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Effect of compression speed on the relationship between normalised solid fraction and mechanical properties of compacts

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Summary

Theoretically, when a given particulate system is compacted to the same degree of consolidation it should manifest the same mechanical properties independent of the rate of compaction. To test this, a preliminary study has investigated the effect of compression speed on the semilogarithmic relationship of indentation hardness and elastic modulus as functions of normalized solid fraction using Avicel®, a viscoelastic material, and lactose, a relatively non-viscoelastic material.

The experimental results support the thesis within the range of speeds of compression used in this study.

The effect of compression rate on the consolidation behaviour of powders as regards pressure-porosity and pressure-strength relationships have been extensively investigated (Waring et al., 1987; Rue and Rees, 1978; Sartor, 1978; York, 1979; Armstrong, 1989 and the references quoted in it). In summary, the results indicated that compacts of viscoelastic substances (because of their time-dependent characteristics) became denser and stronger with decreasing rate of compression, whereas substances that were not prone to time-dependent deformation, notably brittle substances, were relatively neutral to changes in speeds of compression.

Accepting that the degree of interparticulate bonding is responsible for the mechanical properties of a compact, it follows that for a particular substance the hardness or elasticity of its compacts at a given degree of interparticulate bonding should be a constant which is independent of the speed of compression. Consequently, the relationships of the mechanical properties as a function of the interparticle contact area should be neutral to changes in compression rates.

In a preliminary study to test this deliberation, Avicel® (visco-elastic) and lactose (brittle) were compressed at three different compression rates – 15, 25 and 40 strokes per minute and the indentation hardnesses, H, and relaxed complex elastic Young's moduli, E*, of the resulting compacts were tested. The experimental conditions and procedure have been described elsewhere (Holman

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and Leuenberger, 1989a). The relaxed complex elastic modulus was calculated using a modification of the Hertz's equation (Hertz, 1896) as described elsewhere (Holman and Leuenberger, 1988). Elastic moduli values for only Avicel are reported here because values obtained for lactose were associated with high standard errors as reported in Holman and Leuenberger (1988).

The hardness and elasticity values obtained from the measurements were plotted as functions of the normalized solid fraction (Holman and Leuenberger, 1988). The semilogarithmic relationship between hardness, H, or elastic modulus, E^*, and the normalized solid fraction, P, consists of two (if the material consolidates by plastic deformation) or three (if the material undergoes extensive fragmentation during consolidation) linear segments (Holman and Leuenberger, 1988). The different slopes may be designated as k_s, k_m, and k_a and the different y-intercepts may correspondingly be referred to as A_s, A_m and A_a (Holman, 1988). k_s or A_s is the slope or y-intercept respectively of the initial linear segment and describes the regression of lnH or lnE^* as a function of P when a continuous network of pores occur in the compact. For materials which undergo a brittle-ductile transition the semilogarithmic relationship between H or E^* and P crosses over from this initial linear region to another linear region having a slope, k_m and a y-intercept, A_m. The transition is a manifestation of the compaction behaviour crossing over from a predominantly brittle behaviour to a predominantly plastic one (Holman and Leuenberger, 1988). At high P-values, the pores become isolated and dispersed in the continuous solid medium. The slope and y-intercept in this region has been referred to as k_a and A_a, respectively.

The slopes, k, and y-intercepts, A, are given in Tables 1 and 2, respectively. The hardness, H_o, and elasticity modulus, E_o^*, of the fully-dense compact as obtained by extrapolation (Holman and Leuenberger, 1988) are also listed, in Table 3.

For lactose, a relatively non-viscoelastic material, the hardness at zero porosity, H_o, the slopes, k, and the y-intercepts, A, of the relationship lnH(P) – which are designated k_s(H), k_m(H), k_a(H) and A_s(H), A_m(H), and A_a(H) to differentiate them

TABLE 1

Effect of compression speed on the slopes, k, of the semilogarithmic plots of indentation hardness or elasticity modulus as a function of normalized solid fraction

	Slopes [MPa]		
	k_s	k_m	k_a
Hardness			
Lactose			
15 strokes/	6.124 ± 0.103 *	4.882 ± 0.05	3.445 ± 0.362
min	5.839 – 6.410 +	4.667 – 5.097	2.293 – 4.597
25 strokes/	6.491 ± 0.350	5.139 ± 0.179	3.369 ± 0.327
min	4.986 – 7.996	4.679 – 5.599	1.961 – 4.777
40 strokes/	6.232 ± 0.211	5.082 ± 0.182	2.897 ± 0.222
min	5.562 – 6.904	4.503 – 5.661	1.942 – 3.881
Avicel			
15 strokes/	3.712 ± 0.043	–	2.678 ± 0.045
min	3.610 – 3.815	–	2.106 – 3.250
25 strokes/	4.065 ± 0.139	–	2.369 ± 0.066
min	3.679 – 4.452	–	1.536 – 3.202
40 strokes/	4.362 ± 0.097	–	2.579 ± 0.093
min	4.112 – 4.612	–	2.179 – 2.979
Elasticity modulus			
Avicel			
15 strokes/	3.452 ± 0.070	–	1.927 ± 0.029
min	3.286 – 3.618	–	1.562 – 2.293
25 strokes/	3.508 ± 0.072	–	2.050 ± 0.073
min	3.307 – 3.709	–	1.738 – 2.362
40 strokes/	3.946 ± 0.086	–	2.167 ± 0.072
min	3.725 – 4.166	–	1.857 – 2.477

*, Mean ± S.D. +, 95% confidence interval. k_s, slope of the initial linear section; k_m, slope of the linear section after brittle-ductile transition; k_a, slope of the linear section in the closed pore state.

from the slopes and y-intercepts of lnE^*(P) which have been designated as k_s(E), k_m(E), k_a(E) and A_s(E), A_m(E), A_a(E) – are insensitive to changes in speeds of compression in the range of compression speeds used in this study. A similar trend was found for Avicel[R]. Except the slope, k_s, and the y-intercept, A_s, of the initial linear segment of lnH(P) and lnE^*(P) plots which show a dependency on the rate of compression (k_s increases and A_s decreases with increasing compression rate), all the parameters i.e. H_o, E_o^*, the slope, k_a and the y-intercept, A_a, derived from the plots for Avicel remain essentially constant with varying speeds of compression (see Tables 1 and 2).

A test for parallelism shows that although k_s(H) at a compression rate of 25 strokes/min

TABLE 2

Effect of speed of compression on the y-intercept, A, of the semilogarithmic plot of indentation hardness and elasticity modulus as a function of the normalized solid fraction

	y-intercept		
	$\ln A_s$	$\ln A_m$	$\ln A_a$
Hardness			
Lactose			
15 strokes/min.	0.717 ± 0.058 *	1.489 ± 0.033	2.523 ± 0.276
	0.557 − 0.878 +	1.348 − 1.630	1.644 − 3.402
25 strokes/min.	0.530 ± 0.190	1.299 ± 0.117	2.576 ± 0.250
	−0.289 − 1.348	0.999 − 1.599	1.500 − 3.653
40 strokes/min.	0.754 ± 0.116	1.426 ± 0.120	3.019 ± 0.172
	0.386 − 1.121	1.043 − 1.808	2.277 − 3.760
Avicel			
15 strokes/min	1.114 ± 0.032	–	1.997 ± 0.040
	1.038 − 1.190	–	1.493 − 2.501
25 strokes/min	0.837 ± 0.103	–	2.267 ± 0.057
	0.552 − 1.123	–	1.540 − 2.994
40 strokes/min	0.679 ± 0.067	–	2.117 ± 0.081
	0.506 − 0.852		1.770 − 2.464
Elasticity modulus			
Avicel			
15 strokes/min	−2.466 ± 0.052	–	−1.175 ± 0.025
	−2.589 − −2.344	–	−1.497 ± −0.853
25 strokes/min	−2.517 ± 0.053	–	−1.309 ± 0.062
	−2.665 ± −2.369	–	−1.577 − −1.040
40 strokes/min	−2.816 ± −0.059	–	−1.344 ± −0.063
	−2.969 − −2.663	–	−1.613 − −1.075

*, Mean ± S.D.; +, 95% confidence interval. Subscripts are the same as in Table 1.

TABLE 3

The hardness, H_o, and elasticity modulus, E_o^*, at zero porosity at various compression rates

	$(H_o$-S.D.)–H_o–(H_o + S.D.) [MPa]	95% Confidence interval
Hardness		
Lactose		
15 strokes/min	358.248 − 390.747 − 426.194	296.402 − 515.122
25 strokes/min	353.445 − 381.966 − 412.787	273.536 − 533.377
40 strokes/min	352.533 − 370.695 − 389.793	298.638 − 460.139
Avicel		
15 strokes/min	106.677 − 107.263 − 107.852	100.048 − 114.997
25 strokes/min	102.211 − 103.074 − 103.945	92.623 − 114.704
40 strokes/min	108.095 − 109.465 − 110.853	103.691 − 115.561
Elasticity modulus	(E_o^*–S.D.)–E_o^*–(E_o^* + S.D.)	95% Confidence interval
Avicel	[GPa]	
15 strokes/min	2.115 − 2.123 − 2.130	2.030 − 2.219
25 strokes/min	2.077 − 2.098 − 2.120	2.009 − 2.192
40 strokes/min	2.254 − 2.277 − 2.299	2.183 − 2.374

does not differ from that of the 40 strokes/min, the $k_s(H)$ value at the compression speed of 15 strokes/min differs from the $k_s(H)$ values at both speeds of 25 and 40 strokes/min. Considering the elasticity results, the $k_s(E)$ values at speeds of 15 and 25 strokes/min are not significantly different from each other but both values differ from the $k_s(E)$ value at 40 strokes/min.

The A_s and k_s values like the constants of any linear regression behave like typically a flip-flop system. As such, the A_s values are likely to show the same significant or insignificant differences as the k_s values. They were thus not analysed.

The apparent non-uniformity of the statistical significance tests on the effect of the compression speed on the k_s values is attributable to the minimal difference between speeds of compression used which was limited by the range of speeds over which the single-punch tableting machine, EK-0, could be run. Nevertheless, it is clear from the statistical tests that when the difference in compression speeds is big enough (e.g. 15 and 40 strokes/min) the slopes show a clear dependency on the compression speed.

By plotting the hardness data for the Avicel compacts in the form of the relative hardness, $\ln H/\ln H_o$, versus the normalized solid fraction, P, it is seen that the slopes of the initial linear region increase with increasing rates of compression (see Table 4) indicating an increasing fragmentation propensity (Holman and Leuenberger, 1989b). Following the report of Sixsmith (1982) that Avicel fragments under pressure and keeping in mind that Avicel is viscoelastic, its response to increasing compression rates with increasing fragmentation may be explained by the fact that generally the stress-relieving properties of viscoelastic materials diminish with increasing compression rates.

TABLE 4

Slopes, b, of the relationship between relative hardness, $\ln H/\ln H_o$, and the normalized solid fraction, P, for Avicel compacts compressed at different speeds of compaction

Compression speed [strokes/min]	Slopes b_s	Slopes b_a
15	0.794 ± 0.009 *	0.573 ± 0.010
	$0.772 - 0.816$ +	$0.451 - 0.695$
25	0.877 ± 0.021	0.511 ± 0.014
	$0.794 - 0.960$	$0.311 - 0.691$
40	0.929 ± 0.021	0.549 ± 0.020
	$0.876 - 0.982$	$0.464 - 0.634$

*, Mean ± S.D.; +, 95% confidence interval. Subscripts are the same as in Table 1.

The observation that the slope, k_s, for Avicel is dependent on the speed of compression does not contradict the hypothesis that the hardness or elasticity of compacts with the same degree of interparticle contact area should be the same independent of compaction rates. This is because as explained elsewhere (Holman and Leuenberger, 1989b), due to the changing fragmentation propensity of Avicel with changing compression speed, the compacts compressed at different speeds to a certain normalized solid fraction will have different degrees of interparticle bonding. In other words, the compacts compressed at different speeds, although they exhibit the same normalised solid fraction, are bonded (particle-particle bonds) to varying degrees. Therefore the dependence of the slope on the rate of compression is to be expected. If, however, the mechanical properties of the tablets compacted at different rates are compared at that point where it is certain that the interparticle contact area is the same for all the compacts in spite of the rate of compaction, that is at zero porosity, the truth in the hypothesis becomes obvious.

Generally, it may be said that although for predominantly viscoelastic materials, represented by Avicel in this study, the slope, k_a, the hardness, H_o, and elasticity, E_o^*, at zero porosity obtained by extrapolation show no dependency on the rate of compression in the range of compression speeds used in this study, the slope, k_s, increase with increasing speeds of compression. For relatively non-viscoëlastic materials as represented by lactose in this study, all the parameters are essentially insensitive to the changing speeds of compression used in this study.

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