

A New Method of Estimating Volume During Powder Compaction and the Work of Compaction on a Rotary Tablet Press from Measurements of Applied Vertical Force

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Abstract—The volume per unit mass of a powder bed, V' , during compaction on a rotary tablet press has been expressed as a function of pressure, P using a modification of Kawakita's equation: $V = (V'_0 - V')P' / (P + P') + V'$, where V'_0 , V' and P' constitute a set of unique values for a given powder or powder mix under specified tableting conditions. The volume, V , is determined from the machine deformation constant which is the relationship between applied vertical force and the deformation of the tablet press and the punches. An iterative method is described which allows the determination of V'_0 , V' and P' from the slope and intercept of V vs $1/(P + P')$ where all values are evaluated at peak pressure. By substituting these values into the equation, the volume of a given powder bed during compaction up to peak pressure can be accurately predicted from the pressure vs time curve. This method of estimating volume and hence punch displacement, is much simpler than an earlier analytical method which was derived from direct measurements of punch displacement under running conditions. Since volume is an explicit function of pressure, the work of compaction is also a function of pressure. Estimates of the work of compaction are in good agreement with values calculated using our previous method. Values of V'_0 , V' and P' are reported for 35 pharmaceutical materials and could be incorporated into a database library of drugs and tableting excipients. This database could then be used for the quality control of incoming raw materials (batch to batch assessment) and for the comparison of materials from alternative sources. The experimental methodology and method of calculation should, in principle, be applicable to any rotary tablet press and together with other tableting parameters (such as compression time, peak offset time, decompression time, elastic recovery and work of compaction) would provide a simple, inexpensive method for the in process validation of tablet compression.

Numerous equations have been proposed which give a relationship between the volume of a powder bed during compaction in a punch and die assembly and punch pressure (MacLeod 1983). The equation most widely used in the pharmaceutical literature is due to Heckel (1961) which can be expressed as:

$$\ln\left(\frac{V}{V - V_\infty}\right) = c_1P + \ln c_2 \quad (1)$$

where V = the powder volume under an applied pressure, P , V_∞ = the volume of the compact when its porosity is zero, and c_1 and c_2 are constants. Where a straight line relationship exists between $\ln(V/(V - V_\infty))$ and P , the reciprocal of the slope, $1/c_1$, is said to be numerically equal to the mean yield stress, P_y , (Hersey & Rees 1971; Roberts & Rowe 1987). Unfortunately, many reported estimates of P_y are questionable. The slope is rarely linear and, all too frequently, the volume between the punch faces has been incorrectly estimated because either the linear variable differential transformers (LVDTs) used to measure the distance between the punch faces have been incorrectly mounted, or the deformation in the punches and the press whilst under load has been ignored.

Kawakita & Ludde (1970/71) related the volume of a powder bed to the applied pressure. One form of Kawakita's equation was written:

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$$\left(P + \frac{1}{b}\right)(V - V_\infty) = \frac{V_0 - V_\infty}{b} \quad (2)$$

where V_0 = the initial apparent powder volume and b is a constant. Equation 2, which is similar in form to that of van der Waal's equation of state for gases, can be expressed:

$$(P + P'')(V - V_\infty) = (V_0 - V_\infty)P'' \quad (3)$$

where $1/b = P''$ and all volumes are normalized for powder mass. Rearranging to express volume as a function of pressure gives:

$$P = (V_0 - V_\infty) \cdot P'' / (V - V_\infty) - P'' \quad (4)$$

which implies a linear relationship between P and $1/(V - V_\infty)$ with a slope of $(V_0 - V_\infty)P''$ and an intercept of $-P''$. The values of V_0 and P'' are not material constants but are constants for a given powder bed. Both V_0 and P'' vary with such factors as particle size, shape and roughness which affect the initial packing.

Heckel's equation and Kawakita's equation, both model the P - V relationship from $P = 0$ up to $P = \infty$, where the corresponding volumes are V_0 and V_∞ , respectively. The volume, V_∞ , is the minimum theoretical volume of the compact per unit mass (i.e. $V = V_\infty$ when the porosity of the compact = 0).

Using a Manesty Betapress, we previously evaluated the compaction profiles of a wide range of pharmaceutical materials using a novel method of estimating punch displacement from measurements of applied vertical force and turret position only (Oates & Mitchell 1989, 1990; Dwivedi et al

1991, 1992). The compression data has now been reanalysed using Kawakita's equation and modifications thereof which take elastic deformation in the press, punches and die into account. The analysis of punch displacement is much simpler than our previous method and requires only an accurate determination of the dependence of press and punch deformation on applied vertical force under static conditions to give a machine deformation constant, K_m^{-1} .

Materials and Methods

The instrumentation of the Betapress and the methods of data collection were as described previously. Flat-faced 1/2" (1.270 cm) IPT tooling operated at a turret revolution time of 1 s was used throughout. For a given material, V_∞ in equation 3 is a constant and was calculated from the true density of the solid:

$$V_\infty = 1/\rho \quad (5)$$

The values of ρ , as determined by helium-displacement pycnometry, are given in Table 1 or were taken from Dwivedi et al (1992). Oates & Mitchell (1989) showed that the Betapress undergoes reversible elastic deformation when a vertical force is applied and that the extent of deformation is proportional to the applied force. At peak force, F_{\max} , the tablet thickness per unit mass, H_p , is given by:

$$H_p = (H_o + K_m^{-1} \cdot F_{\max})/\text{mass} \quad (6)$$

where H_o = the minimum distance between the upper and lower punch faces in an empty die and, K_m^{-1} = the machine deformation constant = 2.3×10^{-6} cm N⁻¹. The tablet thickness setting was fixed to give $H_o = 0.314$ cm (Dwivedi et al 1992). If it is assumed that die expansion is negligible when the radial force is applied, then the volume of the compact per unit mass at peak pressure, V_p , is given by:

$$V_p = H_p \cdot A_o \quad (7)$$

where A_o = the cross-sectional area of the die when the radial pressure, P_r , equals zero.

Table 1. True densities of various pharmaceutical materials^a.

Material	Density (g cm ⁻³)	Manufacturer
Calcium phosphates		
A-Tab	2.774	Rhone-Poulenc
Anhydrous Emcompress	2.780	Edward Mendell
CalStar	2.316	FMC Corporation
Di-Tab	2.330	Rhone-Poulenc
Tri-Tab	2.883	Rhone-Poulenc
Lactose		
Anhydrous	1.564	Sheffield
DCL 21	1.561	De Melkindustrie Veghel
Fast-flo	1.553	Foremost
Microcrystalline cellulose		
Avicel Large	1.555	FMC Corporation
Avicel PH101	1.556	FMC Corporation
Avicel PH105	1.556	FMC Corporation
Sugars		
Emdex	1.504	Edward Mendell
Mannitol MG	1.482	Roquette
Neosorb	1.487	Roquette
Xylitol	1.533	Roquette

^a See Dwivedi et al (1992) for true densities of other materials.

Results and Discussion

Equation 4 was evaluated at peak pressure where $P = P_{\max}$ and $V = V_p$. Fig. 1 shows typical plots of P vs $1/(V - V_\infty)$ for various solids where P_{\max} ranged from about 25 to 250 MPa. For some materials (e.g. anhydrous Emcompress, paracetamol and sucrose) the relationship is linear but for others (e.g. Avicel PH102, Emdex and STA-Rx-1500) there is distinct curvature. Table 2 gives the correlation coefficients and values of P'' determined from the intercept of the line of best fit for a wide range of materials. As expected the error in P'' increases with the increase in nonlinearity.

Equation 4 contains certain assumptions which contribute to the anomalous values shown in Table 2. Unlike gases, the pressure is not applied hydrostatically. The applied axial pressure produces an unequal pressure in the radial direction. The die undergoes radial expansion thereby introducing errors in the estimation of V_p . This was particularly noticeable with aspirin and ibuprofen where, if die expansion is assumed to be negligible, the calculated porosity goes to less than zero above a certain pressure.

In an attempt to obtain a better fit, some of the terms in equation 3 were modified to give an equation which takes radial expansion of the die into account. At peak pressure the term $V - V_\infty$ was written:

$$\begin{aligned} V - V_\infty &= V_p - V_\infty = (H_p - V_\infty/A(P_r)) A_o \\ &= (H_p - H_\infty(P_r)) A_o \end{aligned} \quad (8)$$

where A = the true cross-sectional area of the die as a function of the radial pressure, P_r , and H_∞ = the minimum theoretical compact thickness per unit mass in the limit where P goes to infinity and the porosity of the compact = 0.

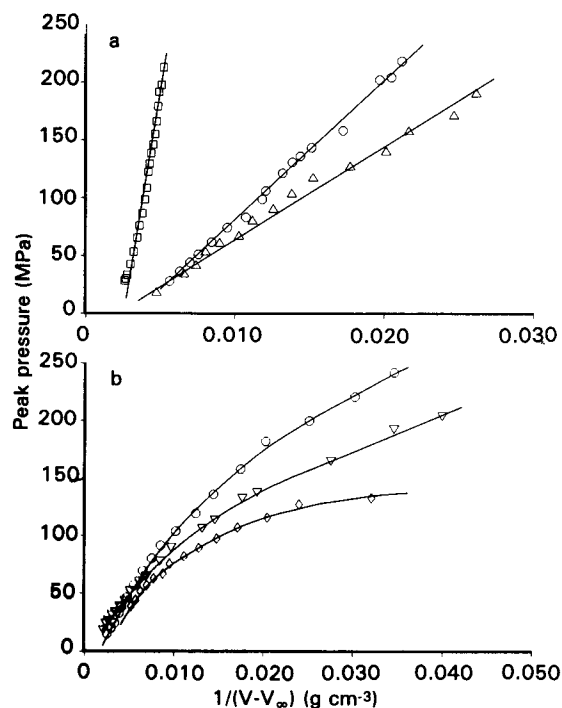


FIG. 1. Plots of P_{\max} vs $1/(V - V_\infty)$ according to equation 4. a. \square Anhydrous Emcompress, \circ sucrose, \triangle paracetamol powder. b. \circ Emdex, ∇ Avicel PH102, \diamond STA-Rx-1500.

Table 2. Values of P' , V_0 , and V_∞ for equation 4.

Material	P' (MPa)	V_0 ($\text{cm}^3 \text{g}^{-1}$)	V_∞ ($\text{cm}^3 \text{g}^{-1}$)	r^2
Aspirin	a	a	0.7172	
Calcium phosphates				
A-Tab	175 (9)	0.61 (0.01)	0.3605	0.9787
CalStar	98 (11)	0.52 (0.02)	0.4318	0.9737
Di-Tab	104 (8)	0.52 (0.02)	0.4291	0.9812
Emcompress	109 (10)	0.52 (0.02)	0.4250	0.9746
anhydrous	173 (9)	0.61 (0.02)	0.3598	0.9789
Tri-Tab	258 (12)	0.57 (0.02)	0.3468	0.9693
Ibuprofen				
Crystalline	a	a	0.8932	
DCI-63	a	a	0.8112	
Lactose				
Anhydrous	27 (7)	0.97 (0.02)	0.6394	0.9864
DCL 21	26 (7)	0.92 (0.02)	0.6406	0.9863
Fast-flo	8 (7)	1.91 (0.02)	0.6523	0.9853
Monohydrate	46 (7)	0.69 (0.01)	0.6502	0.9878
Spray-dried	26 (5)	0.85 (0.01)	0.6502	0.9944
Microcrystalline cellulose				
Avicel Large	-20 (10)	-0.13 (0.02)	0.6431	0.9768
Avicel PH101	-32 (17)	0.07 (0.04)	0.6427	0.9350
Avicel PH102	-26 (12)	0.03 (0.03)	0.6457	0.9653
Avicel PH105	-24 (13)	-0.01 (0.03)	0.6429	0.9676
Emcocel	-48 (14)	0.22 (0.04)	0.6499	0.9427
Paracetamol (crystalline)				
Fine powder	-17 (19)	-0.12 (0.06)	1.7684	0.9143
Powder	-2 (12)	-3.93 (0.04)	0.7715	0.9675
Granular	-7 (13)	-0.32 (0.04)	0.7731	0.9646
Paracetamol (direct compression)				
Compap L	140 (7)	1.19 (0.02)	0.7655	0.9885
Rhodapap DC-P3	164 (7)	1.17 (0.02)	0.7716	0.9897
Powdered cellulose				
Elcema G250	-30 (13)	0.11 (0.04)	0.6558	0.9469
Sodium chloride				
STA-Rx-1500	-34 (10)	0.21 (0.06)	0.6758	0.9077
Sugars (crystalline)				
Mannitol	10 (8)	1.72 (0.02)	0.6710	0.9841
Sucrose	41 (4)	0.61 (0.01)	0.6315	0.9964
Xylitol	32 (6)	0.61 (0.01)	0.6521	0.9932
Sugars (direct compression)				
Di-Pac	5 (9)	2.94 (0.02)	0.6479	0.9800
Emdex	-16 (15)	-0.20 (0.03)	0.6648	0.9620
Mannitol MG	13 (5)	1.09 (0.01)	0.6749	0.9932
Neosorb	-17 (11)	-0.13 (0.03)	0.6726	0.9658
Sugartab	-20 (15)	0.10 (0.03)	0.6409	0.9607

^a During compression aspirin and ibuprofen approach zero porosity and equation 4 fails.

The relationship between A and P_r is not known except at $P_r = 0$ where there is no radial expansion and $A = A_0$. Consequently H_∞ is also unknown and hence $H_p - H_\infty(P_r)$ cannot be accurately evaluated. By introducing new constants H' and V' to replace H_∞ and V_∞ , respectively, equation 8 can be approximated by:

$$V_p - V_\infty \cong V_p - V' = (H_p - H') A_0 \quad (9)$$

where $V' = H' \cdot A_0$. For materials which approach zero porosity with increasing applied pressure, then V_p goes to V' .

Equation 10 was obtained by replacing $P + P'$, $V - V_\infty$ and $V_0 - V_\infty$ in equation 3 with $P + P'$, $V - V'$ and $V_0 - V'$, respectively:

$$(P + P') (V - V') = (V_0 - V') P' \quad (10)$$

This equation has three unknowns namely P' , V' , and V_0 .

Table 3. Values of P' , V_0 , and V' for equation 11.

Material	P' (MPa)	V_0 ($\text{cm}^3 \text{g}^{-1}$)	V' ($\text{cm}^3 \text{g}^{-1}$)	r^2
Aspirin	11	0.94 (0.03)	0.700 (0.003)	0.9799
Calcium phosphates				
A-Tab	48	0.90 (0.01)	0.479 (0.003)	0.9982
CalStar	25	0.86 (0.01)	0.479 (0.002)	0.9984
Di-Tab	34	0.80 (0.01)	0.471 (0.001)	0.9988
Emcompress	28	0.83 (0.01)	0.475 (0.002)	0.9976
anhydrous	43	0.91 (0.01)	0.486 (0.003)	0.9982
Tri-Tab	50	0.82 (0.01)	0.507 (0.003)	0.9958
Ibuprofen				
Crystalline	8.4	1.49 (0.04)	0.859 (0.003)	0.9889
DCI-63	9.7	1.46 (0.02)	0.777 (0.003)	0.9961
Lactose				
Anhydrous	68	1.05 (0.01)	0.606 (0.003)	0.9982
DCL 21	49	1.09 (0.01)	0.625 (0.003)	0.9977
Fast-flo	44	1.14 (0.01)	0.623 (0.002)	0.9987
Monohydrate	68	0.95 (0.01)	0.640 (0.003)	0.9951
Spray-dried	38	1.09 (0.01)	0.643 (0.003)	0.9975
Microcrystalline cellulose				
Avicel Large	7.0	2.84 (0.02)	0.604 (0.004)	0.9993
Avicel PH101	8.5	2.39 (0.01)	0.595 (0.002)	0.9997
Avicel PH102	7.7	2.50 (0.03)	0.599 (0.009)	0.9966
Avicel PH105	7.3	2.36 (0.01)	0.610 (0.004)	0.9994
Emcocel	6.2	2.75 (0.03)	0.605 (0.003)	0.9983
Paracetamol (crystalline)				
Fine powder	60	1.12 (0.03)	0.719 (0.006)	0.9902
Powder	36	1.09 (0.02)	0.754 (0.003)	0.9961
Granular	25	1.09 (0.02)	0.761 (0.003)	0.9960
Paracetamol (direct compression)				
Compap L	35	1.37 (0.01)	0.720 (0.004)	0.9985
Rhodapap DC-P3	26	1.46 (0.01)	0.751 (0.003)	0.9987
Powdered cellulose				
Elcema G250	27	1.22 (0.01)	0.614 (0.002)	0.9988
Sodium chloride				
STA-Rx-1500	50	1.12 (0.02)	0.555 (0.003)	0.9967
Sugars (crystalline)				
Mannitol	65	1.06 (0.01)	0.634 (0.004)	0.9940
Sucrose	36	0.95 (0.01)	0.635 (0.002)	0.9986
Xylitol	29	0.96 (0.01)	0.655 (0.002)	0.9970
Sugars (direct compression)				
Di-Pac	68	1.01 (0.01)	0.602 (0.003)	0.9975
Emdex	28	1.30 (0.01)	0.624 (0.005)	0.9979
Mannitol MG	31	1.10 (0.01)	0.664 (0.003)	0.9971
Neosorb	30	1.20 (0.02)	0.639 (0.004)	0.9966
Sugartab	48	0.93 (0.03)	0.604 (0.006)	0.9860

By rearranging the terms in equation 10, V can be expressed as a function of P :

$$V = (V_0 - V') P' / (P + P') + V' \quad (11)$$

Equation 11 is a linear relationship between $1/(P + P')$ and V having a slope $(V_0 - V') P'$ and intercept V' . This equation is indeterminate since to derive the unknowns $(V_0 - V') P'$ and V' it is necessary to know P' which is also unknown. The following procedure was used to resolve these three values. A pressure P' was selected and then linear regression analysis was performed on equation 11. Successive values of P' were taken so as to determine a value which gives the best linear fit as determined by the maximum r^2 value. Once a value of P' had been selected, the constants $(V_0 - V') P'$ and V' were given by the slope and intercept, respectively. V_0 was then calculated from P' and V' .

The values of P' , V' , and V_0 derived using this method are listed in Table 3 for a wide range of pharmaceutical materials. As stated above, the compression of a powder in a

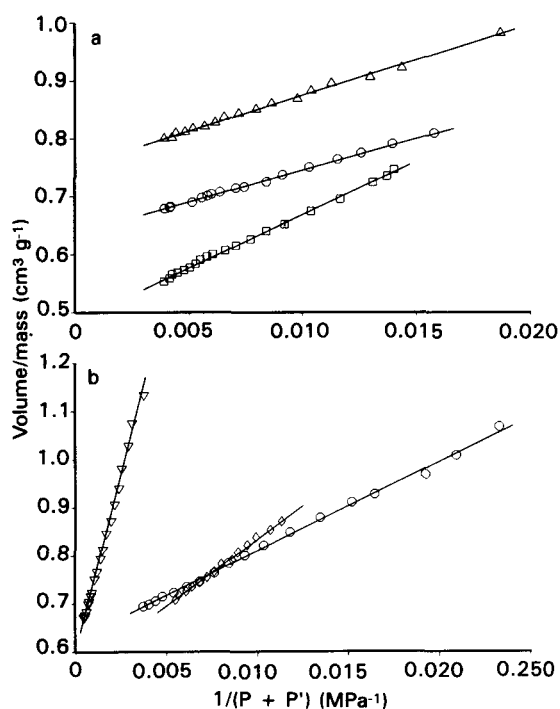


FIG. 2. Plots of V vs $1/(P+P')$ according to equation 11. a. \square Anhydrous Emcompress, \circ sucrose, Δ paracetamol powder. b. \circ Emdex, ∇ Avicel PH102, \diamond STA-Rx-1500.

die does not occur hydrostatically and the values of P' , V' and V_0 will depend on the initial packing of the die, i.e. on factors such as particle size, shape and surface roughness and, most likely, on the diameter of the die. An examination of Table 3 shows that for paracetamol, a hard material, P' decreases markedly with increase in particle size, whereas for the Avicels, P' is independent of particle size. Also of note is that ductile materials such as aspirin, ibuprofen and microcrystalline cellulose have values of $P' < 12$ MPa while harder materials such as paracetamol, the phosphates and sugars have $P' > 24$ MPa. Plots of V vs $1/(P+P')$ were linear over the entire range of data for all the materials studied. Some typical results are shown in Fig. 2.

P' , V' , and V_0 were all determined at the turret position where P_{max} occurs. To determine if these constants accurately predict the P - V relationship for compression from 25 MPa up to a specific peak pressure when substituted into equation 11, different materials were compressed to the same P_{max} . Each material should have a unique pressure vs time profile, $P(t)$. In contrast, since all materials were compressed on the same tablet press under the same conditions, they should have very similar volume vs time profiles, $V(t)$, when compressed to the same P_{max} . This is because the distance between the upper and lower punch faces is predominantly controlled by the position of the punches with respect to the compression rolls, ($D(fr)$, in equation 12 below (Oates & Mitchell 1990)). The volume $V(t)$ was calculated from $P(t)$ using equation 11 by replacing P with $P(t)$ and multiplying the right hand side of the equation by the compact mass. Fig. 3 shows $V(t)$ plots for three representative materials when compressed to $P_{max} = 150$ MPa. Although each material in Fig. 3 has a unique $P(t)$ curve, their $V(t)$ curves, as predicted,

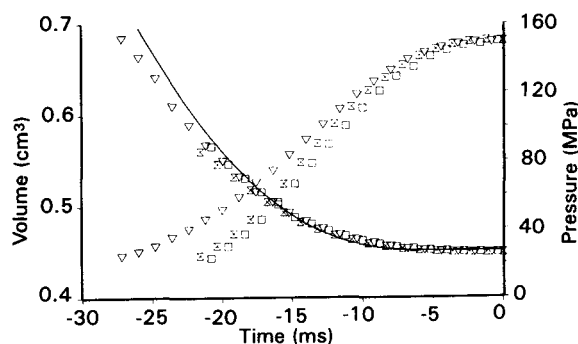


FIG. 3. Comparison of plots of P vs time and V vs time for ∇ Avicel PH102, \square Emcompress, \times spray-dried lactose. Points calculated from equation 11. Line derived using equation 12.

are very similar. This observation supports the claim that equation 11 accurately predicts the volume of a powder bed during compression up to P_{max} . The only experimental measurement required is the applied axial pressure. A significant advantage of this approach is that it is not necessary to measure punch displacement, radial pressure or even time.

The distance between the punch faces, D , and hence volume reduction during powder compaction can also be calculated using the equation of Oates & Mitchell (1990) which can be expressed as:

$$D(fr, F, t) = D(fr) + D(F) + D(t) \quad (12)$$

where $D(fr)$ = the distance between the punch faces as they come together in an empty die as a function of turret position, fr , $D(F)$ = an increase in the distance between the punch faces due to machine deformations where the press is under load = $K_m^{-1} \cdot F$, and $D(t)$ = a term which modifies the equation to account for the overestimation of punch displacement during the onset of compression when the punches are accelerating from their resting positions.

A comparison of V vs time plotted using equations 11 and 12 in Fig. 3 shows excellent agreement between the two approaches. Both methods require an accurate estimate of the machine deformation constant, K_m^{-1} . This was determined under static conditions using a series of shortened feeler gauges inserted between opposing flat-faced punches. The resultant force was measured when the pressure rolls and punches are vertically aligned at the dead centre position. The elastic deformation of the machine is directly proportional to the vertical force with a proportionality constant, K_m^{-1} and an intercept of H_0 . These constants are required to solve both equations 11 and 12 but, in addition, equation 12 requires estimates of $D(fr)$ and $D(t)$ under running conditions. It was necessary to mount an LVDT-slip ring system on the turret to evaluate these terms.

The distance $D(fr)$ was determined by filling the die cavity with a viscous oil which maintains a sufficient force to keep the punch heads pressed against their respective upper and lower pressure rolls, whilst the distance between the punch faces was measured using the LVDT (Oates & Mitchell 1990).

Displacement measurements were performed on various materials to obtain $D(fr, F, t)$, and $D(t)$ was then calculated from:

$$D(t) = D(fr, F, t) - D(fr) - D(F) \quad (13)$$

Once the terms in equation 12 have been determined, the LVDT-slip ring system can be removed and punch displacement calculated from measurements of vertical force only. Thus, equation 12 is more complicated both mathematically and experimentally than equation 11 but, nevertheless, has provided many insights into the compaction process with respect to the interaction between the press and the powder. Equation 11 has the advantages that it is much simpler and no direct measurements of punch displacement using an LVDT or other measuring device are necessary either for calibration or for in-process measurements.

Work is an integral calculated from pressure and volume. Since equation 11 allows V to be expressed as a function of P , the work of compaction per unit mass, W_c , done to the powder bed by the machine can be expressed as a function of P . The work W_{12} from a pressure P_1 to a pressure P_2 is given by:

$$W_{12} = \int_{P_1}^{P_2} PdV \quad (14)$$

Equation 11 can be differentiated to give an expression for dV in terms of P and the constants V' , V_o , and P' :

$$dV = -dP(V_o - V')P'/(P + P')^2 \quad (15)$$

Substituting equation 15 into equation 14 gives W_{12} as an integral having a single variable, P :

$$W_{12} = - (V_o - V')P' \int_{P_1}^{P_2} P/(P + P')^2 dP \quad (16)$$

Solving equation 16 gives the following expression for W_{12} :

$$W_{12} = (V_o - V')P \cdot (\ln\{[P_1 + P']/[P_2 + P']\} + P' \cdot [P_2 - P_1]/\{[P_1 + P']\{P_2 + P'\}\}) \quad (17)$$

The work per unit mass from the time when $P_1 = 25$ MPa to the time when $P_2 = P_{max}$ was calculated from equation 17 knowing V_o , V' , P' , and P_{max} . Fig. 4 shows typical plots of W_{12} vs P_{max} . Since the P - V relationship (eq 4) was evaluated for values of P_{max} between about 25 and 220 MPa, the calculation of W_c can be considered valid only for pressures within this range. Oates & Mitchell (1990) also derived an expression for W_{12} based on equation 12. The

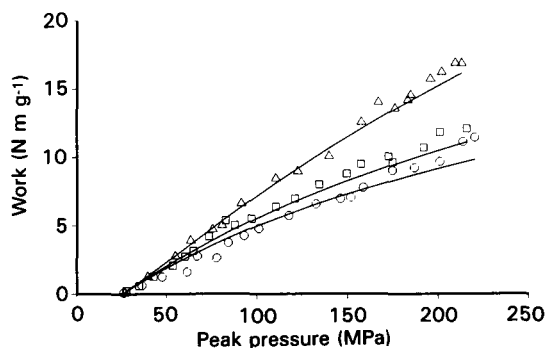


FIG. 4. Plots of work of compaction, for pressures greater than 25 MPa, vs P_{max} for Δ paracetamol fine powder, \square Di-Tab, \circ xylitol. Points calculated from equation 17. Line calculated from an equation for W_c based on equation 12 (Oates & Mitchell 1990).

Table 4. Work of compaction from 25 to 150 MPa.

Material	Work (Nm g ⁻¹)		
	1 ^a	2 ^b	3 ^c
Aspirin	3.3	4.2	6.9
Calcium phosphates			
A-Tab	11.9	12.7	17.9
CalStar	8.5	8.6	12.4
Di-Tab	8.3	8.8	12.6
Emcompress	8.2	8.9	12.9 ^d
anhydrous	11.4	12.4	17.5
Tri-Tab	8.7	8.7	12.5
Ibuprofen			
Crystalline	7.1	8.1	13.2
DCI-63	8.5	10.8	16.6
Lactose			
Anhydrous	13.0	14.2	20.7
DCL 21	13.1	13.4	19.3
Fast-flo	13.8	15.4	22.2
Monohydrate	8.9	10.0	16.6
Spray-dried	11.8	13.7	20.8 ^d
Microcrystalline cellulose			
Avicel Large	22.2	26.0	32.9
Avicel PH101	20.6	25.3	32.8
Avicel PH102	20.4	24.6	33.8 ^d
Avicel PH105	18.0	21.0	28.6
Emcocel	20.2	25.4	32.1
Paracetamol (crystalline)			
Fine powder	11.5	11.7	20.4
Powder	8.7	10.3	14.8
Granular	7.4	7.6	12.4
Paracetamol (direct compression)			
Compap L	16.4	17.9	25.3
Rhodapap DC-P3	16.1	17.1	25.0
Powdered cellulose			
Elcema G250	13.9	16.5	24.0
Sodium chloride	9.7	10.9	13.8
STA-Rx-1500	15.6	16.3	23.8
Sugars (crystalline)			
Mannitol	13.7	15.3	22.3
Sucrose	8.1	9.2	14.3
Xylitol	7.3	7.3	12.6
Sugars (direct compression)			
Di-Pac	11.6	12.2	19.7
Emdex	15.8	17.9	26.4
Mannitol MG	13.7	15.3	18.6
Neosorb	13.4	14.7	22.3
Sugartab	9.5	11.5	17.3

^a Calculated from equation 17. ^b Calculated using displacement evaluated using equation 13. ^c Calculated using displacement estimated from machine and punch head geometry. ^d Previous calculation of W_c using machine and punch head geometry (Oates & Mitchell (1990), Table 2) is in error.

values of work per unit mass in Fig. 4 and Table 4 obtained using this method are in good agreement with results derived using equation 17. Since consolidation of powders into compacts occurs over very small distances, any errors in measured or calculated punch displacement will cause large errors in estimates of W_c . Included in Table 4, are values of W_{12} estimated from force and punch displacement where displacement was calculated from machine dimensions and punch head geometry (Hoag 1990) without taking machine deformation into account. It can be seen that failure to consider machine deformation leads to serious overestimates in W_{12} .

Sinko et al (1992) used the values of W_c of Emcompress

reported by Oates & Mitchell (1989) to estimate the machine deformation constant of a Korsch Pharmapress PH106. They found reasonable agreement between our values for the W_c of some other direct compression agents and their values determined using the calibrated Korsch press.

This paper describes a method of estimating volume and hence punch displacement which is much simpler than that described previously by Oates & Mitchell (1989, 1990). The method can be applied to both Manesty and IPT punches, and apart from some means of measuring the applied vertical force, there is no requirement for expensive measuring or calibrating devices. Estimates of work of compaction between a specified lower pressure and P_{max} are in good agreement with previous values estimated using the more complicated analysis of machine deformation and punch displacement. The results of Sinko et al (1992) suggest that the new method should be applicable to the Korsch Pharmapress PH106 in addition to the Manesty Betapress. In principle the method should work for any press, but this will need to be tested.

The work of compaction and of decompression, together with other tableting parameters such as compression time, peak offset time, decompression time and elastic recovery (Dwivedi et al 1991, 1992), provide useful parameters for the in-process validation of tablet compression.

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