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THE INTERPRETATION OF POWDER COMPACTION DATA - A CRITICAL REVIEW

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ABSTRACT

Some of the most common methods of interpreting powder compaction data are described. These include compaction energy *versus* tablet hardness profiles, Heckel plots, stress relaxation and elastic recovery measurements and radial *versus* axial pressure cycles. By comparing and critically evaluating the techniques employed and results obtained by independent studies, it is shown that considerable confusion exists in the literature. As well, it is demonstrated that in numerous

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studies, further substantiation of the techniques employed is required. As a guide for future research, some of the most useful and possibly least ambiguous methods of interpreting compaction data are recommended.

INTRODUCTION

Tablets are the most common oral dose form. Consequently, since the mid-1950's the interpretation of compaction data in tabletting operations has received considerable attention in the pharmaceutical literature. Initially powder compaction was quantitatively described by pressure/volume relationships, and subsequently by relating compaction pressure to tablet hardness. With the current widespread use of instrumented tablet machines, which monitor axial upper and lower punch pressures, radial die wall pressures and punch displacement, an extensive number of parameters are available for evaluating compaction mechanisms and comparing the compressibility of pharmaceutical powders. The most popular methods for interpreting compaction data include the use of terms to quantitate the energy required for elastic and plastic deformation, pressure/volume relationships (such as Heckel plots), stress relaxation and elastic recovery measurements, and pressure cycle plots of radial *versus* axial pressure. However, with such a variety of methods available for

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treating compaction data, many discrepancies have arisen in the literature. Not only is there a lack of data comparing the various techniques, but there are also serious conflicts in the conclusions of researchers that are employing similar methods. The aim of this review is to describe and critically evaluate the above-mentioned techniques and to demonstrate the areas where further substantiation and correlation of derived data are required.

ENERGY UTILIZATION *versus* TABLET STRENGTH

One of the most direct means of comparing the tabletting characteristics of powders is to plot tablet crushing force *versus* mean compaction pressure [1-7] where the tablet crushing force is measured with a constant loading rate tablet strength testing apparatus [1] and mean compaction pressure (P_m) is given by

$$P_m = \frac{P_a + P_b}{2} \qquad .....(1)$$

where P_a is the maximum applied pressure by the top punch and P_b is the maximum transmitted pressure to the botton punch. Other studies have simple correlated tablet strength (in arbritrary units) with maximum applied force or pressure [8-11]. This latter approach can be criticized since it fails to account for the length of the compact, whilst mean compaction pressure is taken

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arithmetically instead of the anticipated logarithmic decay of applied force down the length of the compact. Additionally, tablet tensile strength (σ_x) given by

$$\sigma_x = \frac{2P}{\pi Dt} \qquad .....(2)$$

where P is the load necessary to cause fracture, and D and t are the diameter and thickness of the compact respectively, when applied to a diametral compression test where failure occurs in tension (defined by Rudnick and co-workers [12]), ensures that failure occurs by only one mechanism and is independent of tablet dimensions [13-18]. Subsequently, tensile strength has been widely employed in compaction studies [19-22]. Nevertheless, Hiestand and Peot [23] pointed out that due to the lack of uniform density within a powder compact, σ_x values (as determined from equation 2), are, at best, an estimate of the correct values. Rees and co-workers [24, 25] suggested that the work required to cause tablet failure (W_f) given by

$$W_f = \frac{2}{\pi Dt}\int F dx \qquad .....(3)$$

where F and x are the diametral compaction force and deformation respectively, and D and t are the diameter and thickness of the compact respectively, is a more suitable parameter than tensile strength. They demonstrated that the work of failure was a more sensitive measurement than tensile strength and related

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better to tablet 'toughness', as defined by Dieter [26]. In addition, it also related better to the ability of tablets to resist mechanical failure. Consequently, W_f could be the most useful parameter for evaluating tablet strength.

Not only is an energy measurement probably the most appropriate for tablet strength, it is also probably the best parameter for measurement of compaction. Powders with different packing densities and different plastic and elastic deformational properties, and dies with different fill capacities could result in the utilization of different amounts of energy in compaction for equally applied pressures. Consequently, the use of compression pressures could result in difficulties in comparing the compressional characteristics of different formulations. de Blaey and co-workers [27-30] used compaction energy in quantitating the different stages of compaction. The use of compaction energy also allows the results of compaction studies, obtained by different investigators to be compared and assessed [31].

Although energy of compaction is the most logical parameter for comparison with tablet strength, considerable confusion has arisen over its definition. A number of workers [32-34] have ignored die wall friction and calculated work of compaction as the total area under the upper punch force *versus* displacement

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graph, area OAC (Figure 1a). Nelson and co-workers [35] realized that the upper punch force included the force needed to overcome die wall friction, and subsequently based their calculations on the force transmitted to the lower punch. Work of compaction was defined by these workers as area ODF (Figure 1b). However, from Figure 1a, a small component of the work done by the top punch is recoverable as work done by the powder compact on the receding top punch (area ABC). Area ABC will not however represent the total recoverable work unless the tablet could carry back completely to the upper punch the work utilized in elastic deformation. This recoverable work could be very closely approximated if force-displacement curves in the decompression part of the cycle were effected over a time period of the order of the time required for complete elastic recovery. However, accurate determination of recoverable elastic deformation energy has not as yet been achieved as a three dimensional force-displacement profile would have to be charted. This is verified by the fact that elastic recovery still takes place when the tablet is completely removed from the die [36].

Since measurement of the force registered on the lower punch (Figure 1b) takes loss of energy due to particle rearrangement, interparticulate friction and particle-die wall friction into account, area ODE has also been employed for assessment of powder compaction

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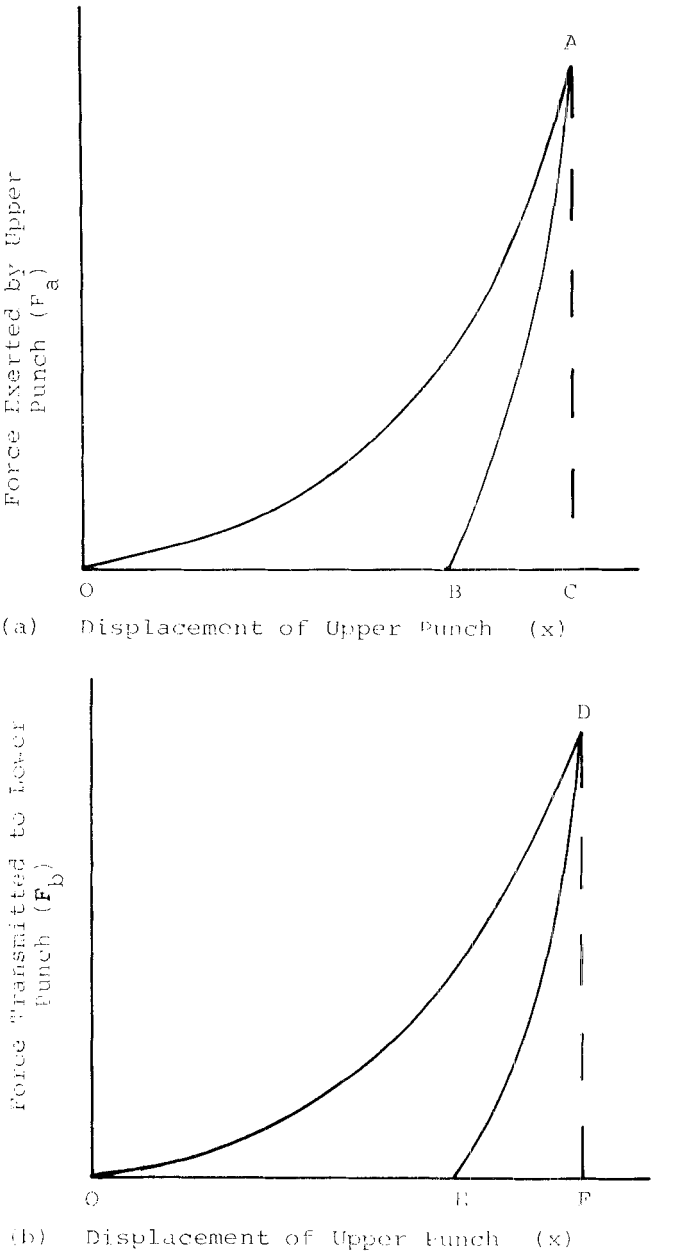

FIGURE 1

Typical force-displacement curves for a first compression employing (a) the force exerted by the top punch, and (b) the force transmitted to the lower punch.

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(37, 38). Area ODE has been termed the 'apparent net energy' input, associated with compression. The term 'apparent' has been applied because, as explained above, there is often incomplete registration of the expansion energy (area DEF) due to the difference in speed between the ascending upper punch and the expanding tablet (27, 39). Thus, the 'apparent net energy' as calculated from these force-displacement profiles is not truely indicative of any specialized characteristic of compaction.

In an attempt to define a 'true' net compaction energy input, a more detailed assignment of the utilization of compaction energy was explored. Energy in compaction is consumed by: (i) particle rearrangement; (ii) interparticle friction; (iii) particle-die wall friction; (iv) elastic deformation; (v) plastic deformation and bond formation (37, 40). By making a number of assumptions, de Blaey and co-workers (27, 30) derived and utilized the following equations. The subscripts 1 and 2 refer to a first and a second compaction respectively. Other notations are as follows:

F_a, F_b = applied force on upper punch and experienced on lower punch

dx = infinitely small displacement of the upper punch relative to the lower punch

F = die wall frictional force

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Dm, Ds	=	position of upper punch where force applied is maximum and minimum
We	=	energy of elastic deformation
Wpl	=	energy of plastic deformation
UPW	=	upper punch work
LPW	=	lower punch work

If it is assumed that (i) and (ii) above are negligible, then the applied energy (UPW_1) = (iii) + (iv) + (v) + (vi), that is: -

$$UPW_1 = \int_{Ds_1}^{Dm_1} Fa\ dx = \int_{Ds_1}^{Dm_1} F\ dx + We + Wpl \quad ...(4)$$

Rearranging Equation (4)

$$\int_{Ds_1}^{Dm_1} (Fa - F)\ dx = We + Wpl \quad ....(5)$$

$$\therefore\ LPW_1 = \int_{Ds_1}^{Dm_1} Fb\ dx = We + Wpl \quad ....(6)$$

If a second compression is performed on the tablet prior to ejection, where it is assumed that no further plastic deformation occurs, then by a similar derivation: -

$$LPW_2 = \int_{Ds_2}^{Dm_2} Fb\ dx = We \quad .....(7)$$

$$\therefore\ LPW_1 - LPW_2 = Wpl \quad .....(8)$$

($LPW_1 - LPW_2$) has been used as the 'net energy input' [22, 29, 41], while LPW_2, and even UPW_2 [34], have been used as measures of elastic deformation during compaction [22, 30]. A possible criticism of this

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arguement is that the use of net energy *versus* tablet strength could bias the results in the case of elastically deforming compacts. These compacts could exhibit a poor energy utilization in forming strong tablets, and this characteristic would go undetected due to low net energy values. In addition, the assumption that no plastic deformation occurs in recompression, especially with the modified tablet machine employed [27], is incorrect as it contradicts stress relaxation data (see STRESS RELAXATION section). The assumption is also in conflict with the observations that increased dwell time increases tablet strength [42] and that decreasing the speed of compaction increases tablet densification [43]. The suggestion then might be that, even though elastic and plastic deformation are components of the second compression, elastic deformation being much greater, may overshadow the small plastic deformation component. Hence plastic deformation in the second compression may in many instances be deleted from realistic measureable consideration.

From the above discussion, it would appear that the work required to cause tablet failure (W_f) *versus* lower punch work of compaction, area ODF (Figure 1b), is the most useful means of evaluating energy utilization in compaction. This strength/energy profile also provides a useful means of comparing the compaction properties of different materials or formulations.

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ELASTIC RECOVERY

The tendency of tablets to cap has been related to many factors including the storage of elastic energy during compaction, with subsequent elastic recovery after the removal of axial pressure [4, 44-46], and/or the inability of the material to reduce the shear stresses by localized shear flow, i.e., by plastic deformation [79]. Consequently, the quantitation of elastic recovery is a useful exercise in the elucidation of compaction mechanisms. Besides LPW_2, as discussed previously, there are many other means of evaluating elastic recovery. Huffine and Bonilla [47] measured the percentage axial recovery of the compact in the die, while Carless and Leigh [36] demonstrated that the percentage of elastic recovery of the tablet after ejection was considerably higher than that in the die. Other studies [48, 49] also measured the percentage increase in height after ejection. York and Baily [49] measured both axial and radial expansion and they suggested that a decreased ratio of axial to radial expansion was an indication of improved force distribution during compression. Summers and co-workers [50] employed a modulus of elasticity (E) defined: -

$$E = \sigma_A L/\Delta L \quad \ldots\ldots(9)$$

where σ_A is the applied axial pressure on a second compression and L and ΔL are the height and change in

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height respectively on re-compression. E is inversely related to the percentage elastic recovery measurements employed by other workers, and consequently relates indirectly to elastic recovery.

From the techniques presented, the most logical method of evaluating elastic recovery would be a plot of percentage axial recovery after ejection *versus* energy of compaction (see 'ENERGY UTILIZATION *versus* TABLET STRENGTH' for a discussion of energy of compaction). This technique makes no assumption about the extent of plastic deformation, nor does it suffer from interpretative difficulties.

HECKEL PLOTS

Kawakita and Lüdde [51] have reviewed many of the equations that describe the volume changes of a powder mass under pressure. However the equation developed by Heckel [52, 53],

$$\ln \left(\frac{1}{1-D}\right) = kP + A \quad .....(10)$$

where D is the density of the compact relative to the absolute density of the material being compacted, P is the applied pressure, k is equal to the reciprocal of 3Y, where Y is the yield strength of the material, and A is a function of the original compact volume, has been found to be the most informative. From the theoretical work of Hencky [54] and Ishlinsky [55], Hersey and Rees

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(56) showed that

$$k = \frac{1}{Py} \quad \ldots\ldots (11)$$

where Py is the mean yield pressure of the material. This yield pressure, calculated from the slope of the Heckel Plots, has been found to be in reasonable agreement to yield pressures found by use of radial versus axial pressure cycles (50).

Hersey and Rees (56, 57) also defined type A and type B compaction behaviour (Figures 2a and 2b). Type A behaviour is characteristic of a powder that has an initial particle size dependent bulk density. Densification under pressure is due initially to particle slippage or repacking, and then subsequently, to plastic deformation. The compacts however retain different degrees of porosity depending upon the initial packing arrangement in the dye. Type A curves also usually exhibit a steeper slope than Type B, hence a lower Py value. They are in general softer materials which undergo plastic deformation easier than Type B materials. A material that exhibits Type B behaviour is usually harder, has a higher yield pressure and undergoes consolidation by initial fragmentation to form a consistent packing and then plastic deformation.

In a study on the effect of compression on fatty acids and on lactose mixed with high percentages of fatty acids, a Type C behaviour was described (58).

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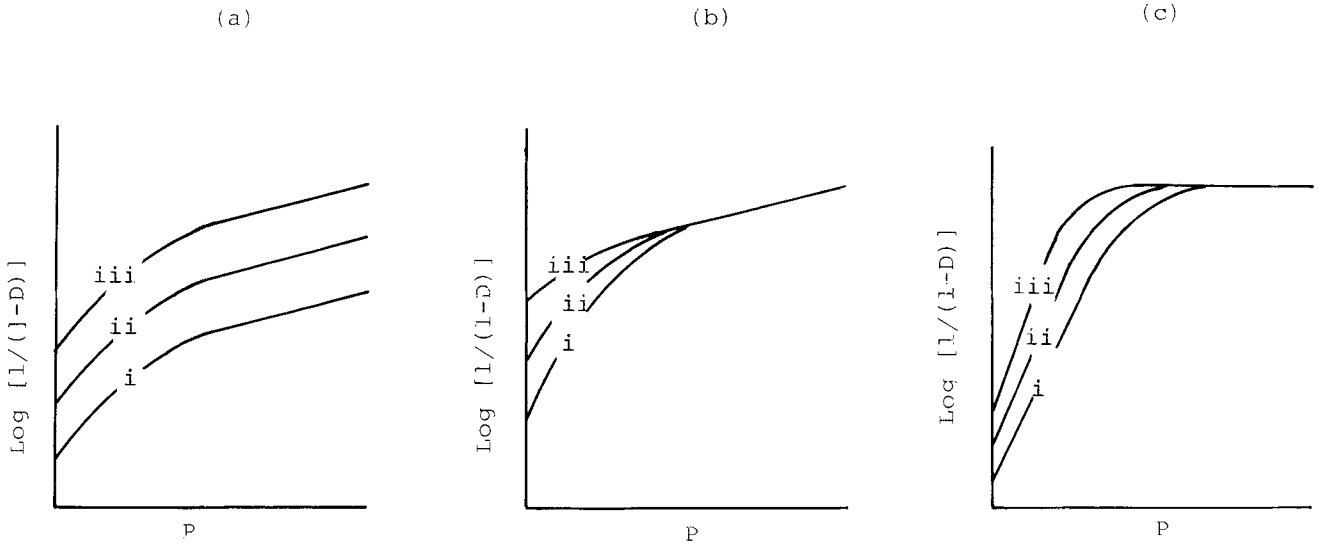

FIGURE 2

Distinction between (a) Type A, (b) Type B and (c) Type C behaviour Heckel plots. For types A and B behaviour lines i to iii refer to increasing particle size, while for type C behaviour, lines i to iii refer to increasing fatty acid concentration.

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The described Type C behaviour (Figure 2c) however fits a Type A profile except that with the lactose mixed with a high proportion of fatty acids, because of the nature of the mixture, the initial densification due to particle repacking is so slight as to be missed. The steep slopes plotted by York and Pilpel [58] indicate very low yield pressures, the yield pressure, as expected, increasing with the melting point of the fatty acids. A limiting value is perceived in the described Type C plots because the packing fraction approaches unity at much lower compressive pressures than have been experienced for other compounds.

By measuring the relative density, D under pressure, P, the Heckel equation has been utilized in numerous compaction studies [43, 59-64]. However when the results of various workers are compared the picture becomes very confusing. Rue and Rees [65] could not obtain linear Heckel plots for a granular microcrystalline cellulose, Elcema G250®, and suggested measuring areas under Heckel plots for different upper punch contact times. Recently, York [66] demonstrated that the numerical values for yield pressure (1/k) obtained by various workers for crystalline lactose were dependent on experimental conditions such as the degree and type of lubrication of punches and dies, punch diameter, compaction rate and method of measuring relative density; these problems have also been

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suggested by Hersey and co-workers [67]. It is interesting to note that the yield pressure is the pressure at which plastic deformation starts and has been quoted in most instances from 'at pressure' relative density measurements. Fell and Newton [43] and York [66] have shown that 'at pressure' density measurements include an elastic component in the powder deformation and results in falsely low yield pressure values. Heckel [53] used 'zero pressure' density but measured it in the die and consequently did not allow for full elastic recovery (see ELASTIC RECOVERY). The authors believe that the density, D, should be measured at 'zero pressure' after tablet ejection (assuming further densification does not occur during ejection) and that Heckel plots should be employed only for the comparison of different powders under identical experimental conditions.

STRESS RELAXATION

Under static compression, a decrease in the applied force occurs; this phenomenon is known as stress relaxation or creep and results from plastic deformation of the compressed material into interstitial space. Considerable fundamental research has been carried out to explain creep mechanisms [68-73], but the relevance of this phenomenon to the elucidation of powder

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compaction mechanisms has not received the attention in the pharmaceutical literature it warrants. The time-dependent increase in strength of sodium chloride tablets after ejection has been attributed to stress relaxation within the compact due to the stress exerted by a work-hardened outer shell [74, 75]. Various workers [46, 76-80] have employed stress relaxation curves to qualitatively compare the plastic flow of powdered pharmaceuticals under pressure. However, the quantitation of stress relaxation data has received scant attention. The one notable exception is the work of David and Augsburger [42]. By treating the plastic flow concept mathematically as the Maxwell model under constant strain [81, 82] which involves combining one viscous and one elastic parameter in series, David and Augsburger [42] derived the relationship,

$$\ln \Delta F = \ln \Delta F_o - kt \quad \ldots\ldots(12)$$

where ΔF is the amount of force left in the viscoelastic region, i.e., the compression stage where plastic flow and fracture takes place [83], at time, t; ΔF_o is the total magnitude of this force at t = 0 and k is the viscoelastic slope. By using the viscoelastic slope, k, these workers were able to quantitate the degree of plastic flow of direct compression tablet fillers under compaction. Materials with a higher viscoelastic slope, k, exhibited a greater degree of plastic flow under

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compression. This parameter was found to correlate well with tablet tensile strength measurements. For example, materials that displayed large viscoelastic slope constants formed strong tablets at low compaction pressures.

Hiestand and co-workers [79] looked at stress relaxation in a slightly different way. They plotted relative pressure (ratio of pressure at time t to maximum pressure applied) *versus* the logarithm of time of decay. In contrast to David and Augsburger [42], Hiestand and others [79] followed stress relaxation over a relatively long time period (up to 1000 s contrasting with 6 or 7 s). They found that there was a change in the rate of stress relaxation after a short time period (2-6 s) suggesting that some initially prominent mechanism soon becomes negligible. They also demonstrated that materials that tend to cap exhibit slower stress relaxation.

In quantitating the tendency of materials to laminate or cap, Hiestand and co-workers [79] developed a direct test for assessing what they have termed BFP (brittle fracture propensity). This is defined as:-

$$BFP = 0.5 \left[\frac{\sigma_T}{\sigma_{TO}} - 1\right] \quad .....(13)$$

and is obtained by performing a transverse compression test on a compact with and without a small hole in it, the tensile strengths being σ_{TO} and σ_T, respectively.

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The BFP factor is a quantitation of the stress relaxation by plastic deformation of the compact at the hole. If no relaxation occurs, tensile fracture might be expected to occur at exactly one-third of the tensile stress required to produce tensile failure when no hole is present [79]. In contrast, if all excess stresses at the edge of the hole were relieved, no observable differences in the tensile force would be observed. Real materials should fracture at some intermediate values, i.e., $0 < BFP < 1$, the magnitude depending on their ability to relieve localized stresses. Thus since the BFP value is an inverse measure of localized stress relief, a high value was shown to indicate a high tendency of a compact to cap or laminate.

A low BFP however does not necessary mean that capping or laminating problems will not exist. For example, paracetamol, which has a low BFP value, has however a high capping propensity. Carstensen [84] has pointed out that capping is possible both within the die or on ejection. If the latter occurs, then the volume expansion of the compact may be the prime factor. Thus, if a compound has a low BFP but expands considerably, this excessive expansion may account for the capping.

RADIAL *VERSUS* AXIAL PRESSURE CYCLES

Since 1960 when Long [85] elucidated the significance of radial *versus* axial pressure cycles, these plots have

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found widespread use in the evaluation of compaction data [45, 50, 86-98]. For ideal *non-porous* plugs, Long [85] described two types of behaviour (Figure 3). A material with a constant yield stress in shear, exhibits slopes OA = BC = ν (the Poisson ratio, defined as the transverse expansion per unit dimension of a solid of uniform cross section to its contraction per unit length when subjected to a uniaxial compression stress (during elastic deformation)), slopes AB = CD = 1, where point A is the yield point or elastic limit. The other case is for a material that behaves like a Mohr body where the yield stress in shear is a function of the normal stress on the plane of shear; here, slopes OA' = B'C' = ν, while slopes A'B' = $\frac{1 - \mu}{1 + \mu}$ and C'D' = $\frac{1 + \mu}{1 - \mu}$, where μ is a constant known as the coefficient of internal friction. These two distinct types of cycle represent different compaction mechanisms. Where the material has a constant yield in stress, this frequently represents the case of plastic deformation. Where the material behaves as a Mohr body, this represents compaction by brittle fracture.

However, it must be remembered that the above applies to a solid non-porous plug. When an analogy is made to porous tablet compacts there seems to be significant confusion and difficulty in interpretation of whether the compound exhibits constant yield stress in shear or Mohr behaviour.

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For example, Leigh and co-workers [86] found that paracetamol, which caps after compression, exhibits behaviour that resembles a Mohr body. However when granulated with polyvinylpyrrolidone, it behaves like a body with a constant yield stress and exhibits no capping. It should be noted that Leigh and co-workers [86] have relaxed the requirement of slope AB = CD = 1 for a classification of constant yield stress in shear type. They only require slope AB to equal CD.

Obiorah and Shotton [45] found that, in contrast to Leigh and co-workers [85], both paracetamol, which capped, and paracetamol mixed with gelatin hydrolysate or water, which did not cap after compression, exhibited pressure cycle plots resembling a Mohr body. Obiorah [87], in a later publication, concluded that paracetamol behaved much like an elastic body.

These inconsistencies have not been restricted solely to paracetamol. From radial pressure cycles, Obiorah and Shotton [7, 87] concluded that sodium chloride behaved like a Mohr body. However, earlier work by Leigh and co-workers [86] concluded from similar plots that sodium chloride, with an axial loading of about 2000 lb, behaves like a perfect model of a body with a constant yield stress in shear. At higher pressures, it more closely resembled a Mohr body. Recently, Carstensen and Touré [88] re-analysing the data of Leigh and co-workers [86] in terms of hysteresis

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areas, rather than slope determinations, concluded that sodium chloride behaves like a Mohr body. This result does not resolve the conflict as independent studies using microscopical observations [99] and Heckel plots [56, 57, 59] have demonstrated that sodium chloride compacts by plastic deformation rather than by brittle fracture. Additionally, Huffine and Bonilla [47] found that the surface area of sodium chloride compacts decrease on compression, i.e. no maximum surface area, corresponding to brittle fracture followed by fusion, was encountered.

It is apparent from the confusion that exists in the literature that the above method of classifying constant yield stress in shear or Mohr behaviour from radial *versus* axial pressure cycles is ill-defined.

Other studies [50, 89, 90] have made detailed analyses of pressure cycle plots. In these publications the slopes of between five and seven straight lines were used to describe these plots; each of which was given physical significance. The problem of fitting straight lines to experimentally derived pressure cycles was recently recognized by Carstensen and co-workers [88, 91] where the accuracy and validity of slope and intercept determinations were questioned. It was also pointed out [88] that the criteria employed by various workers for the determination of deformation behaviour differed.

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These criticisms help to explain the conflicting results reported above. Carstensen and Touré [88] went on to present a method to distinguish between Mohr body and constant yield in stress behaviour. By treating the compression of a powder in an analogous manner to the compression of a non-porous solid, it was shown [88] that pressure cycle hysteresis area is linearly related to maximally applied pressure if compression is by plastic deformation. However, if brittle fracture is the principle mechanism of compression, the hysteresis area is quadratically related to maximally applied pressure. As noted above this tehcnique gives results that remain in conflict with microscopic observations, Heckel plot interpretations and surface area measurements. Additionally, in a later publication, Carstensen and co-workers [92] found that various polymer formulations, expected to compress primarily be plastic flow, exhibited hysteresis areas that were quadratically related to maximally applied pressure. It was concluded that due to the changing porosity of the compact during compression, it was unrealistic to derive pressure cycle equations based on a non-porous compact.

Another parameter that can be derived from pressure cycle plots is the Poisson ratio. This, as defined previously, is an intrinsic property of the material. However it must be noted that the slope of OA (Figure 3) is in fact a stress ratio and will only be numerically

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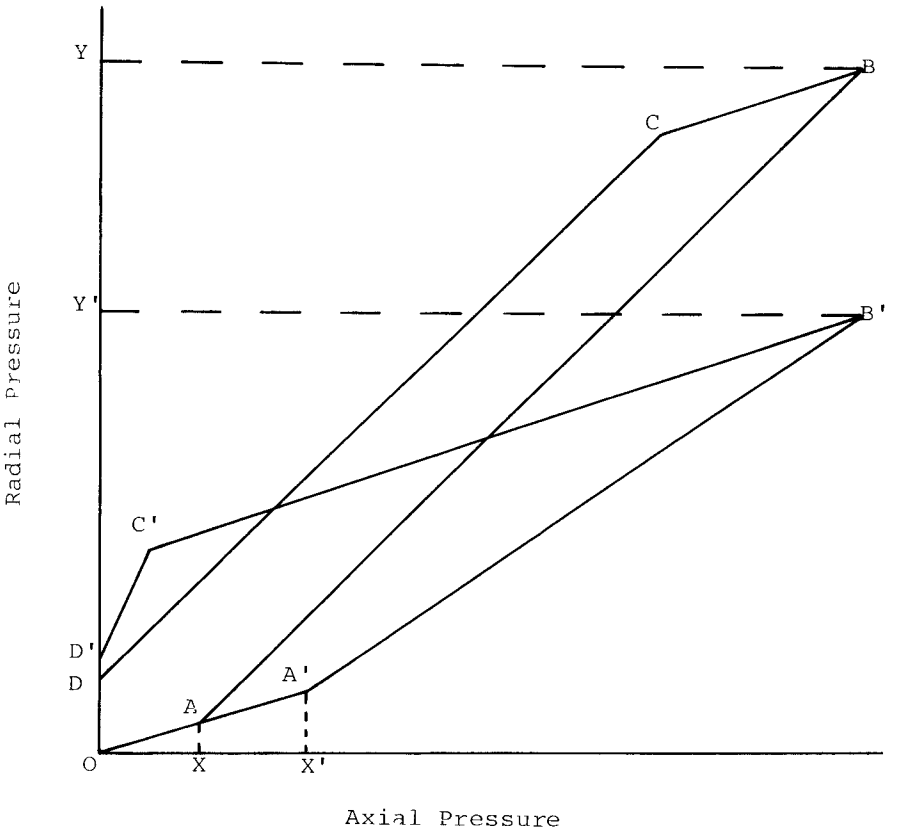

FIGURE 3

Theoretical radial versus axial pressure cycles. OABCD refers to a solid material exhibiting behaviour like a body with a constant yield stress in shear, while OA'B'C'D' refers to a compact exhibiting behaviour akin to a Mohr body. X, X' are yield pressures, while Y, Y' and D, D' are maximum and residual die wall pressures respectively.

equal to Poisson's ratio for non-porous, ideal isotropic materials behaving perfectly elastically. Powdered materials are neither non-porous, isotropic nor behave perfectly elastically.

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Long [85] and several other workers [7, 87, 90] have interpreted the slope of OA (Figure 3) for a powder compact to be the Poisson ratio for the material. Leigh and co-workers [86] assumed a proportionality between the slope OA and the Poisson ratio. Summers and co-workers [50] presented the equation,

$$n_F = 2\nu \quad .....(14)$$

where n_F is equal to the ratio of radially transmitted to axially applied force for a compact after yield. Carstensen and Touré [88] state that the slope of OA adheres to the relationship,

$$F = [\nu/(1 - \nu)]P \quad .....(15)$$

where F and P are the axially and radially transmitted pressures respectively.

Qualitative and quantitative interpretations of the slope of OA and its relationship to the compaction properties of powdered materials has also been attempted by a number of workers. As early as 1955, Nelson [93] employed percentage transmittance of punch pressure to the die wall in the evaluation of die wall lubricants. Windheuser and co-workers [94] found that the inital slope could also be related to crystal hardness. This was further developed by Ridgway and co-workers [95]. They showed that the transmittance ratio (ratio of radial pressure at the die wall to axial pressure applied by the punch) was inversely proportional to the Vickers hardness

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value of the material being compressed. However, none of these early workers admitted the existence of point A (Figure 3), the yield point. In all three studies, pressure cycle data was fitted to either smooth curves or single straight lines. Other interpretations are that the slope of OA (called the stress ratio by Carless and Leigh [36]) was inversely related to the yield pressure (value X in Figure 3) [36], and that materials that compressed well had higher initial slopes than poorly compressible materials [87].

Two other derivable parameters that have been extensively employed are maximum and residual die wall pressures[45, 50, 87, 89, 90, 96, 97], points Y and D respectively in Figure 3. These two variables have been related to the degree of plastic deformation undergone during compaction. Because compacts exhibit radial stress relaxation [79, 98] after release of axial pressure, residual die wall pressures should strictly be quoted as either maximum or equilibrium residual die wall pressures, as the case may be.

In general, residual die wall pressure (adequately defined) is probably the most useful parameter as it relates directly to the irreversible deformation undergone during compaction. Low values for residual die wall pressure and Poisson ratio would indicate the compact had recovered axially

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and contracted radially. This would induce considerable strain within the compact, because during the recovery process the tablet would be subjected to a residual pressure acting from the die wall and friction restricted peripheral movement. Under these conditions, separation or capping can occur along the stress loci [87]. This is intimately related to the brittle fracture propensity discussed previously.

Assuming then that the tablet in the die has only a negligible residual elastic energy, then a useful technique for quantifying the extent of plastic flow during a compaction cycle would be a plot of maximum (or equilibrium) residual die wall pressure *versus* applied pressure. In this instance, applied pressure is more useful than work of compaction, as it relates directly to the radial axial pressure cycle from which residual die wall pressure is calculated. Maximum die wall pressures are not generally used as these could contain a high elastic compound that does not relate to the extent of plastic flow undergone by the compact.

CONCLUSION

A number of the most common methods employed in the evaluation of powder compaction data have been reviewed. It has been shown that independent studies on the same materials have yielded conflicting results. Many

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TABLE 1

Recommendation of the most suitable techniques for the quantitation of powder compaction properties (see text for explanation of symbols).

Compaction Property to be investigated	Methods for Quantitation (In order of preference)
Qualitative description of consolidation mechanism	Type of Heckel plot (A, B or "C")
Utilization of the energy of compaction	1) Tablet Work of Failure, W_f (Eq 3) vs. Lower Punch Work, LPW_1 (Eq 6) 2) Tablet Tensile Strength, σ_x (Eq 2) vs. LPW_1 3) Crushing Force vs. Mean Applied Pressure, Pm (Eq 1)
Ability of material to deform plastically	1) ln ΔF vs. t (Eq 12) Measure the viscoelastic slope, k 2) ln $(^{l}/_{l} - D)$ vs. P (Eq 10) Employ zero-pressure density measurements, calculate the yield pressure, Py
Extent of plastic flow during compaction	Maximum residual die wall pressure vs. Applied pressure, Pa
Ability of material to deform elastically	Lower Punch Work on Re-compression, LPW_2 (Eq 7) vs. Applied Pressure on re-compression)
Extent of elastic deformation during compaction	Percentage Elastic Recovery vs. LPW_1

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formulae have also been utilized without substantiation of their validity or applicability.

In summary, Table 1 presents possible alternative methods of evaluating compaction properties, with their order of preference. These suggestions are tentative and it is hoped that future research will verify their validity and justify the inclusion of any techniques omitted.

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