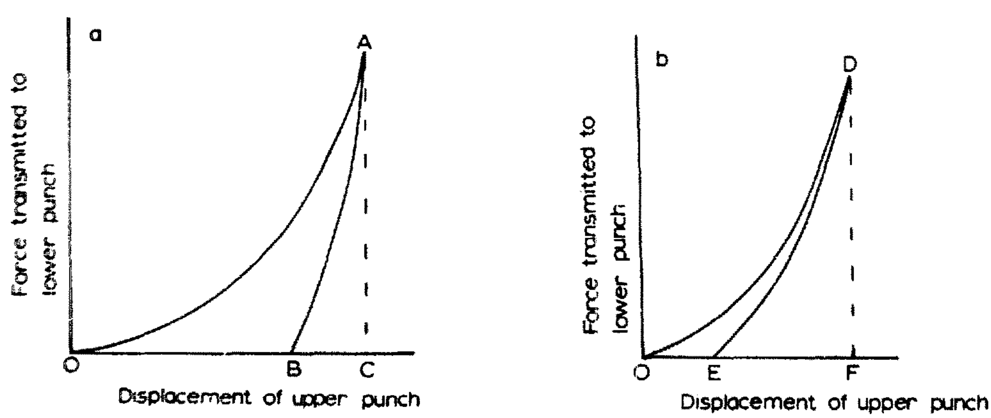


Fig. 1. Force displacement curves: (a) first compaction and (b) recompaction. (Not drawn to scale.)

Tablet strength

A useful means of evaluating energy utilization in the production of strong tablets is to plot tablet strength versus LPW_1. The mechanical strength of tablets can be described by crushing strength, hardness (Aulton, 1977), tensile strength (Newton et al., 1971) and the work required to cause tablet failure (Rees et al., 1978). Tensile strength (σ_x) given by

$$\sigma_x = \frac{2P}{\pi Dt} \tag{1}$$

where P is the load necessary to cause fracture and D and t are the diameter and thickness of the compact, respectively, is widely employed as it is independent of tablet dimensions. Work of failure has been shown to relate better than tensile strength to the ability of tablets to resist mechanical failure in a multiple diametral impact test (Rees and Rue, 1978); however, it is difficult to measure accurately and consequently rarely employed. Tablet hardness is a measure of the resistance of a tablet to local permanent deformation (Bowden and Tabor, 1964) and it is possible to have brittle tablets that are extremely hard. In this study, crushing force and tensile strength (Eqn. 1) were employed to predict the ability of a tablet to withstand mechanical handling.

Elastic deformation

Besides the questionable use of LPW_2 there are other methods available which give an indication of the extent of elastic deformation undergone by a compact during compression. Various workers (Armstrong and Haines-Nutt, 1972; Carless and Leigh, 1974; York and Bailey, 1977) employed percentage elastic recovery (E) defined by:

$$E = 100 \times \left(\frac{H - H_c}{H_c} \right) \tag{2}$$

where H_c and H are the heights of the compact under pressure and after ejection, respectively. Although this parameter does not provide a direct measurement of elastic deformation, it is useful as a measure of the disruptive effects of elastic deformation. Summers and co-workers (1976) employed an elastic modulus, which, like LPW_2, relied on the assumption that no plastic deformation occurred on recompression.

The Poisson ratio calculated from pressure cycle plots, has also been used to measure the ability of materials to deform elastically (Long, 1960; Leigh et al., 1967; Summers et al., 1976). However, Poisson ratios are, in reality, strain ratios in stressed isotropic solids and may not be identical to stress ratios in powdered materials which are likely to be anisotropic when compressed in one direction.

Plastic deformation

Numerous techniques have been employed to quantitate the extent of plastic flow that has occurred within a compact during compression. The most useful of which are residual die wall pressure (see *Radial versis axial pressure cycles*), stress relaxation and Heckel plot measurements. The most direct measurement of plastic flow is stress relaxation and has been successfully quantitated by David and Augsburger (1977). By combining one viscous and one elastic parameter in series these workers derived the relationship:

$$\ln \Delta F = \ln \Delta F_0 - kt \tag{3}$$

where ΔF is the amount of force left in the viscoelastic region, i.e. where plastic flow and fracture take place, at time t, ΔF_0 is the total magnitude of this force at $t = 0$ and k is the viscoelastic slope. k and ΔF_0 are directly related to the rate and extent of plastic flow, respectively, undergone by a compact under static strain.

The Heckel equation (Heckel, 1961) is given by:

$$\ln \left(\frac{1}{1 - D} \right) = kP + A \tag{4}$$

where D is the density of the compact relative to the true density of the material being compacted, P is the applied pressure, k is the reciprocal of the yield pressure of the material (Hersey and Rees, 1970) and A is a function of the original compact volume. The yield pressure, k, is inversely related to the ability of the material to deform plastically under pressure. Additionally, by employing Heckel plots for various sized fractions, depending on whether the plots are parallel or converge, type A or B behaviour, respectively, is defined (Hersey and Rees, 1970; 1977). It is thought that type A behaviour is exhibited by a material that deforms solely by plastic deformation, while type B behaviour occurs when brittle fracture proceeds plastic flow. It is of interest to note that no known studies exist in the literature which attempt to compare the results obtained by these techniques employed to measure plastic flow.

Radial versus axial pressure cycles

Since Long (1960) elucidated the potential usefulness of radial versus axial pressure cycle plots, they have received considerable attention in the literature. By employing pressure cycle plots, powders can be classified as having behaviour akin to a Mohr body, compacting by brittle fracture, or exhibiting a constant yield stress in shear, i.e. compacting by plastic flow (Leigh et al., 1967; Obiorah and Shotton, 1976; Obiorah, 1978). As already mentioned (see *Elastic deformation*) these pressure cycles have been employed to measure material Poisson ratios. Plots of maximum and residual die wall pressure versus axial pressure have been employed as an indication of plastic flow during compaction (Shotton and Obiorah, 1973; Obiorah and Shotton, 1976). In this study, die wall pressures are related to axial pressure rather then work of compression as they are derived from radial versus axial pressure cycles.

Materials and methods

Four model powders were chosen for this study. The direct-compression tabletting excipients Avicel PH-101 (FMC) and Dipac (Amstar) were chosen as materials that exhibit a high standard of compression, while mannitol (CSR) and paracetamol (Graesser Salicylates) were chosen as model poorly compressible materials.

Mannitol and paracetamol, being fine powders, were slowly recrystallized with seed crystals, from distilled water and analytical reagent ethanol, respectively, so that the desired particle size could be obtained. Differential thermal analysis, with a Rigaku-Denki Model M8076 TG-DTA unit, was employed to verify that polymorphic transitions had not occurred on recrystallization.

All 4 materials were sized between 125 and 180 μm with test sieves (Endecott) with the aid of an air-jet sifter (Alpine) and oven-dried at 60°C to negligible surface adsorbed moisture, as determined by Karl Fischer titration (Johnson, 1967). All powders were stored in a silica gel desiccator. No attempt was made to control laboratory humidity during tabletting.

Lead spheres (technical grade), with a diameter of 2.94 mm, and porcelain powder (125–180 μm) made by crushing porcelain china-ware, were employed as model materials compacting by plastic flow and brittle fracture, respectively.

Compaction was carried out in an instrumented single-punch Manesty F3 tablet machine fitted with half-inch diameter die and punches. Top and bottom punch pressures were monitored with the use of strain gauges (Dentronics AP20NC6EL) and die wall pressures by the use of a symmetrical cut-away die (Windheuser et al., 1963) bonded with strain gauges (Dentronics AP201C6EL) and filled in with silicone rubber (Silastic 738 RTV sealant). Strain gauge output was amplified with a 3-channel strain bridge and current-drive unit. A displacement transducer (Hewlett-Parkard 24 DCDT-250-B11) was employed to monitor top punch displacement. All 4 signals were fed into a multi-channel UV recorder (New Electronic Products, Type 1050). A rubber plug was employed to transmit axial pressures radially during calibration of the die. Punch deformation under pressure was measured with a $0-8 \times 10^{-3}$ inch dial gauge (Tesa), graduated in 10^{-4} inch divisions, and included in calculations of compact height under pressure.

LPW_1 was determined by plotting a force–displacement graph with a minimum of 6 points; the area under the graph (area OAC, Fig. 1a) was calculated by carefully cutting and weighing the graph paper.

Tablets were compacted manually to eliminate the need to incorporate the lubricant within the bulk of the tablet and to effect a recompaction. The die wall as well as the upper and lower punch surfaces were lubricated with a 1% suspension of magnesium stearate in acetone prior to each compression. Sufficient time was allowed for the acetone to evaporate before hand-filling the die. Tablet masses were calculated from true density values, as determined with a Beckman air comparison pycnometer (Table 1), to give a tablet thickness of 3 mm at zero porosity. To effect multiple compressions the fly wheel was turned in the opposite direction for each successive compression preventing premature tablet ejection. In the measurement of stress relaxation the top punch was lowered at a constant rate of approximately

TABLE 1

POWDER TRUE AND BULK DENSITIES

Material	True density (g/cm^3)	Bulk density (125–180 μm)(g/cm^3)
Avicel	1.558	0.264
Dipac	1.593	0.637
Mannitol	1.485	0.640
Paracetamol	1.285	0.629

8 mm/s until a pressure of approximately 120 MPa was obtained (David and Augsburger, 1977). The decay of applied pressure due to stress relaxation within the compact was found to be negligible after 2 min static strain.

Tablet crushing strength was determined with a constant loading rate tablet hardness tester (Schleuniger, Model 7410). The plattens were padded with filter paper and only tablets that failed in diametral crushing, where the tensile stress was the major stress (Fell and Newton, 1970) were accepted. Tablet testing was carried out 24 h after compression to allow for full elastic recovery. Tablets were stored in a silica gel desiccator at room temperature prior to testing. Tablet thickness after ejection was measured with a dial thickness gauge (Mitutoyo), graduated in 10^{-3} inch divisions.

Bulk density was measured by pouring powder down a 45° funnel into a 50 ml measuring cylinder, and determining the volume and weight per each 5 ml of added powder. The powder weight was then plotted against the measured volume. The slope of this plot is equal to the bulk density (Table 1).

Except where an obviously non-linear function exists, linear regression was employed to draw all graphs. The lines presented represent least-square estimates.

Results and discussion

Fig. 2 presents tablet tensile strength, σ_x (Eqn. 1) versus lower punch work of compression (LPW_1) plots. As discussed in the Introduction, LPW_1 is preferable to the use of UPW_1 (work done by the top punch) as it eliminates the inclusion of energy expended against die wall friction. Although net input, defined as $LPW_1 - LPW_2$, has been extensively employed (de Blaey and Polderman, 1970; 1971; de Blaey et al., 1971), it will be demonstrated in this study that LPW_2 does not accurately quantify the energy expended in elastically deforming the compact in the first compression. Paracetamol has been omitted as tablets were either too soft to register on the tablet hardness tester or capped on ejection. From Fig. 2, Dipac appears to require the least energy input to produce strong tablets. However, Avicel tablets were observed to be considerably thicker than tablets made from the other materials at the same energy of compaction. Consequently, from Eqn. 1, Avicel tablets have a comparatively low tensile strength. By plotting crushing force, as

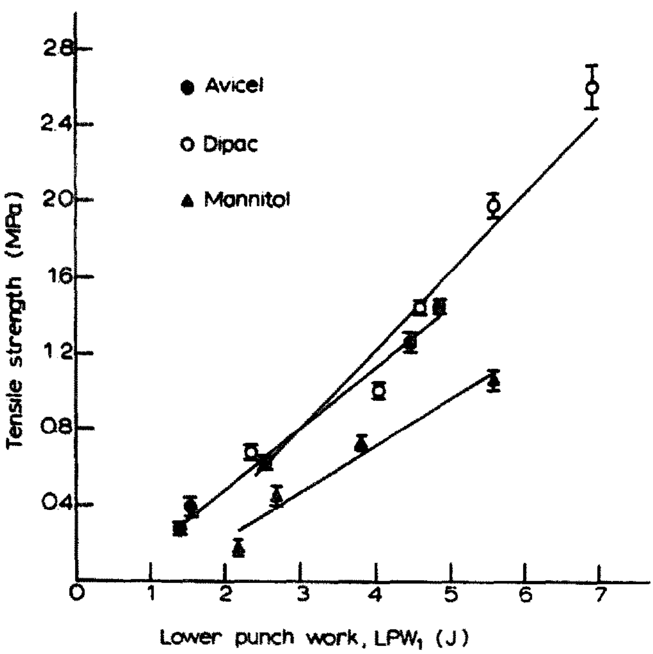


Fig. 2. Tablet tensile strength versus lower punch work. Paracetamol has been omitted as these tablets were too weak to measure crushing strength. Error bars represent 1 standard deviation (n = 5).

measured directly by the tablet hardness tester, versus LPW_1 (Fig. 3) a different picture is obtained. In this case Avicel produced the strongest tablets followed by Dipac, then mannitol.

Another way to overcome this difficulty of comparing tablets of variable thickness is to plot σ_x versus maximum axially applied pressure (Fig. 4). Since for a constant tablet mass axial pressure is a function of top punch displacement, thicker tablets are formed as a result of lower compaction pressure and consequently Avicel tablets exhibit the greatest slope in Fig. 4. Additionally, Avicel tablets absorb a relatively large amount of energy during compaction at low axial pressures (see Fig. 13) which contributes to the separation of plots in Fig. 4. This trend would be even more pronounced if crushing force were to be plotted against axial pressure. However, as indicated in the Introduction, the amount of work performed on a compact will vary according to the shape of its pressure volume curve. Consequently, materials with different compressional properties will absorb various amounts of energy for the same maximum applied axial pressure (see Fig. 13). Hence, the use of crushing force versus LPW_1, is preferable to σ_x versus axial pressure, as the former is a more reliable method of assessing relative tablet strength.

The elastic and plastic deformational behaviour of the 4 materials can be employed to elucidate the major mechanisms of compaction and account for the tablet strength results. To quantitate the extent of elastic deformation undergone by the compact, lower punch work on recompression (LPW_2) was measured (Fig. 5); Fig. 5 demonstrates that Avicel exhibits the greatest degree of elastic deformation the assumption being made that LPW_2 only measures elastic energy, with differences

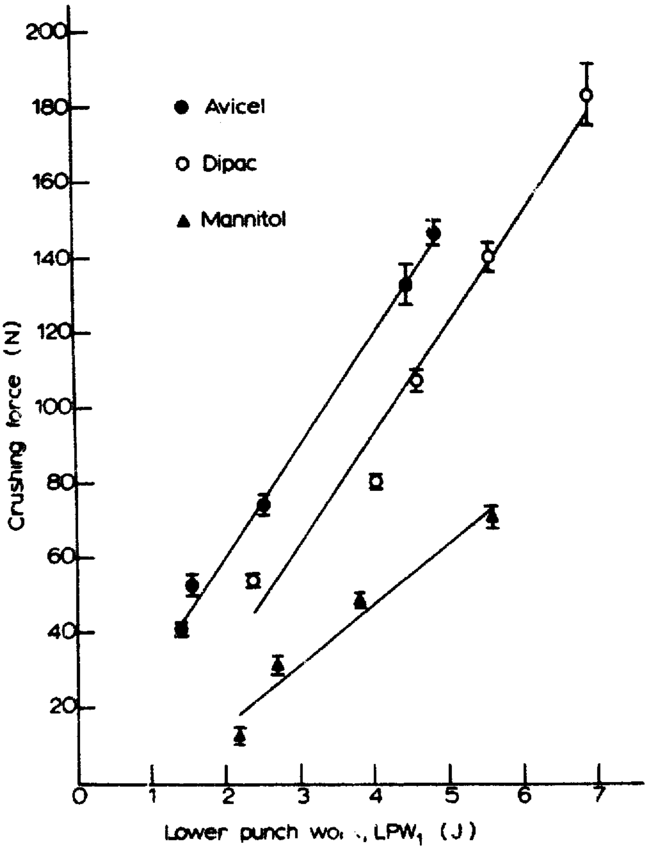


Fig. 3. Crush... force versus lower punch work. Paracetamol has been omitted as these tablets were too weak to measure crushing strength. Error bars represent 1 standard deviation (n = 5).

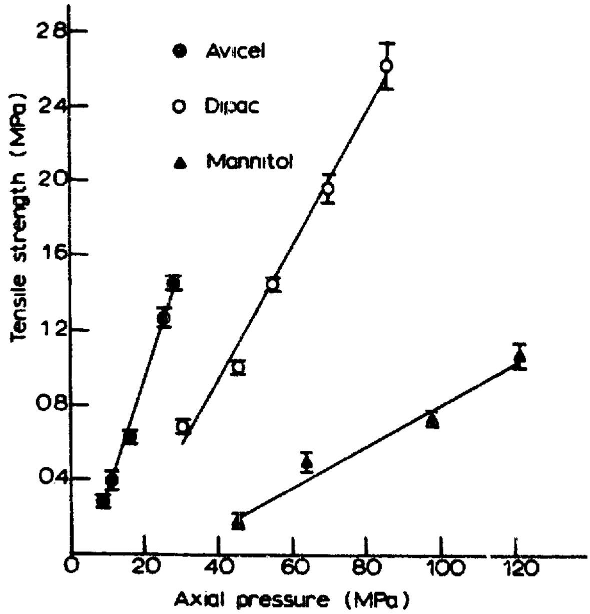


Fig. 4. Tablet tensile strength versus axial pressure. Paracetamol has been omitted as these tablets were too weak to measure crushing strength. Error bars represent 1 standard deviation (n = 5).

between the other materials only discernable at higher applied pressures. To test the assumptions made in the interpretation of LPW_2, multiple compressions were conducted on compacts of the 4 materials. The maximum descent of the top punch was kept constant and tablet ejection was only initiated after the sixth compression. Each compression was of approximately 0.8 s duration with 2 s between compression. Fig. 6 shows that for all 4 materials stress relaxation occurs as a result of multiple compression Although most of the plastic flow occurs on the first compression, the assumption that no plastic deformation occurs on successive compressions is invalid. Hence, the occurrence of stress relaxation on re-compression limits the utility of LPW_2 as a measure of elastic deformation.

In contrast, Fig. 7 shows that percentage elastic recovery, E (Eqn. 2), is a sensitive measurement of the disruptive effects of elastic deformation. In this instance, paracetamol has been included as these tablets, although extremely weak, could have their thickness monitored. It is noteworthy that E is free from dubious assumptions about compaction behaviour. The seemingly anomalous high elastic deformation

Fig. 5.

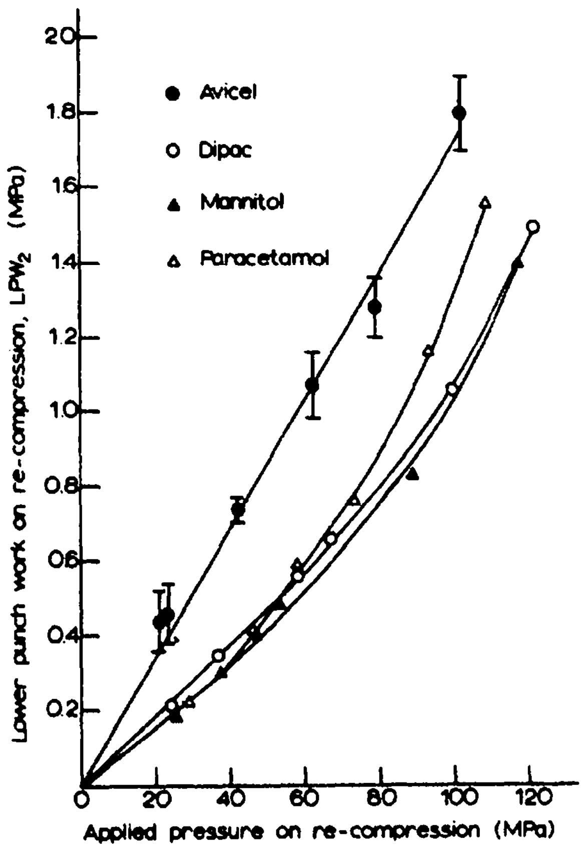


Fig. 6.

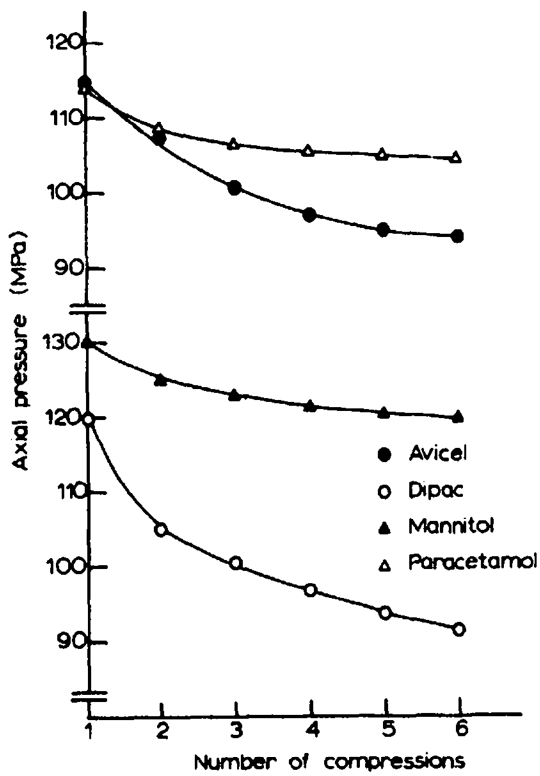


Fig. 5. Lower punch work on recompression versus applied pressure. Error bars represent 1 standard deviation (n = 5) and have been omitted from Dipac, mannitol and paracetamol profiles for clarity.

Fig. 6. Effect of multiple compressions on applied pressure for a constant maximum upper punch descent. Each point is the mean of 5 determinations.

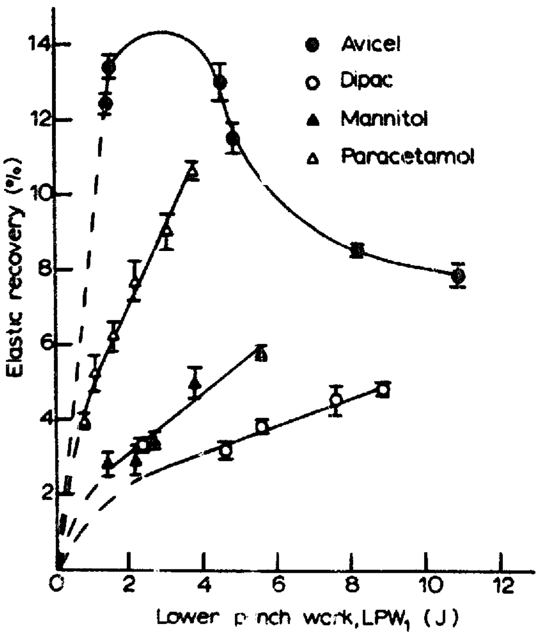


Fig. 7. Percentage elastic recovery versus lower punch work. Error bars represent 1 standard deviation (n = 5).

that occurs in Avicel has been previously reported (Aulton et al., 1974). Avicel has a hollow microfibrillar structure (Marshall and Sixsmith, 1974/5) that does not collapse easily under pressure (Sixsmith, 1977). Consequently it exhibits a high degree of elastic deformation and recovery. However, due to numerous slip-planes and dislocations (Lamberson and Raynor, 1976) it undergoes extensive plastic flow and also hydrogen bonding (Reier et al., 1966; Lamberson and Raynor, 1976) during compaction. Therefore, although elastic deformation increases to a maximum with applied pressure, large increases in interparticulate bonding, due to the formation of hydrogen bonds, limit the disruptive effects of elastic deformation. Thus elastic recovery actually decreases. For most materials, however, the disruptive effects of elastic deformation increase with energy of compaction. With the crystalline materials, paracetamol exhibits the highest elastic recovery, then mannitol and finally Dipac. The high elastic recovery coupled with a low extent of plastic flow (Fig. 11a) exhibited by paracetamol leads to its capping tendencies.

Fig. 8 presents radial versus axial pressure cycles. Significant differences between materials in the overall shape of these plots are not discernable. Additionally, the determination of point A, the yield point, is impossible due to only minor changes of gradient. Consequently, Poisson's ratios cannot be calculated accurately by this technique, for the 4 materials under investigation. However, Fig. 9 represents pressure cycles for the two model materials, lead spheres and porcelain powder. The compaction behaviour of lead (Fig. 8a) resembles a body with a constant yield stress in shear (Long, 1960) as lead compacts mainly by plastic flow. In addition, the Poisson ratio of 0.40 obtained for lead is in agreement with the literature value of

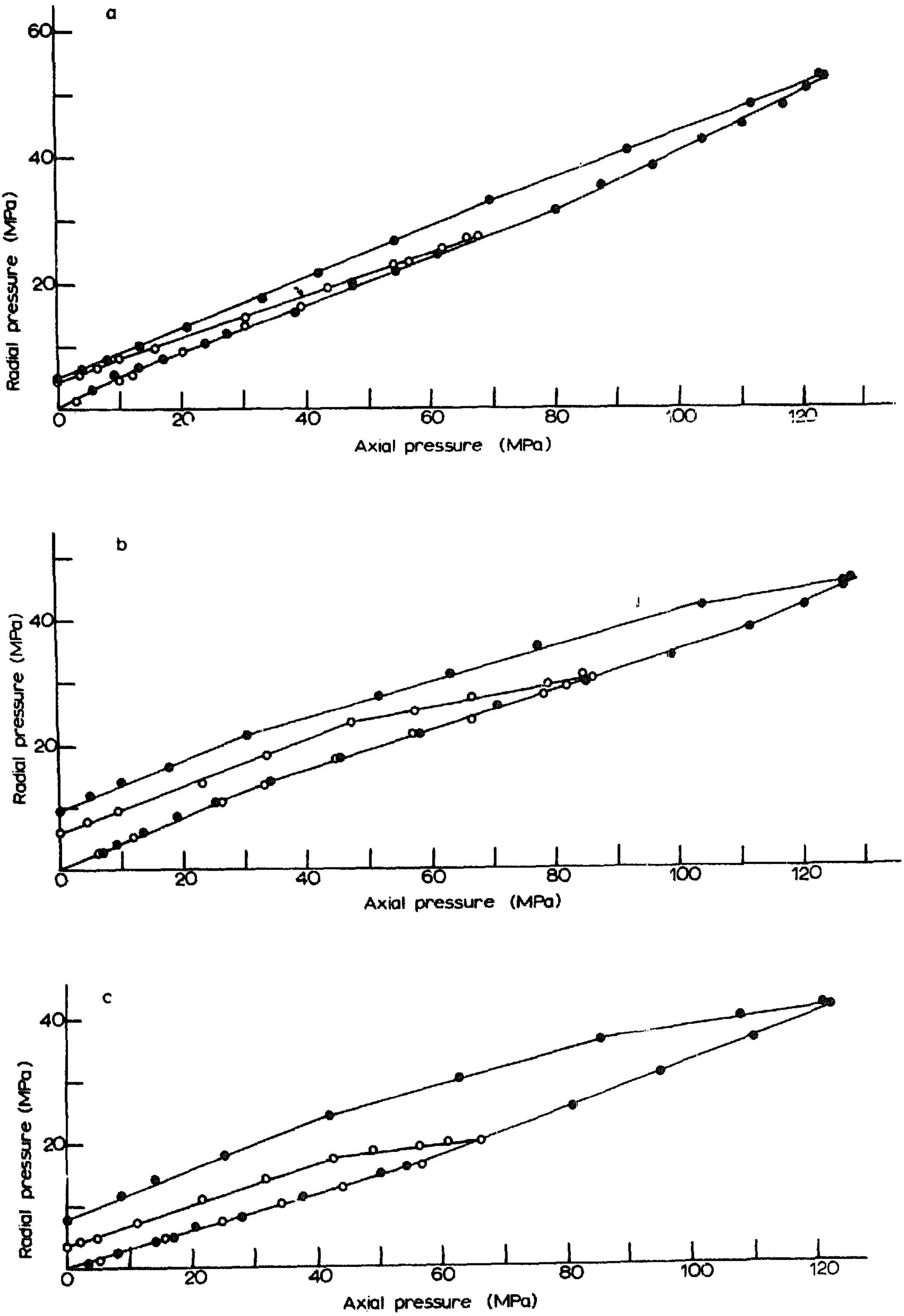


Fig. 8a–d. Continued overleaf.

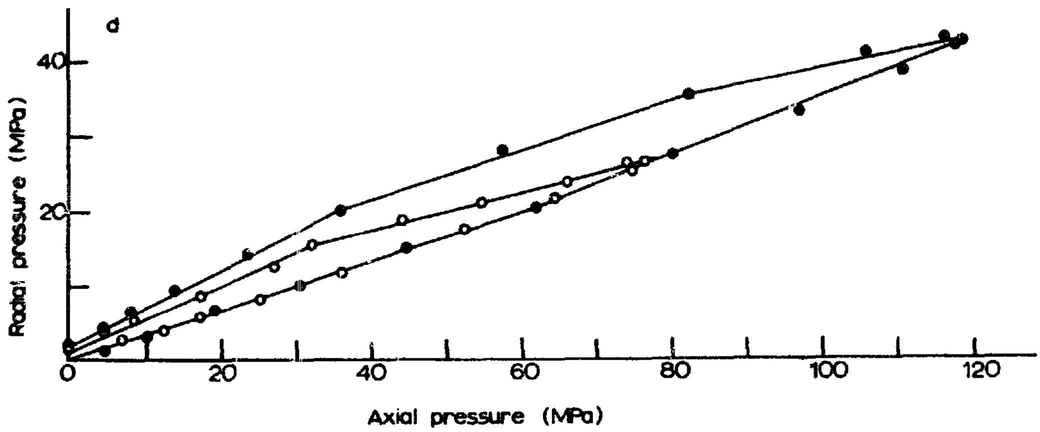


Fig. 8. Pressure cycle plots for: (a) Avicel; (b) Dipac; (c) mannitol; and (d) paracetamol. Open circles represent a pressure cycle conducted to a considerably lower maximum applied pressure compared to the cycle represented by closed circles.

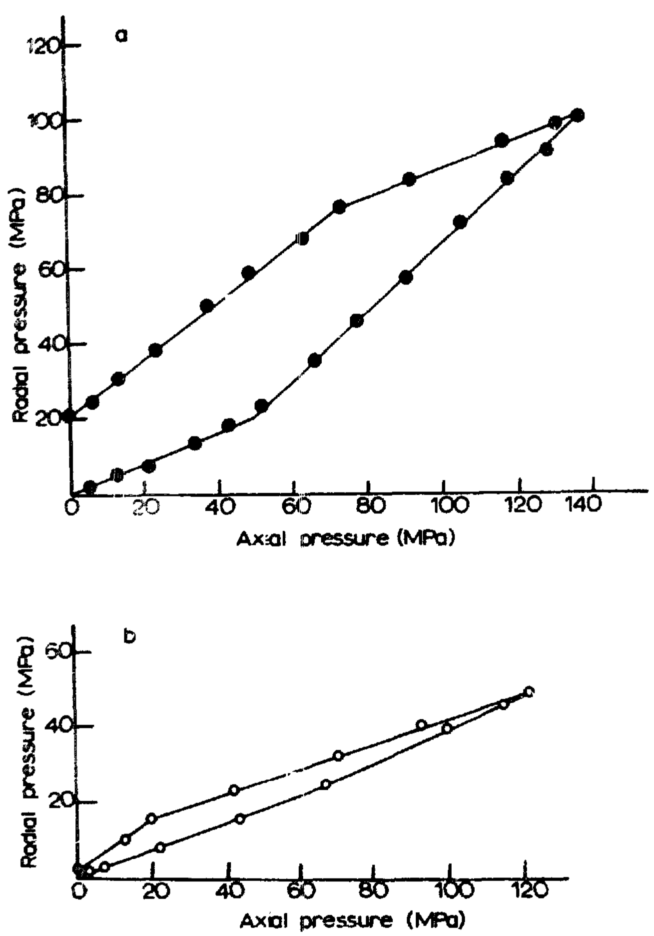


Fig. 9. Pressure cycle plots for model materials: (a) lead spheres (diameter = 2.94 mm); and (b) porcelain powder (125–180 μm).

0.40–0.45 (Bolz and Tuve, 1977). In contrast, porcelain powder (Fig. 9b) did not form a coherent compact under pressure and exhibited behaviour akin to a Mohr body (Long, 1960), due to compaction by brittle fracture. The pharmaceutical powders studied all exhibited behaviour somewhere between these two extremes, but more closely resembling the porcelain powder than the lead spheres. For these 4 powders, the overall shape of pressure cycle plots is therefore not a very useful technique in classifying compaction behaviour.

Nonetheless, two other parameters are derivable from pressure cycle data, namely maximum die wall pressure (MDWP) and residual die wall pressure (RDWP). As compacts exhibit radial stress relaxation after the release of axial pressure (Higuchi et al., 1965), maximum RDWP values have been employed. A plot of MDWP versus axial pressure (Fig. 10) illustrates the insensitivity of this technique to the differences in compaction behaviour between the materials employed in this study. MDWP contains both an elastic and a plastic component and consequently does not adequately measure plastic deformation. However, RDWP values (Fig. 11) relate mainly to the irreversible deformation undergone during compaction. In Fig. 11a tablets were subject to a single compaction at each pressure setting, while in Fig. 11b tablets were compacted at successively higher pressures with ejection only after the

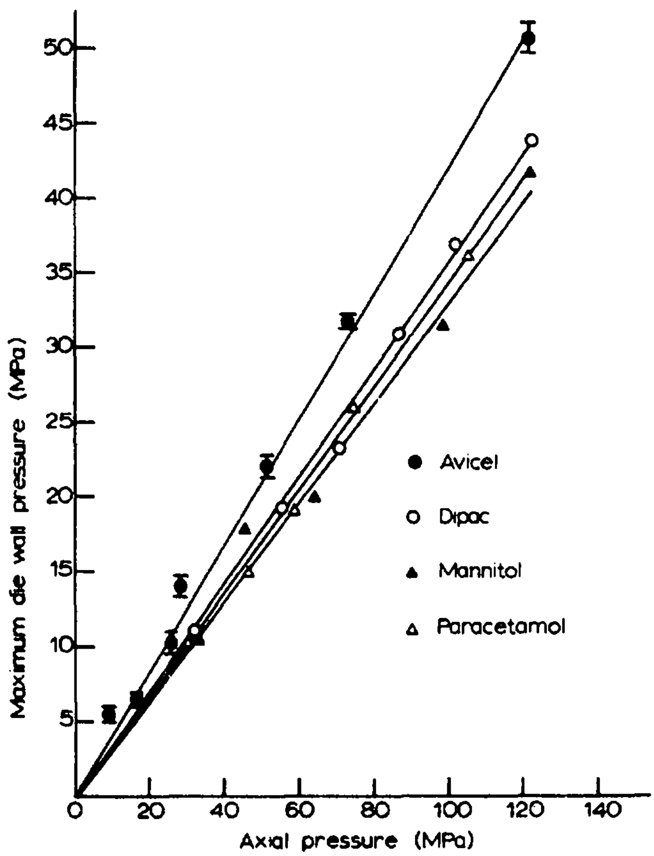
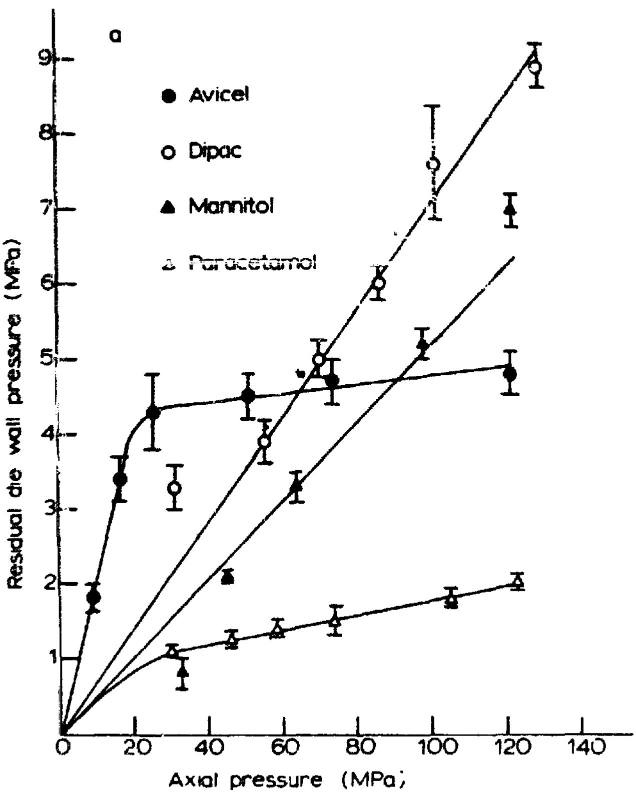


Fig. 10. Maximum die wall pressure versus axial pressure. Error bars represent 1 standard deviation (n = 5) and have been omitted for Dipac, mannitol and paracetamol for clarity.



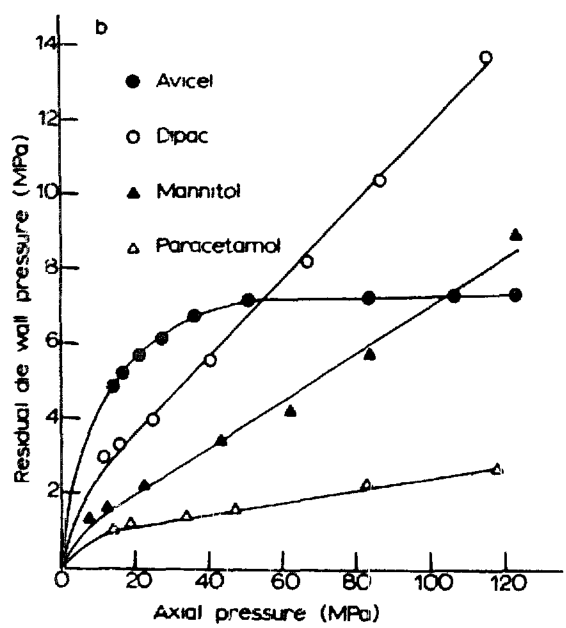


Fig. 11. Residual die wall pressure versus axial pressure: (a) single compression, multiple tablets (error bars represent 1 standard deviation, n = 5); and (b) multiple compressions, single tablets (each point is the mean of 5 determinations).

predetermined number of compressions. The same trend is apparent for both techniques. However, multiple compression plots (Fig. 11b) only show linearity after the first compression, except for Avicel, and RDWP values are higher indicating an overall greater extent of plastic flow compared to single compaction at the same pressure. The multiple compression technique has been included in this study as it has the advantage that only relatively small quantities of material are required to obtain RDWP profiles.

From the data thus far presented for Avicel it is apparent that its mechanism of compaction varies with applied pressure. It has already been postulated that due to the extensive formation of hydrogen bonds at about an energy input (LPW_1) of 3 J, elastic recovery decreases. This LPW_1 corresponds to an applied pressure of approximately 20 MPa. Fig. 11a demonstrates that at this pressure the rate of increase of plastic flow with applied pressure is inhibited. Consequently, the hydrogen bond formation exhibited by Avicel inhibits plastic flow as well as the disruptive effects of elastic deformation. In addition, Fig. 11 demonstrates that paracetamol undergoes a relatively minor degree of irreversible deformation and exhibits considerable elastic deformation (Fig. 7), which accounts for the low strength and capping observed with paracetamol compacts. The initial slope observed on the RDWP profile for paracetamol (Fig. 11a) could be due to incomplete elastic recovery after the removal of applied pressure. This contributes to the RDWP and results in slightly higher than expected values.

It is of interest to note that a plot of RDWP versus LPW_1 (Fig. 12) results in a much more confused picture. In this instance, separation of plots is not so distinct and the same rank order of the extent of plastic flow, as indicated by Fig. 11, is not obtained. This seemingly anomalous result can be explained by Fig. 13 where LPW_1 is plotted against axial pressure. It is apparent that the energy absorbed by a compact, LPW_1 (Fig. 13), is related to its ability to deform plastically (Fig. 11 and Table 2). Since LPW_1 and RDWP are both related to the extent of plastic deformation undergone by a compact during compression, for these 4 model materials, plots of RDWP versus LPW_1 (Fig. 12) are meaningless. Fig. 13 also reinforces the need to use work of compression rather than axial pressure for correlation with parameters such as crushing force and elastic recovery. Tablet crushing force is dependent on the amount of energy expended in plastic flow and bond formation. Although net input, $LPW_1 - LPW_2$, has been employed as a measure of this energy, it has been shown (Figs. 5 and 6) that there are severe interpretive difficulties with the use of LPW_2. In contrast, elastic recovery is related to the energy expended in elastic deformation of the compact and is best correlated with the gross energy input to the compact (excluding die-wall friction), LPW_1. A further noteworthy point is that for Avicel and Dipac, LPW_1 starts to level off at higher applied pressures (Fig. 13). It is expected that at higher pressures these materials become more resistant to both elastic and plastic deformation.

A further technique that measures the ability of powders to deform plastically is the use of Heckel plots (Fig. 14). In this study, relative density values, D (Eqn. 4) were measured at 'zero pressure', i.e. 24 h after tablet ejection. This eliminates the elastic component of compaction and prevents falsely low yield pressure values (Fell

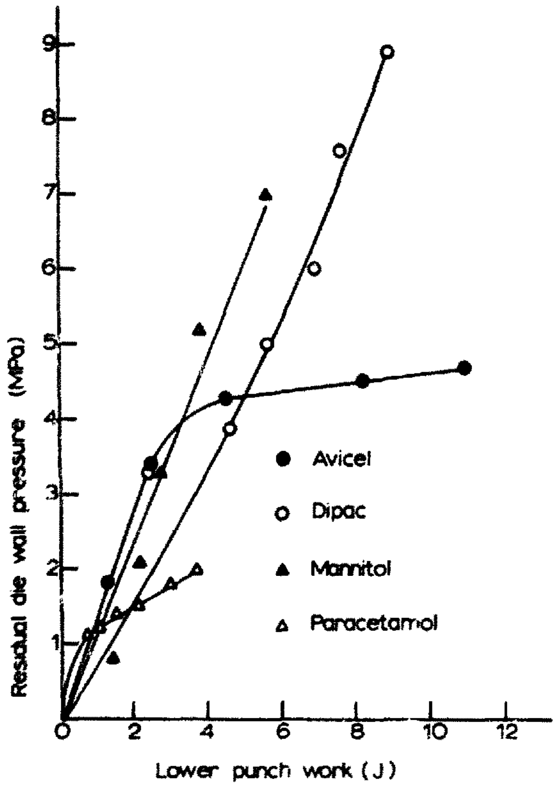


Fig. 12. Residual die wall pressure versus lower punch work. Each point is the mean of 5 determinations. (Error bars have been omitted for clarity.)

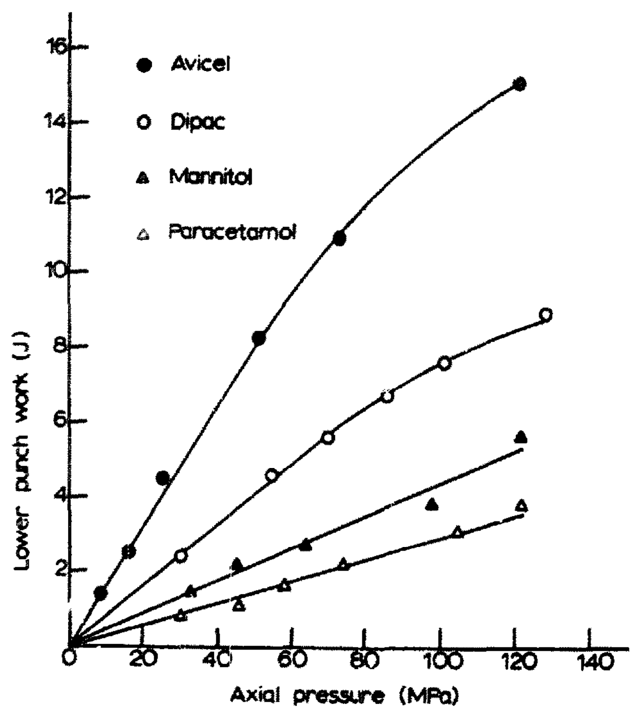
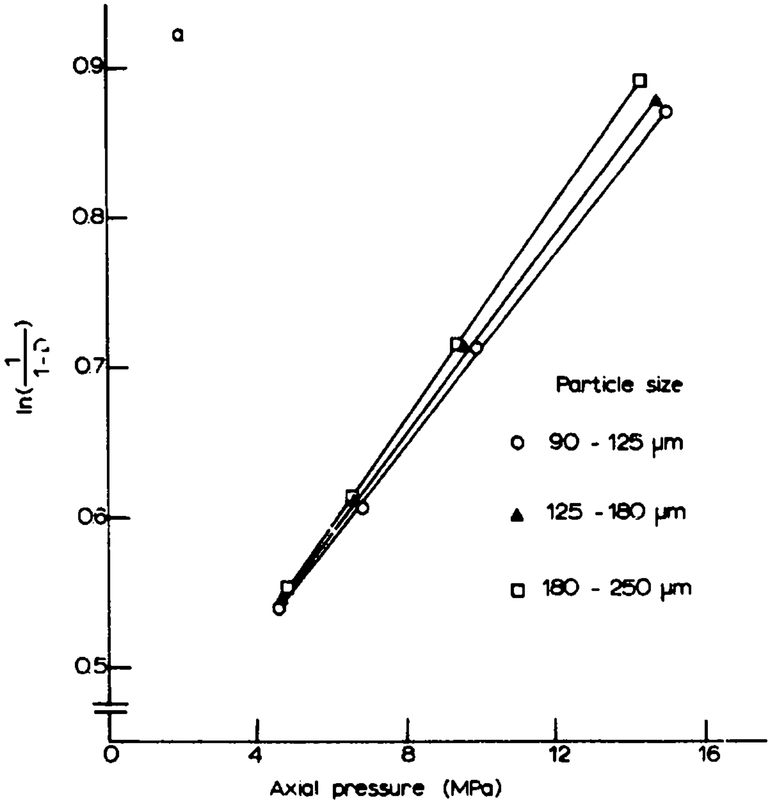
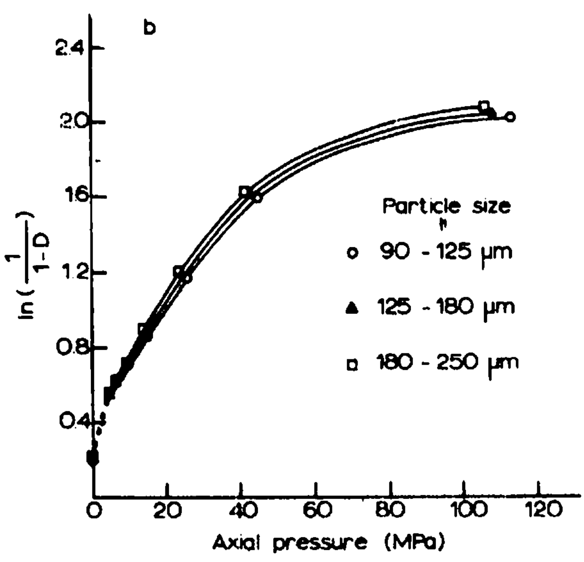


Fig. 13. Lower punch work versus axial pressure. Each point is the mean of 5 determinations.

and Newton, 1971; York, 1979). Relative density values before the application of pressure were calculated from bulk density determinations. In the case of Avicel two phases of compression are discernable. Up to 20 MPa, linear Heckel plots are obtained (Fig. 14a) and a yield pressure is calculable (Table 2). However, at higher pressures (Fig. 14b), a continuous decrease in slope is apparent. This type of





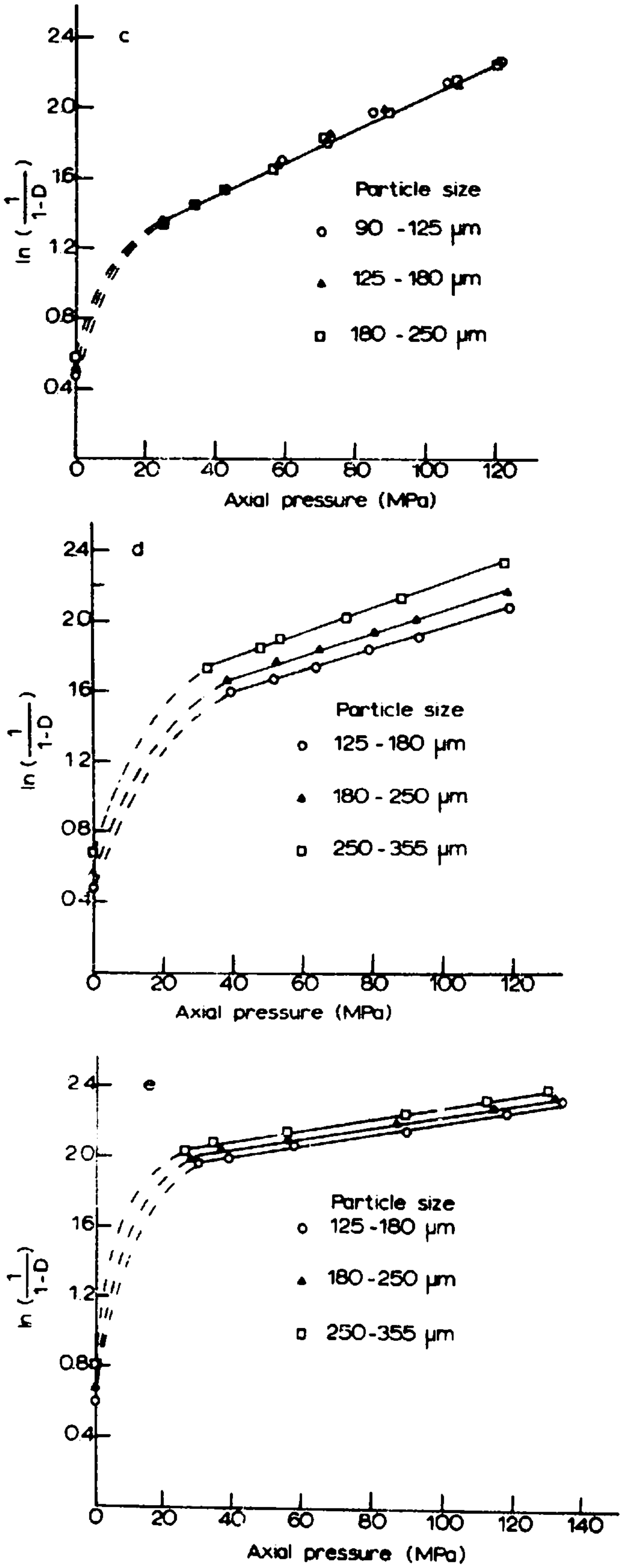


Fig. 14. Heckel plots for: (a) Avicel, 0–20 MPa; (b) Avicel, 0–120 MPa; (c) Dipac; (d) mannitol; and (e) paracetamol. Each point is the mean of 5 determinations. (Error bars lie too close to the mean to be depicted accurately.)

TABLE 2

SUMMARY OF HECKEL PLOT, STRESS-RELAXATION AND MULTIPLE COMPRESSION DATA.

Material	Type of Heckel plot	Yield pressure (Py)(MPa) (125–180 μm fraction	Viscoelastic slope (k) (S^{-1})	Total force lost (ΔF_0) (kN)	Pressure loss after 6 compressions (MPa)
Lead spheres			0.265	2.21	
Advicel	A	29.8 *	0.105	1.02	20.3
Dipac	B	107	0.0223	1.95	28.2
Mannitol	A	165	0.0118	1.03	10.2
Paracetamol	A	299	0.0110	0.92	9.0

* Calculated from the less than 20 MPA section of the Heckel plot (Fig. 14a).

continuously decreasing slope has also been observed for a microfine cellulose, Elcema (Rue and Rees, 1978; York, 1979). Fig. 14a and b are consistent with Fig. 11a where it was demonstrated that at a critical pressure of about 20 MPa the rate of increase of plastic flow with pressure decreases. The results obtained from Heckel plots (Fig. 14) are summarized in Table 2. It is apparent that the type of Heckel plot, A or B, is not useful in the prediction of compaction behaviour. However, yield pressure values (Table 2) are in agreement with RDWP data (Fig. 11).

Finally, to verify the data on plastic deformation, stress relaxation measurements were conducted. Data was plotted according to Eqn. 3 and presented in Fig. 15. The

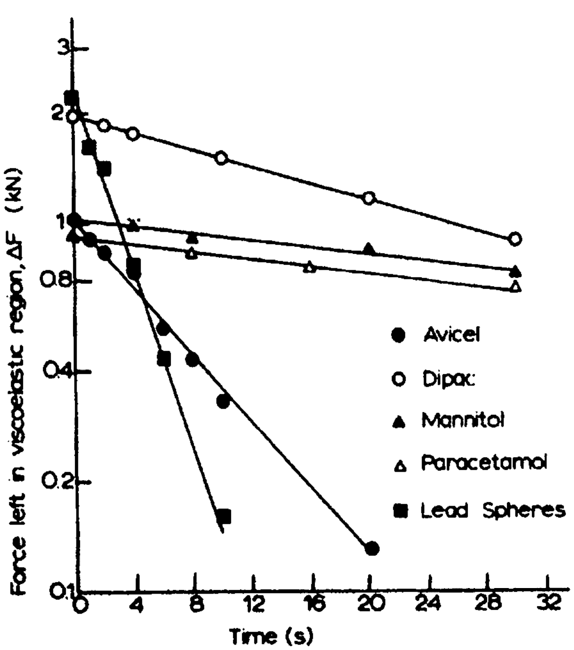


Fig. 15. Stress decay profiles. Each point is the mean of 5 determinations.

TABLE 3

SUMMARY OF THE MOST USEFUL TECHNIQUES AVAILABLE FOR THE EVALUATION OF POWDER COMPACTION BEHAVIOUR

Property to be investigated	Method
Energy utilization in the production of strong tablets	Tablet Crushing Force vs. Lower Punch Work, LPW_1
Disruptive effects of elastic deformation	Percentage Elastic Recovery, E (Eqn. 2) vs. LPW_1
Extent of plastic flow and interparticulate bonding	Residual Die Wall Pressure vs. Axial Pressure

total force lost, ΔF_0, was measured after exactly 2 min static strain. It is significant that the data fits Eqn. 3 and that as expected, lead spheres exhibit the greatest degree of plastic flow under static strain. Table 2 also presents values of the viscoelastic slope constant, k and ΔF_0. In the assessment of the ability of a material to deform plastically, it is the slope constant, k, that is important, rather than ΔF_0. As tablet machines have a very short dwell time, typically a fraction of a second, the rate of plastic flow, k, is more important than the total plastic flow in 2 min, ΔF_0. As with Heckel and RDWP data, plastic flow according to the viscoelastic slope, k, is in the rank order greatest to least, Avicel, Dipac, mannitol then paracetamol. Dipac exhibits a greater extent of plastic flow (ΔF_0), although at a slower rate (k) when compared to Avicel. This result is consistent with the multiple compression data (Fig. 6) which is also summarized in Table 2. As multiple compression with a fixed top punch descent is very similar to the application of static strain, there is close agreement between ΔF_0 and the pressure lost after 6 compressions. Furthermore, the two phases of compression of Avicel observed in Figs. 11a and 14b are consistent with its stress-relaxation data. The high initial extent of plastic flow results in a large k, but the onset of the second phase limits ΔF_0. It can also be concluded from Table 2 that stress relaxation occurs during multiple compression.

In conclusion, 3 useful and simple techniques can be employed in the evaluation of powder compaction behaviour during a tabletting operation. These have been summarized in Table 3. Advantages of these selected techniques are that they relate directly to a normal compaction cycle in a tabletting machine, assumptions about compaction behaviour are unnecessary and linearity of these plots is not essential for comparative purposes.

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