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Comparison of two different experimental procedures for determining compaction parameters

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Summary

Data obtained using two different experimental procedures for determining compaction parameters are compared. The different procedures used in Basle and in Kalamazoo to determine hardness and tensile strength gave similar but different absolute values. Compaction parameters calculated from these data exhibited larger differences. The results suggest that the differences in the procedures for making the tablets, e.g. decompression and ejection, may influence the parameters more than the differences of procedures for measuring the mechanical properties. The compaction parameters evaluated were the compressibility and compactibility of the Leuenberger equation and the bonding index as defined by Hiestand. These provide useful information on the deformation and bond during tableting.

Introduction

Both tensile strength and indentation hardness have been used to characterize the strength of compacts of solids. Leuenberger et al. (1981) proposed Eqn. 1 to relate the compression pressure, σ_c, and the solid fraction, ρ_r, to the development of compact strength, compactibility, as manifest by the indentation hardness, P.

$$P = P_m[1 - \exp(-\gamma_P \sigma_c \rho_r)] \quad (1)$$

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P_m is the value of P when $\rho_r = 1$, i.e. when the porosity is zero. γ_P is a proportionality constant that may reflect the compressibility, i.e. volume change per unit of energy. Also it is possible to apply the equation to characterize compact strength as manifest by the tensile strength, σ_T, as shown in equation 2.

$$\sigma_T = \sigma_{Tm}[1 - \exp(-\gamma_T \sigma_c \rho_r)] \tag{2}$$

σ_{Tm} is the tensile strength when $\rho_r \leqslant 1$ and γ_T is the compressibility parameter. Hiestand and Smith (1984) proposed that the ratio σ_T/P be called a bonding index (BI) [1]. The bonding index is believed to reflect the extent of survival during decompression of the true area established when under compression. The magnitude of the BI varies with the solid fraction of the compact produced by the compression pressure used to make the compact. Jetzer and Leuenberger (1984) report for their measurements a discontinuity of the ratio σ_T/P when fracture problems occur.

Eqns. 1 and 2 may be combined and rearranged to obtain Eqn. 3. Obviously $\sigma_T/P = \sigma_{Tm}/P_m$

$$\frac{\sigma_T}{P} = \frac{\sigma_{Tm}}{P_m}\left[1 - \frac{1 - \exp[(\gamma_P - \gamma_T)\sigma_c \rho_r]}{1 - \exp(\gamma_P \sigma_c \rho_r)}\right] \tag{3}$$

only when [2] $\gamma_P = \gamma_T$.

The observed strength of a compact varies with the stress state at yield. This is because the strength is a function of the hydrostatic stress. In other words, compacts of organic solids are Mohr bodies (Polakowski and Ripling 1966). Consequently, the shear strength under a tensile stress, σ_T, is different from the shear strength under a compressive stress as reflected in the magnitude of P. Also, the observed magnitude of σ_T and P will vary with the stress states that result from the experimental procedures used in the measurements.

The purpose of this paper is to compare data for the same lot of 3 compounds obtained by two similar but different experimental procedures. Comparison will be made of the magnitudes of the compressibility parameters, γ_P and γ_T, the compactibility parameters, P_m and σ_{Tm} and the BI.

Materials and Methods

For clarity, in this manuscript the two different experimental procedures are referred to as the Kalamazoo and Basle method. The experimental procedures used

[1] The bonding index is one of 3 tableting indices proposed. The brittle fracture index requires additional tensile measurements when a stress concentrator is present. The strain index requires information on the elastic constants. Only the procedures described by Hiestand and Smith (1984) provide these data. Therefore, only the BI is discussed in this communication.

TABLE 1

PRINCIPAL DIFFERENCES OF THE TWO EXPERIMENTAL METHODS USED IN KALAMAZOO AND BASLE

Procedure	Kalamazoo	Basle
Compression	Uniaxial	Uniaxial
Decompression	Triaxial	Uniaxial
Ejection	No	Yes
Tablet	10–15 g Square 38.1 mm on one side	0.4 g Cylindric, biplane 11 mm diameter
Indentation Hardness	Dynamic method Indenter diameter 25.4 mm	Pseudostatic method Indenter diameter 1.76 mm
Tensile strength	Transverse compression of square compacts between platens 0.4 the width of the compacts	Diametral compression of round (disc) compacts

to prepare and test the compacts are described in detail in previous papers by Hiestand and Smith (1984), Jetzer et al. (1983) and Jetzer and Leuenberger (1984). For a comparison and discussion of the results, it is important to consider the main differences between the two procedures which are mentioned in Table 1.

The results are the means of 6 determinations in Basle and at least 3 in Kalamazoo. All tests were done on the same lots of material.

Results and Discussion

The values of indentation hardness and tensile strength which vary with the solid fraction ρ_r of the compact are represented graphically on Fig. 1A–C. They are of interest, principally, because the results are very similar, even though the test procedures are very different. Identical results by different methods would be unexpected. The evaluated compactibility parameter P_m, σ_{Tm} respectively, and compressibility parameter γ computed by compression Eqns. 1 and 2 respectively, using non-linear regression analysis are given in Table 2. The parameters are characteristic of individual substance and are suggestive of its deformation behaviour (Jetzer et al., 1983). Both the parameters determined in Kalamazoo and Basle lead to the same general conclusion, even though the absolute magnitudes are different. Also, the pattern is consistent with the Brittle Fracture Index (Hiestand et al., 1977) determined additionally in Kalamazoo (see Table 3). Aspirin, which predominantly undergoes plastic deformation, exhibits a steep increase in relative density, and consequently tablet hardness with an increase in compression stress. The larger γ values for the hardness test indicate this. A solid fraction ρ_r close to unity is attained at relatively low compression stress and at the same time maximum deformation hardness is attained. Caffeine and mannitol, by way of contrast, show a

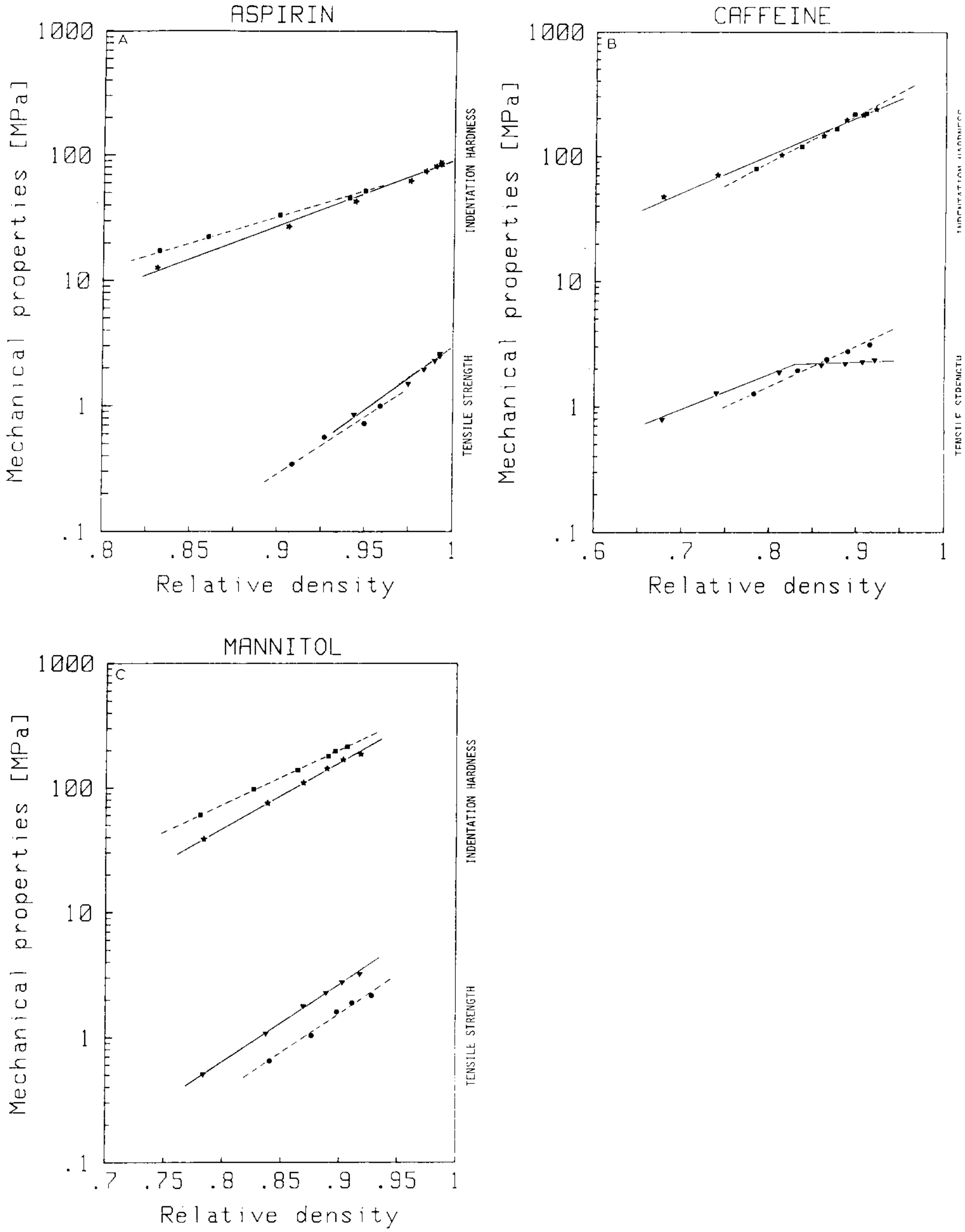

Fig. 1A–C: mechanical properties of the materials studied as a function of the relative density of the compact. ★———★, indentation hardness (Basle); ■- - - - -■, indentation hardness (Kalamazoo); ▼———▼, tensile strength (Basle); ●- - - - -●, tensile strength (Kalamazoo).

more brittle behaviour. In fact, the relative density of the compact and, consequently, tablet hardness build up gradually and extremely high compression stress is required to produce relatively non-porous tablets. (The low γ values for the

TABLE 2

COMPACTION PARAMETERS DETERMINED IN BASLE AND KALAMAZOO

C.I. = asymptotic 95% confidence interval on parameters

	Pseudostatic method Basle				Dynamic method Kalamazoo			
	P_m[MPa]	C.I.	γ_p[MPa]$^{-1}$	C.I.	P_m[MPa]	C.I.	γ_p[MPa]$^{-1}$	C.I.
Indentation hardness								
Aspirin	87.17	82.38– 91.96	0.0134	0.0110–0.0158	53.61	49.27– 57.96	0.0347	0.0279–0.0415
Caffeine	288.12	196.68–379.56	0.0058	0.0024–0.0092	494.60	−140.00–1 129.19	0.0037	−0.0024–0.0099
Mannitol	308.92	252.44–365.40	0.0032	0.0024–0.0041	273.69	224.45– 322.91	0.0091	0.0062–0.0120

	Basle				Kalamazoo			
	σ_{Tm}[MPa]	C.I.	γ_T[MPa]$^{-1}$	C.I.	σ_{Tm}[MPa]	C.I.	γ_T[MPa]$^{-1}$	C.I.
Tensile strength								
Aspirin	2.96	2.87– 3.04	0.0070	0.0066–0.0075	2.34	−1.51– 6.19	0.0063	−0.0067–0.0193
Caffeine	2.26	2.18– 2.34	0.0219	0.0189–0.0250	4.47	3.80– 5.14	0.0071	0.0054–0.0088
Mannitol	8.76	3.84–13.68	0.0016	0.0005–0.0027	10.65	9.86–11.44	0.0013	0.0012–0.0015

TABLE 3

BRITTLE FRACTURE INDEX (BFI) DETERMINED IN KALAMAZOO

	BFI	ρ_r
Aspirin	0.16	0.95
Caffeine	0.34	0.91
Mannitol	0.19	0.93

hardness test indicate this.) The P_m values of brittle materials are therefore only the best estimate obtained as an extrapolation of the data at lower relative density. Comparison of the magnitude of the single parameters shows many differences. While agreement cannot be expected due to differences in experimental procedures (e.g. compaction speed, decompression, ejection, etc.), the absence of a consistent pattern is puzzling.

Fig. 2A–C shows the graphical representation of indentation hardness P against tensile strength σ_T as a function of increasing pressure to form the compact or solid fraction. In the case of caffeine tablets made in Basle, the BI shows a discontinuity at higher pressure levels indicating fracture or capping tendency (Jetzer and Leuenberger, 1984). By contrast, the caffeine tablets produced in Kalamazoo show a linear increase of BI values, indicating that the triaxial decompression method brings about a reduction in or avoidance of fractures in the compact. This confirms results obtained by Amidon et al. (1981) with a rotary press utilizing a flexible die wall.

Comparing the tableting performance of the other two materials (see Fig. 2A and 2C), one notices that for aspirin the plots practically overlap, whereas for mannitol higher BI values were found in Kalamazoo. The ability of aspirin to easily relieve the internal stress (die wall pressure) by plastic flow probably reduces the differences of tableting performance.

In Table 4 it is interesting to compare the theoretical $\rho_r = 1$ BI values ($\sigma_{Tm}/P_m = BI_m$) calculated from the extrapolations at relative density $\rho_r = 1$. The values determined in Kalamazoo tend to be constantly higher (factor 1.13–1.39), representing a better bond survival during decompression (possibly effected by the triaxial decompression). Statistically, this difference is not significant. Because the experimental procedures are different, the differences of the values of the BI_m were expected to be larger than observed. In part this could be because the BI is a dimensionless ratio in which some effects common in both σ_T and P cancel.

The quantity $(\gamma_P - \gamma_T)$ is given in Table 5. If Eqn. 3 is rearranged after replacing $\exp(\gamma_P \sigma_c \rho_r)$ with $P_m/(P_m - P)$, which is obtained from Eqn. 1, and then one sets $nP = P_m$ so that $P_m/(P_m - P) = n/(n-1)$, one obtains Eqn. 4.

$$\left\{\frac{1}{n-1}\right\}\left\{1 - \frac{BI}{BI_m}\right\} = \exp\left[(\gamma_P - \gamma_T)\sigma_c\rho_r\right] - 1 \qquad (4)$$

It is obvious that the difference $(\gamma_P - \gamma_T)$ is a parameter that reflects the departure of BI/BI_m from unity. When $(\gamma_P - \gamma_T) < 0$, $BI/BI_m > 1$; and when $(\gamma_P - \gamma_T) > 0$,

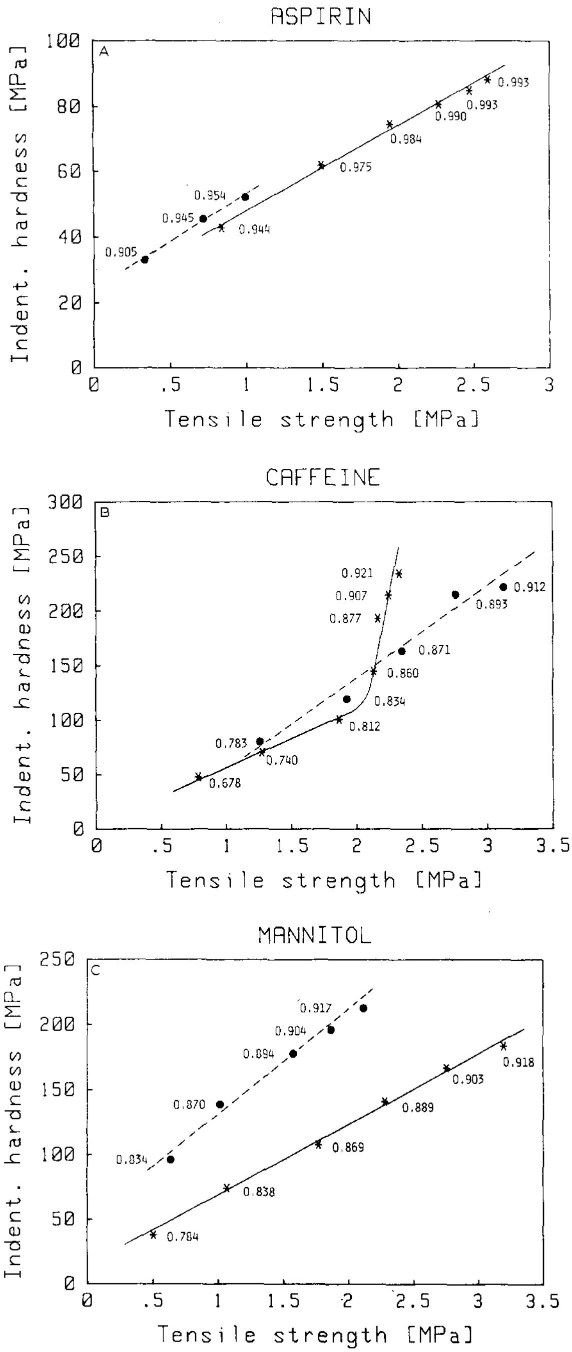

Fig. 2A–C: indentation hardness P as a function of tensile strength σ_T with increasing relative density ρ_r of the compact. ★———★, Basle; ●- - - - -●, Kalamazoo. The values shown on the diagram refer to ρ_r.

TABLE 4

a: BONDING INDEX (BI) AND RELATIVE DENSITY (ρ_r) OF THE MATERIALS STUDIED

	Basle		Kalamazoo	
	ρ_r	BI	ρ_r	BI
Aspirin	0.944	0.020	0.905	0.010
	0.975	0.024	0.945	0.016
	0.984	0.026	0.954	0.019
	0.990	0.028		
	0.993	0.029		
	0.993	0.029		
Caffeine	0.678	0.016	0.783	0.016
	0.740	0.018	0.834	0.016
	0.812	0.018	0.871	0.014
	0.860	0.015	0.893	0.013
	0.887	0.011	0.912	0.014
	0.907	0.010		
	0.921	0.010		
Mannitol	0.784	0.013	0.834	0.007
	0.838	0.014	0.870	0.007
	0.869	0.016	0.894	0.009
	0.889	0.016	0.904	0.010
	0.903	0.017	0.917	0.010
	0.918	0.017		

b: BONDING INDEX (BI_m) WHEN $\rho_r = 1$ OF THE MATERIALS STUDIED

	Bonding index (σ_{Tm}/P_m)		$BI_{Kalamazoo}/BI_{Basle}$
	Basle	Kalamazoo	
Aspirin	0.034	0.044	1.29
Caffeine	0.008	0.009	1.13
Mannitol	0.028	0.039	1.39

$BI/BI_m < 1$. Both cases are observed experimentally. (Observations such as this promt the discussion of the significance[2] of γ_P and γ_T). Reference to Tables 4 and 5 and Fig. 2A–C show that aspirin and mannitol illustrate the latter case and caffeine the former one.

Table 5 shows that the ratio of the Kalamazoo data to the Basle data is very approximately 4.5. This seems to dramatically reflect the consequences of the different procedure. Because both the tableting and test procedures were different, one cannot identify with certainty the reason for the differences. However, it is reasonable to believe that the differences in test procedures per se have less influence

[2] The exact physical significance of γ_P and γ_T has not been resolved. The authors have continued to use the previously used term 'compressibility' for lack of a better term. However, the units are volume/energy or area/force. Because the stress state is different for the indentation hardness measurement than for the tensile strength determination, one would not expect the two γ's necessarily to have identical magnitudes.

TABLE 5

($\gamma_P - \gamma_T$) VALUES OF THE MATERIALS STUDIED

f = factor which shows the ratio of the magnitude of the values ($\gamma_P - \gamma_T$) determined in Kalamazoo to those in Basle.

	Basle	Kalamazoo	f
	$(\gamma_P - \gamma_T)[MPa]^{-1}$	$(\gamma_P - \gamma_T)[MPa]^{-1}$	
Aspirin	0.0064	0.0284	4.44
Caffeine	−0.0161	−0.0034	4.74
Mannitol	0.0016	0.0078	4.88

on the data than the differences in the compression–decompression–ejection cycle. This suggestion is made because: (1) the stress states in the two tensile strength tests are similar; and (2) one would expect the dynamic indentation hardness to be larger than the pseudostatic value. The data are not consistent with these expectations.

Conclusions

The compression parameters and indices of tableting performance provide in a quantitative way useful basic information about the compression of powders. It is believed that the decompression and ejection phase predominantly affect the particle–particle bonds which influences the mechanical properties of the compact. However, qualitative conclusions about the basic behaviour of a material remains similar even when using different equipment and procedures. The results suggest that differences may occur in the absolute values observed.

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