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COMMUNICATION

An Investigation of the Influence of the Core Material Properties on the Compression and Properties of Dry-Coated Tablets

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ABSTRACT

The effect of core material properties on the process of compression and physical properties of compression-coated tablets were investigated using microcrystalline cellulose as the coating material (mantle). Three model core materials: borosilicate glass, silicone rubber, and steel, each of different Young's modulus, were selected to give a range of core mechanical properties. Coated tablets were prepared using a single-punch press, with facilities for monitoring the compression cycle and analysis of data using the Heckel (1) equation. This analysis showed a considerable effect of different core materials on the compression process, (i) as an unanchored core, and (ii) due to core material type with differing Young's modulus.

INTRODUCTION

Compression coating of tablets was first introduced during the period 1950–1960, and was superseded approximately a decade later because of the advantages in speed and cost of film coating. Compression coating has been reviewed as a means of producing combination dosage forms in which two active substances are to be targeted to different areas of the gastrointestinal tract, as an alternative to an encapsulated bead formulation. It is possible that compression coating could utilise the advantages of direct compression of both core and coat thus obviating the need for separate coating processes and use of coating solutions.

During the development of compression-coated tablets, it was observed that certain conditions caused these tablets to exhibit fissures on their radial edge. Simulation of this phenomenon with placebo cores increased the incidence of fissures by a factor of 10-fold, implying that core properties could influence the tablet structure and propensity to fracture.

The aim of this research program was to investigate

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Table 1

Density and Young's Modulus of Tableting Materials

Core Material	Density (ρ) (g cm^{-3})	E (GPa)
Avicel	0.8	9.2
Rubber	1.2	0.0007*
Glass	2.2	70
Steel	7.8	210

*E at 100% elongation

the influence of the core on i) the compaction process, and ii) the structure and properties of the tablet, in order to identify critical factors and thus provide a scientific approach to the formulation of dry-coated tablets. In this work, one coating material and three standard cores of differing Young's modulus (E) were investigated in order to assess changes in both the compression cycle and the physical properties of the tablets caused by the core material.

MATERIALS AND METHODS

One batch of microcrystalline celluose (Avicel PH101) was used for all tablets. The mean particle density (ρ_p) of this material was 1.62 g cm^{-3} (mean of 10 determinations) as measured with a helium pycnometer model 1305 (Micromeritics, Norcross, Georgia), compared with the value reported previously (1.53 g cm^{-3}) by air pycnometry (2). Other materials used were magnesium stearate, borosilicate glass, silicone rubber (Amberlite-2035 A/2021B), and steel (Type 01 tool grade). Table 1 provides the Young's moduli for the core and mantle materials, along with the density of the core materials and the density of a coreless Avicel PH101 tablet for comparison. Twenty cylindrical discs, each of 6 mm diameter and 3 mm thickness, were manufactured from the cast silicone rubber, borosilicate glass, and steel, named above and were cut and ground to size.

Compression coating was achieved on a model F3 (Manesty, Liverpool, UK) press with displacement transducers and strain gauges on the upper and lower punches. Data acquisition was by means of an IBM PS2 model 30 286 computer fitted with a mathematics coprocessor and an analog-to-digital board. Circular 10-mm-diameter flat-faced punches were used for compression. Lubrication of the die was achieved by swabbing with 10% w/w magnesium stearate suspended in acetone before commencing each run of 20 cycles.

Die filling, core centralization, and machine operation were done by a standardized manual operation. Central placement of the core on an even bed of microcrystalline cellulose was preceded by manual consolidation of the lower coating layer prior to placement. The upper punch velocity was controlled between 13.2 mm s^{-1} and 16.4 mm s^{-1}, and the upper punch die penetration was set to give a tablet density between 0.8 and 0.9 g cm^{-3}. All compressed tablets were stored for 96 hr at 30°C and 32.4% relative humidity to reach equilibrium before thickness, weight, and diametral breaking load (Schleuniger Lab 3 S) were measured.

Compaction-cycle data for individual tablets were analyzed with Heckel plots (1) of compaction pressure vs. log ($^1/_\epsilon$), where ϵ = porosity. The major straight-line portions of the graphs were used for determination of the yield pressure (Py) for microcrystalline cellulose under these experimental conditions. Compaction-work values (3) for individual tablets were calculated. Tablet dimensions and diametral breaking load were measured to calculate the tensile strength (σ) (4) and the Weibull modulus (m) for each type of compact (5).

RESULTS AND DISCUSSION

Yield Pressure Data

Microcrystalline cellulose has been shown to exhibit very little fragmentation (6), and the principal mechanism of its consolidation at high pressure is plastic flow. The three major phases of the compaction cycle could be divided into: i) die filling and particle rearrangement, ii) particle deformation, and iii) plastic flow, with bonding to form the compact. In this case, the die filling and initial particle packing had been standardized by manual consolidation of the powder bed, thus allowing an even bed for placement of the core, followed by manual consolidation of the upper coating layer prior to compression. Comparison of Heckel plots of tablets compressed with and without precompression has shown that the latter does not affect the Heckel plot of microcrystalline cellulose (7).

The value of Py (Table 2) was calculated from the reciprocal of the slope of the major linear portion of the graph, using linear regression ($r >= 0.95$). The Heckel plots of nine compression cycles from each core type were plotted from an available 40 cycles, and the mean value was taken. The results are shown for steel cores (Fig. 1), glass cores (Fig. 2), rubber cores (Fig. 3), and

Table 2

Heckel Analysis Results

Core Type	Py (MPa)	*y*-Intercept
Coreless tablets	93	0.32
Rubber, run 1	229	0.33
Rubber, run 2	202	0.31
Glass (pooled)	1255	0.43
Steel (pooled)	1013	0.42

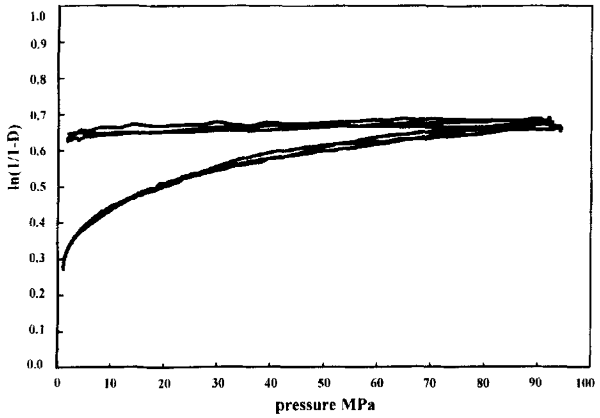

Figure 1. Log (1/porosity) vs. applied axial force for steel-cored tablets.

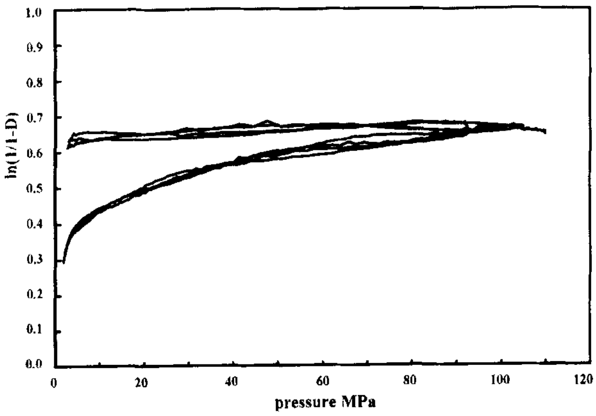

Figure 2. Log (1/porosity) vs. applied axial force for glass-cored tablets.

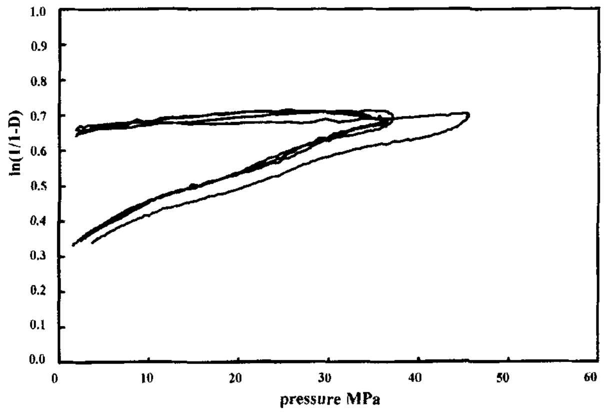

Figure 3. Log (1/porosity) vs. applied axial force for silicone rubber cored tablets

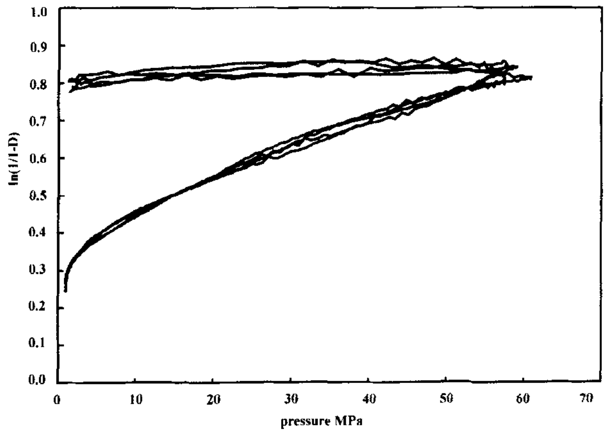

Figure 4. Log (1/porosity) vs. applied axial force for Avicel PH101 tablets.

coreless tablets (Fig. 4). The major straight-line portion of the curve is related to the viscoelastic behavior of the powder bed under high-pressure compression.

The Py value obtained from these results is an approximation of the yield pressure for plastic deformation, since the measurements were taken within the die at maximum applied load. Because elastic recovery of the compact had not taken place, the results included an elastic stress component (8,9). It should also be noted that the low Py value for the rubber-cored tablets may reflect viscoelasticity in the core as well as the mantle. However, the Py value is still considerably higher than that seen for tablets without a core (93 MPa).

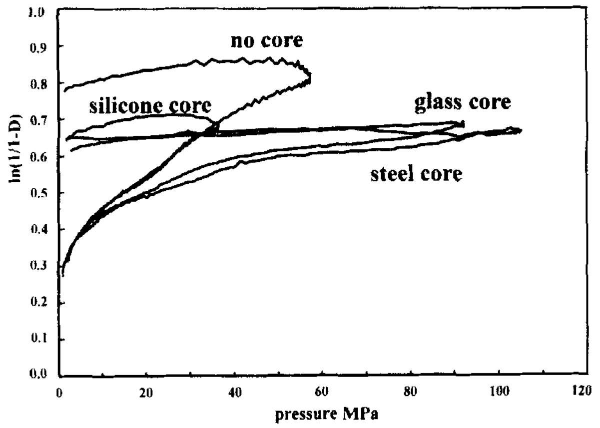

Figure 5. Log (1/porosity) vs. applied axial force for coreless (plain disc) and glass-, rubber-, and steel-cored tablets.

In Table 1, the values of Py are ranked in the order rubber < steel < glass, however this order does not correlate with core density or Young's modulus. It is apparent that there is a significant difference in the Py values between rubber-cored and the other cored tablets, but no significant difference exists between the means for glass and steel ($p = 0.005$). It is possible that the propensity of the core to store compaction energy in terms of its elasticity may affect the measured yield pressure, since there is only a 3-fold decrease in the modulus from steel to glass, as compared to a 10^5-fold decrease from glass to rubber. This indicates that where the difference between the Young's moduli of the core materials is of order of 10^5, a significant change in the yield pressure of the mantle can be determined.

Because of the limited number (20) of each type of core, reuse was necessary. This proved satisfactory for glass and steel, but statistically different compression properties ($p = 0.005$) were obtained for tablets for the two sets prepared with once- and twice-used rubber cores.

Figure 4 shows that for two sets of Avicel coreless tablets, the mean yield pressure values were 91.3 and 93.2 MPa. When the results for Avicel coreless tablets are compared in Figure 5 with Avicel containing different core materials, a considerable distinction is found in the Heckel plots for the coreless and cored tablets. The *y*-intercept values taken from Figs. 1–4, presented in Table 1, are considered to be a measure of the energy used for the particle rearrangement phase. The *y*-intercept values for coreless Avicel and rubber-cored tablets are very similar, but a considerable difference in value is seen in the presence of glass or steel cores. The results for steel- and glass-cored tablets provide evidence for the effect of these cores on the rearrangement of Avicel particles. However, it cannot be concluded that there is less effect on particle rearrangement in the presence of rubber cores, since the low *Y*-intercept value may be partly due to the much lower Py value for these tablets.

Net Work of Compression

The net work of compression (*netw*) was calculated by subtraction of the sum of work of friction and expansion from the gross work input, taken as the mean punch work done (3). The ratio of *netw* to gross work has been proposed as a measure of the proportion of the total work input that contributes to tablet strength, and also as a direct measure of the plasticity of the tablet materials.

Calculation of the *netw* for each batch of tablets (Table 3) again showed a similar rank order to Py (Table 1). The glass and steel *netw* values showed no significant difference, whereas the values for rubber cored tablets showed a significant difference from that of both steel- and glass-cored tablets ($p = 0.005$). The work ratios (Table 4) also showed no difference between glass and steel, but a significant difference between the first rubber batch and all other tablets. The second rubber-cored batch shows a difference, as observed with the Py values. The difference between the first and second batches of rubber-cored tablets suggests that a loss of elasticity occurred in the cores after the first compression and during subsequent storage prior to the second compression. Much lower work-of-expansion values were seen on the second compression, and this resulted in a significant change in the ratio of *netw*/gross work done.

Tensile Strength (σ) and Weibull Modulus (m)

The σ values of the compacts were calculated from the breaking load, and the individual values were then used to plot the log–log probability function (loglog P_f) against the log (σ/mean σ) for each sample. The Weibull modulus was then calculated, using linear regression to determine the slope of the graph.

The results of significance testing of the pooled σ data given in Table 5 show no statistical difference between the means of the steel- and glass-cored tablets, and also between those of the rubber- and glass-cored tablets, but there is a significant difference between the mean σ values for steel- and rubber-cored tablets ($p = 0.005$).

Table 3

Work of Compaction for Compressed Tablets

	Mean Work Done (J)					
Batch	Upper Punch	Lower Punch	Mean Punch Work	Friction Work	Expansion Work	*netw*
Rubber 1	5.2	3.1	4.2	2.1	1.3	0.8
Rubber 2	4.8	3.0	3.9	1.8	0.3	1.8
Steel 1	6.8	4.9	5.9	1.9	0.4	3.6
Steel 2	6.8	4.9	5.9	1.9	0.4	3.6
Glass 1	7.7	5.9	6.8	1.8	0.7	4.3
Glass 2	7.2	5.5	6.4	1.8	0.7	3.8

Table 4

Work Ratios for Compressed Tablets

	Mean Work Ratios	
Batch	Lower/Upper Punch Work	*netw*/ Work Done
Rubber 1	0.60	0.19
Rubber 2	0.63	0.46
Steel 1	0.72	0.61
Steel 2	0.72	0.61
Glass 1	0.77	0.63
Glass 2	0.76	0.59

Table 5

Tensile Strength and Weibull Modules of Compressed Tablets

Batch	Mean σ (MPa)	*m* (pooled)
Coreless tablets	1.28	8.6
Rubber cores 1	0.56	
Rubber cores 2	0.54	9.3
Glass cores 1	0.54	
Glass cores 2	0.66	7.8
Steel cores 2	0.66	
Steel cores 2	0.67	8.1

The data in Table 5 show that differences in ρ and *E* (values given in Table 2) do not have a major influence on the tensile strength of the dry-coated tablet. Although the inclusion of a core has a dramatic influence on the tensile strength, there is no change in the probability of fracture as defined by the Weibull modulus, which appears to show only experimental variation.

CONCLUSIONS

The physical properties of the core can have a dramatic effect on the compression of dry-coated tablets. The yield-pressure and net-work-of-compaction data both show a significant difference between the values for rubber and those for stiffer materials (e.g., glass or steel) used as core materials with an Avicel mantle. No significant difference could be determined for tablets with cores of glass and steel, even though density differences were greater than between rubber and glass. This study suggests that the density of the core material is not a critical factor and that small changes in the Young's modulus of the core do not have a significant effect on the tablet properties. However, the large difference for Avicel tablets with cores of steel and rubber was demonstrated by the significant differences in the Py and *netw* values for tablets with these cores, which was attributed to the behavior of the core under pressure. Figure 5 illustrates the differences in Heckel plot for coated tablets as compared with the coreless tablets, for which there was an obvious change in both the particle rearrangement/die filling phase and the particle deformation and bonding stage. Thus, two effects were seen: i) the influence of an unanchored core, and ii) the change in compression of tablets with core materials of differing elastic modulus. This was confirmed by the change in Py, *netw*, σ, and *m* values between tablets with stiffer cores and rubber-cored tablets. This investigation, using model core materials, has provided a basis for further detailed studies using pharmaceutical powders as the tablet core material.

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