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Force-time Curves of a Rotary Tablet Press. Interpretation of the Compressibility of a Modified Starch Containing Various Amounts of Moisture

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Abstract

On a rotary tablet press, the force-time curves are segmented into three phases: the compression phase, the dwell phase during which both stress and strain are variable for plastically deforming materials and the decompression phase. The following seven parameters were investigated: the compression area (A_1) and the compression slope (Sl_c) describing the initial phase, the area ratio (AR) and the peak offset time (t_{off}) characterizing the dwell time, the decompression area (A_4) and the decompression slope (Sl_d) describing the terminal phase and the total area under the force-time curve (A_{tot}). AR, t_{off}, Sl_c and A_1 (the last with limitations) are used for phase-specific allocation of the occurrence of plastic flow, which is found to be a function of compression force and moisture content. Tablet strength, tablet porosity and in-die bulk porosity provide additional information for comprehensive interpretation.

The values of A_4 for the four starch batches are not significantly different. Sl_d provides somewhat better information about the elastic compact recovery. In general, however, the short decompression phase seems to be inappropriate for characterization by force-time curve parameters, because it is difficult to separate machine recovery from that of the tablet.

Porosity above the porosity limit of the material was found to be a prerequisite for plastic flow within the compact. When the porosity limit is reached, further densification remains elastic and leads to a reduced compact strength during expansion. The area ratio, as a robust in-process control parameter for plastically flowing formulations, is suggested as a means of preventing this effect.

Starches are widely used excipients in the food, paper, textile and pharmaceutical industries. These complex biopolymers consist mainly of amylose and amylopectin with different degrees of polymerization. The structures of the isolated carbohydrates have been studied extensively, whereas the association of the two components in the starch granule is not yet fully understood (Imberty et al 1991). In the pharmaceutical industry, starches serve as wet binders, fillers and disintegrants in tableting. They show time-dependent, plastic behaviour (Shlanta & Milosovich 1964; David & Augsburger 1977) and considerable elastic recovery (Travers et al 1983; Esezobo & Pilpel 1987) during direct compression.

Moisture content has an important effect on the properties of starch and starch-based formulations (Seth & Münzel 1959; Schepky 1974; Hess 1977). The effect of moisture content on the compression properties of starches and their derivatives has been investigated (Horikoshi et al 1977; Li & Peck 1990; Malamataris et al 1991; Ollett et al 1993; Rees & Tsardaka 1994). Tablets were compressed on single-station presses using external lubrication. Except for those of Horikoshi et al (1977) and Rees & Tsardaka (1994), the compressibility studies were based on Heckel parameters only.

The aim of this study is to discuss the values of different force-time curve parameters for a rotary tablet press run under conditions close to industrial practice. Starch 1500 containing various amounts of moisture is used as a model substance.

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Compression phases and parameters of the force-time curve (Fig. 1)

Depending upon the geometry of the punch head, force-time curves of a rotary tablet press can be segmented into three phases (Koch 1990; Schmidt & Vogel 1994): the compression phase, during which time the punches approach each other; the dwell phase (dwell time), where the punch head flats are in direct contact with the compression rollers; and the decompression phase, during which time the punches move away from each other.

Particle porosity and deformability as well as particle shape, roughness, size distribution and inter-particle forces are the basic properties of the powder influencing the complex changes

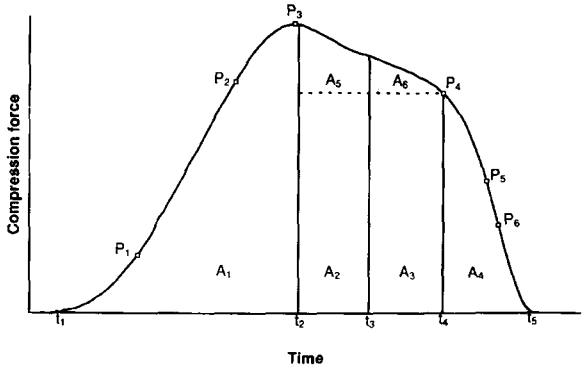

FIG. 1. Tableting phases and parameter of the force-time curve on a rotary tablet press. Phases: compression phase (t_1 to t_2), stress relaxation phase (t_2 to t_4), decompression phase (t_4 to t_5), densification phase (t_1 to t_4). Time parameters: middle of dwell time (t_3), contact time (t_1 to t_5). Other parameters are described in the text.

within the die during the densification phase from t_1 to t_4 (Fig. 1). The biggest changes in volume occur during the compression phase, i.e. from t_1 to t_2. Both the area A_1, as an integral parameter, and the compression slope Sl_c, as a differential parameter, can describe this phase. A_1 simply represents the area under the curve during compression. Sl_c is the slope of the straight line connecting the points P_1 and P_2. At P_1 and P_2, respectively, 20% and 80% of the maximum force in P_3 are reached. The parameter Sl_c is adopted from the slope definition of Jones (1983), based on two distant data points. Chilamkurti et al (1982) defined a similar slope parameter, the maximum slope on the leading edge of the force-time curve, as a one-point parameter.

Concerning the dwell time (t_2 to t_4), there exists a common perception that no further vertical displacement of the punch faces occurs. It is claimed that stress relaxation occurs at constant strain. In real tableting, however, punch faces do approach each other during dwell time, provided that the compact undergoes stress relaxation, because plastic flow leads to reduced tablet porosity, i.e. to thinner tablets. In consequence, punch faces approach each other, thereby enabling the machine (i.e. punches, rollers, pins, rockers) to recover elastically and to reduce strain. According to equation 1:

$$\sigma = E\varepsilon \quad (1)$$

reduced strain ε results in reduced stress σ, so that the punch force is reduced (E = Young's modulus). For plastically deforming materials, a force decay of 1–2 kN between P_3 and P_4 is realistic. On the basis of this knowledge and taking a machine deformation of 25 μm kN^{-1} (Vogel 1992) into consideration, the punches approach each other for another 25–50 μm during the dwell time. Koch (1990) gave experimental evidence for these assumptions for the compression of Starch 1500 on a Fette P2 rotary tablet press. He used a two-point displacement instrumentation described by Schmidt & Tenter (1985). It might, therefore, be concluded that both stress and strain are variable during dwell time for plastically deforming materials.

In Fig. 1, A_2 and A_3 represent the areas under the curve from t_2 to t_3 and t_3 to t_4 respectively. The areas A_5 and A_6 are elements of the areas A_2 and A_3. They are limited by the dotted line passing through P_4, which is the minimum force within dwell time. The area ratio A_6/A_5 (AR) is used to quantify stress relaxation (Vogel 1992; Vogel & Schmidt 1993) during dwell time. A low AR value indicates high stress relaxation. Dwivedi et al (1991) proposed the peak offset time (t_{off}) as a parameter describing stress relaxation. According to these workers, the peak offset time is the difference between the time of P_3 and t_3, where P_3 is the data point at maximum force and t_3 is the middle of the dwell time.

During the decompression phase, between t_4 and t_5, both the machine and the compact undergo rapid recovery. Machine recovery is governed by its elastic modulus and by the inertia associated with the mass of its rockers, pins, compression rollers and punches. Danielson et al (1983) described the elastic and viscoelastic component of tablet recovery at a macroscopic level using a rotary press. Hiestand (1991) discussed tablet recovery at an inter-particle level. He described two different mechanisms, ductile and the brittle extension, each of which reduces the inter-particle contact area previously formed, but to a different extent.

A_4 and Sl_d describe the decompression phase; most of the elastic recovery of the compact occurs during this period. A_4, the decompression area, represents the area under the decompression curve from t_4 to t_5. The decompression slope Sl_d is the slope of the straight line connecting the points P_5 and P_6, where 60% and 40%, respectively, of the minimum force during dwell time in P_4 are reached.

The total area A_{tot} represents the area under the curve from t_1 to t_5 (Chilamkurti et al 1982). This parameter, which equals the sum of A_1, A_2, A_3 and A_4, is also discussed in this work.

Materials and Methods

Materials and storage

Starch 1500 (3·5 kg; lot #402026, Colorcon GmbH, D-61462 Königstein, Germany) containing 0·4% magnesium stearate (lot #190151, O. Bärlocher GmbH, D-80992 München, Germany) were mixed at 40 rev min^{-1} for 2·5 min in a 10-L drum mixer (type 'Röhnrad'). The mixture was dried for 17 h at 50°C in a tray dryer to ensure that the powder absorbs water under all storage conditions. The powder was equally distributed on 20 square glass plates (20 × 20 cm) to form a layer not thicker than 1 cm. Five loaded glass plates were each stacked horizontally with a vertical separation distance of 2·7 cm using metal frames and four loaded metal frames were transferred to desiccators filled with saturated salt solutions (Table 1). After a storage period of 4 months at room temperature in the dark, the powders were transferred into four tightly closed containers, mixed, and then stored for a further 6 days for final equilibration. Directly before compression, the loss on drying of the four batches was determined in an oven according to USP 23/NF 18 (Table 1). The powders were fed to the machine and compressed immediately as described below.

Table 1. Storage conditions and powder parameters of the four equilibrated batches of Starch 1500 containing 0.4% magnesium stearate.

Storage conditions[a] Saturated solution	rh (%)	Powder parameters		
		True density[b,c] (g mL^{-1})	Dry loss[b,d] (%)	Batch description
$CaCl_2$ hexahydrate	30	1·492 ± 0·004	7·8 ± 0·3	S 7·8
K_2CO_3 sesquihydrate	44	1·485 ± 0·004	9·5 ± 0·4	S 9·5
$Mn(NO_3)_2$ hexahydrate	54	1·484 ± 0·001	10·4 ± 0·2	S 10·4
NaCl	75	1·471 ± 0·001	14·2 ± 0·1	S 14·2

[a] Stahl (1980); [b] mean ± s.d. (n = 3); [c] Beckman helium pycnometer model 930, method purge 1 to 2 atmosphere; [d] according to USP 23/NF 18 (loss on drying/starch), drying temperature 120°C, 4 h.

Machine and tableting

A Korsch PH230/17 rotary tablet press (Korsch Pressen GmbH, D-13474 Berlin) was used to prepare the tablets at ambient temperature and humidity. The active die table diameter of the press was 195 mm and the compression roller diameter was 200 mm. Four of the 17 punch stations were active, equipped with 10-mm B-tooling with an overall length of 133·6 mm (flat face, bevelled edge, punch head curvature type "manesty", punch head flat diameter 10 mm). The press and the feeder rotated at a speed of 25 rev min^{-1}.

The filling depth was set to a calibrated value of 5·21 mm (depth micrometer series 129, Mitutoyo Co. Ltd., Japan) resulting in a die filling volume of 430 mm^3 (409 mm^3 calculated from filling depth and die radius, plus 21 mm^3 volume in the lower punch facet (bevelled edge). Force levels of 5, 10, 15 and 20 kN were set approximately by adjusting the edge thickness. The hydraulic-pneumatic overload protection was kept at 43 kN (10·5 MPa).

Instrumentation, data acquisition and processing

Press instrumentation, electronic data acquisition and processing have been described elsewhere (Schmidt & Vogel 1994). Data were sampled at a rate of 5 kHz per channel. Only the force data of the upper rocker were evaluated. The parameter statistics were calculated from 9–10 tableting events. For the porosity at zero force and the total area, error estimation was performed according to Bronstein & Semendjajew (1991).

Tablet parameters

Twenty four hours after ejection, the mass (AE 200 balance, Mettler-Toledo GmbH, D-35396 Gießen, Germany), thickness (0·01 mm micrometer, Mitutoyo Co. Ltd, Japan) and the crushing strength (model 6D, Dr. Schleuniger Pharmatron AG, CH-4501 Solothurn, Switzerland) were measured for ten tablets. The tensile strength, which was introduced to pharmaceutical preparations by Fell & Newton (1970), was calculated according to Frocht (1948). For porosity calculations, the true density of the batches was determined using a Beckman helium pycnometer (model 930, Beckman Instruments, Inc., Fullerton, CA 92634, USA; Table 1). The tablet porosity, which is a measure of the irreversible densification of the compact, was calculated from the dimensions and weight of the tablet and the true density of the powder. From the die filling volume, the weight of the resulting tablet and the true density of the powders, the in-die bulk porosity was calculated for each batch. This parameter reflects changes in the powder as a result of the action of the feeder. The in-die bulk porosity is based on the density at work (Hüttenrauch & Roos 1970).

Results and Discussion

Densification behaviour (compression phase and dwell time)

Fig. 2 shows the projection of force-time profiles for Starch 1500 at two different force and moisture levels. During the dwell time, at a force level of approximately 5 kN, both curves show virtually the same force decay. At a force level of 10 kN during dwell time, however, starch with 14·2% dry loss (S 14·2, Table 1) reveals significantly less stress relaxation than starch with 9·5% dry loss (S 9·5). This might, at first sight, appear an unexpected result because water acts as a plasticizer in starches (Zografi & Kontny 1986), reducing the glass transition temperature (Zeleznak & Hoseney 1987). During the compression phase, however, it is obvious that for both force levels the slopes Sl_c and areas A_1 are lower for S 14·2 than for S 9·5. It is therefore assumed that for S 14·2 stress relaxation by plastic flow mainly takes place during the early stages of the tableting process at low compression forces.

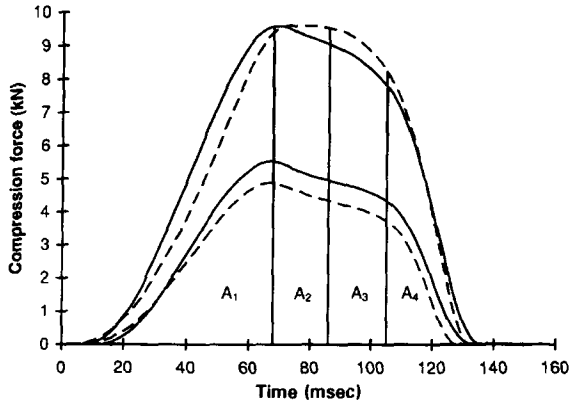

FIG. 2. Compression force-time profiles for two samples of Starch 1500 with different moisture content at two different force levels. —, 9·5; — —, 14·2% dry loss.

Fig 3 shows the densification behaviour of the four batches. It is apparent that the higher the moisture content, the greater the porosity changes at low forces and the lower the porosity limits. S 14·2, moreover, reaches its porosity limit of 0·14 at a force of 10 kN, whereas the batches with lower moisture content reach their respective porosity limits ($\geq$ 0·16) at forces of at least 15 kN. This is in agreement with Malamataris et al (1991) and Ollett et al (1993), who found that the yield pressure decreases with increasing moisture content, thus facilitating densification and porosity reduction in starches. Compressing modified corn starch, Esezobo & Pilpel (1987) also found a porosity limit higher than zero porosity. It is noteworthy that S 14·2 shows the highest bulk porosity at zero force, indicating high cohesiveness and poor flowability. This further supports the statements of Stahl (1980) and Zografi & Kontny (1986) that water binds stoichiometrically to each anhydrous glucose unit within the

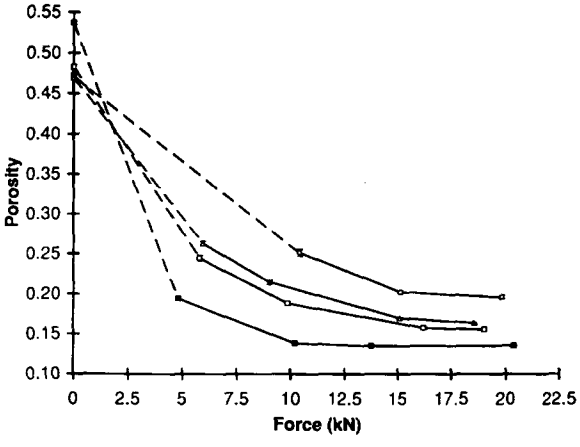

FIG. 3. Effect of force and water content on the porosity of Starch 1500. Data for zero force: in-die bulk porosity determined before compression. Other data: tablet porosity 24 h after compression. Error bars represent the 95% confidence interval of the mean. ○, 7·8; △, 9·5; □, 10·4 and ■, 14·2% dry loss.

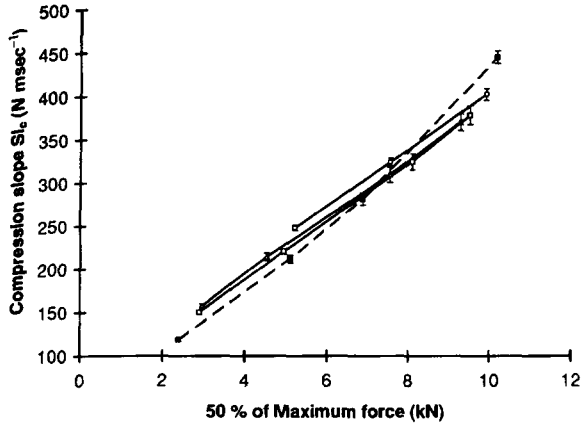

FIG. 4. Effect of force and water content on the slope Sl_c of the force-time curve for Starch 1500 during the compression phase. Error bars represent the 95% confidence interval of the mean. ○, 7·8; △, 9·5; □, 10·4 and ■, 14·2% dry loss.

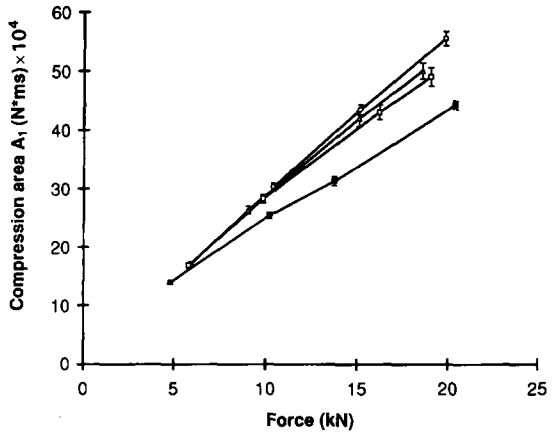

FIG. 5. Effect of force and water content on the compression area A_1 under the force-time curve for Starch 1500 during the compression phase. Error bars represent the 95% confidence interval of the mean. ○, 7·8; △, 9·5; □, 10·4 and ■, 14·2% dry loss.

starch polymer up to a water content of 10 to 11%. At higher water content, water binds less tightly to the polymer, becoming available for starch granule interactions. In addition, granule swelling (Rutenberg 1980) might contribute to the changes in bulk density and porosity.

Fig. 4 shows the effect of force and water content on the compression slope Sl_c. This parameter, which is a measure of the rate of force increase during the compression phase, depends on the actual resistance of a material against further densification and on the actual punch speed. Sl_c represents a mean slope at 50% of the maximum force on the y-axis, as it is calculated from 20% to 80% of the maximum force. Fig. 4 therefore displays the slope Sl_c against 50% of the maximum force on the x-axis and not against the maximum force.

Because the compression time is approximately the same for all experimental conditions (57–62 ms; Fig. 2), higher force levels must be reached at higher rates and slopes. The differences between the slope curves of S 9·5 and S 10·4 are rather small, because the difference in moisture content is only 0·9%. For the two lower force levels, Fig. 4 shows that the higher the plastifying moisture content, the lower the slope. This is expected from Fig. 2. With increasing force, however, the slope values of S 14·2 rise significantly more than the slope values of the other three batches. At the highest force level, the slope of the curve for S 14·2 is the highest of all four curves. The reason for this is probably that the porosity limit is reached at a relatively low force of 10 kN for S 14·2. After passing the force where the porosity limit is reached, no further irreversible densification is achieved and only pure elastic deformation seems to be possible for the tablet. From there the Young's modulus of the tablet should theoretically remain constant. It is therefore proposed that the slope Sl_c is an inverse measure for the ability of the starch batches to undergo stress relaxation, as long as the porosity limit is not yet reached (force level 5 kN, see Fig. 3 and Fig. 4).

Fig. 5 shows the effect of force and water content on A_1. In agreement with Fig. 2, it is seen that the higher the moisture level, the lower is the area A_1. Excluding S 9·5, the differences in A_1 among the remaining batches are statistically significant at the two highest force levels. In contrast with the more localized parameter Sl_c, A_1 covers a force range from zero kN to approximately maximum force. Hence, A_1 is a more general parameter than the slightly more specific property Sl_c.

Fig. 6 shows the effect of force and water content on the total area of the forcetime curve. This plot, on which the A/H ratio of Chilamkurti et al (1982) is based, provides less information than the parameter A_1 in Fig. 5. The total area seems insufficiently specific to reveal significant differences among the three batches with lower moisture content. Because the total area is not, however, related to press-specific tableting phases, it might be helpful for comparison of compression on eccentric and rotary tablet presses.

As Vogel & Schmidt (1993) showed, the area ratio A_6/A_5 (AR) describes the densification behaviour of a powder during dwell time and might be used to distinguish tableting excipients. According to their findings, plastically deforming materials show AR values of 0·3–0·45 at low compression forces and a big increase of the AR with increasing force. Substances with mainly brittle behaviour show AR values higher than 0·6 that do not increase much with increasing force. A symmetrical curve of a purely elastic material ideally shows a maximum AR of 1.

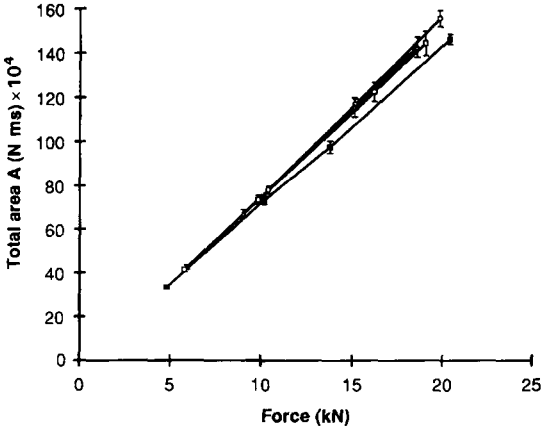

FIG. 6. Effect of force and water content on the total area A_{tot} under the force-time curve for Starch 1500 within contact time. Error bars represent the 95% confidence interval of the mean. ○, 7·8; △, 9·5; □, 10·4 and ■, 14·2% dry loss.

Fig. 7 illustrates the effect of force and water content on the AR. For all the four batches, the AR increases with increasing force. This indicates that further stress relaxation becomes more difficult when the actual force level is higher. It is apparent that at a force of 15 kN the higher the moisture content, the higher the AR, because of the lower tablet porosity which is reached (Fig. 3). S 14·2 has a significantly higher AR than the other batches at forces of 10 kN. Again, this is because S 14·2 reaches its porosity limit at 10 kN, whereas the other three batches reach theirs at 15 kN or slightly higher. When the porosity limit is reached, further densification is supposed to be elastic. Hence, the force-time curve during dwell time becomes more symmetric and the AR rises. At around 5 kN, differences between the ARs of the batches with different moisture content are almost negligible. With increasing moisture content, the plastic deformation of starch is simplified so that an increasing part of the plastic deformation occurs during the compression phase and a decreasing part occurs during dwell time at a given maximum force level. In consequence, stress relaxation during dwell time decreases and elastic deformation increases with increasing moisture content of the starch.

In summary, a certain amount of available void space, i.e. porosity higher than the porosity limit, is a prerequisite for stress relaxation by irreversible plastic flow. Volume reduction by changes in crystal modification may also cause stress relaxation during tabletting (Dietrich 1983). The fact that a finite porosity, but not zero porosity is reached in this experiment, might be explained as follows.

Firstly, increasing force leads to decreasing porosity with increasing inter-particle contact area. Thus, with increasing force, the total tableting stress at punch face level is distributed to a growing inter-particle contact area within the tablet. Local inter-particle shear stress can be defined as shear force divided by contact area. The local shear stress might possibly decrease with increasing punch force, assuming that the shear force does not increase to the same extent as the contact area. The force at which the porosity limit for plastic flow is reached might therefore be defined as the force where local inter-particle shear stress falls below the local yield pressure. It is assumed that the lower the porosity, the more equally the particles are stressed. Thus, the influence of shear stress on the particle is reduced and normal stress is favoured. Because plastic flow is a matter of shear stress, it also is reduced.

Secondly, air entrapped in the closed pores formed during compression might be an additional reason why zero porosity is not reached in the ejected tablet. It must, however, be considered that plastic flow is time-dependent. So, with infinite dwell time, zero porosity could be approached closely at very high punch forces during compression.

The peak offset time (t_{off}) proposed by Dwivedi et al (1991) provides essentially the same information as the AR (Fig. 8). In addition, the relative length of the error bars does not differ in sum for AR and t_{off}. For starch with 14·2% dry loss at 10 kN compression force, however, the error for t_{off} seems to be relatively bigger in comparison with the error of the area ratio. This can probably be explained by the noise in the signal of the corresponding data file, on which the peak time is sensitive. In this circumstance, the AR seems to be more robust, because it is an integral parameter.

Fig. 9 illustrates the dependence of the tensile strength on the compression force. In general, a higher moisture content leads to stronger tablets. Again, the plasticizing action of increasing water content reduces the porosity of the compact. Thus, inter-particle contact area, a prerequisite for inter-particle bonding, increases. The compact strength of S 14·2 is extraordinarily high compared with the other three batches. This might be attributed to the presence of less tightly bound water (Zografi & Kontny 1986), enhancing the plasticity of the material. S 14·2 and S 10·4 show strength optima at 10 and 15 kN, respectively. For these two samples, the force corresponding to the optimum strength is equal to the force where the porosity limit is reached. As stated above, it is reasonable to assume that at higher forces, compact deformation is mainly elastic. Disruption of inter-particle bonds by elastic recovery during decompression is responsible for the decay in compact strength at forces beyond the optimum force.

These results are qualitatively in agreement with recent findings of Rees & Tsardaka (1994) who compressed the same material with different moisture contents on an eccentric press. They found that starch with the highest moisture content shows the lowest yield pressure at a force level of 6 kN but the biggest

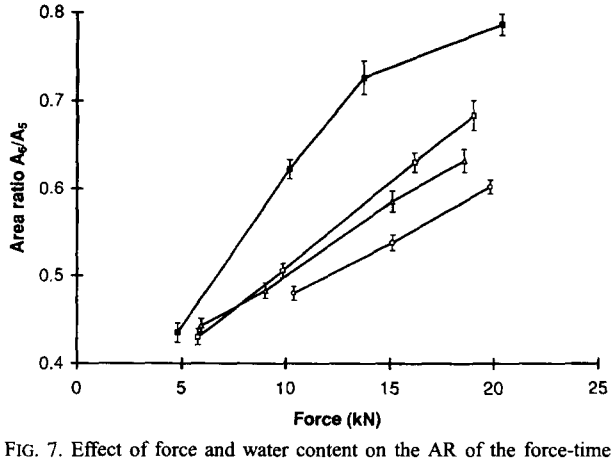

FIG. 7. Effect of force and water content on the AR of the force-time curve of Starch 1500 during dwell time. Error bars represent the 95% confidence interval of the mean. ○, 7·8; △, 9·5; □, 10·4 and ■, 14·2% dry loss.

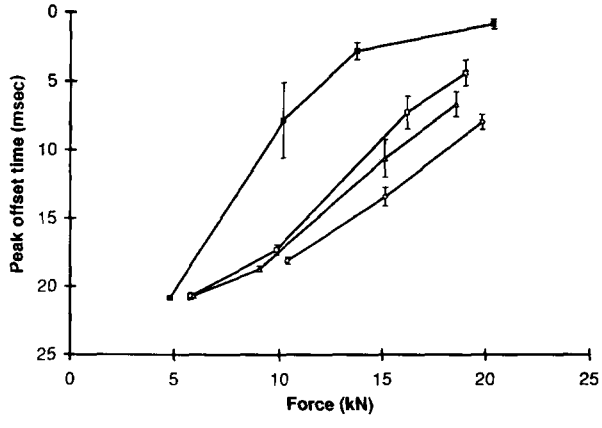

FIG. 8. Effect of force and water content on the peak offset time of the force-time curve of Starch 1500 (according to Dwivedi et al 1991). The y-axis is scaled inversely for easier comparison with Fig.7. Error bars represent the 95% confidence interval of the mean. ○, 7·8; △, 9·5; □, 10·4 and ■, 14·2% dry loss.

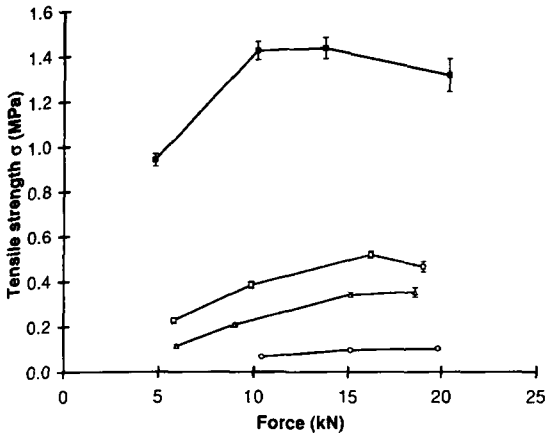

FIG. 9. Effect of force and water content on the tensile strength of tablets prepared from Starch 1500. Error bars represent the 95% confidence interval of the mean. ○, 7·8; △, 9·5; □, 10·4 and ■, 14·2% dry loss.

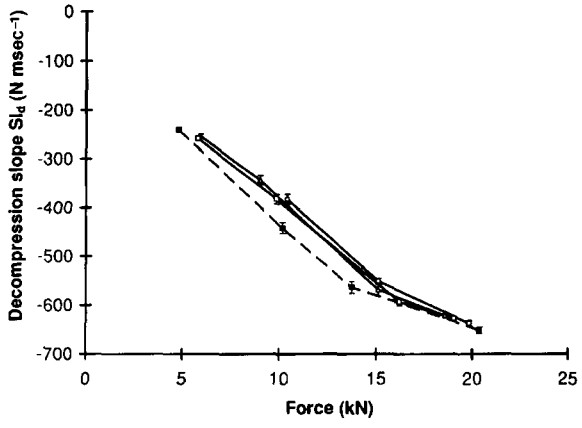

FIG. 10. Effect of force and water content on the slope Sl_d of the force-time curve of Starch 1500 during the decompression phase. Error bars represent the 95% confidence interval of the mean. ○, 7·8; △, 9·5; □, 10·4 and ■, 14·2% dry loss.

increase of the yield pressure with increasing force. They ascertained that the yield pressure is not only dependent on the water content, but also on porosity, as influenced by the compression force. Quantitative comparison with our results is not, however, possible because of the differences in the experimental parameters, e.g. press type, tooling and method of lubrication.

Decompression phase

A_4 describes the area under the force-time curve during the decompression phase, and cannot be used to distinguish among the four batches of corn starch. An explanation of this can, however, be deduced from Fig. 2. At a force level of 10 kN during decompression, the forcetime curve of S 14·2 is initially higher than that of S 9·5. After an intersection at about 5 kN, the force curve of S 14·2 drops below that of S 9·5, which is why there is no difference between the A_4 values of the two batches, although the shapes of the areas are different.

It is, therefore, reasonable to move from the integral parameter A_4 to the slope of the decompression curve Sl_d (Fig. 10), a more localized, specific parameter. Up to forces of 15 kN, S 14·2 shows a significant higher absolute Sl_d value than the other three batches. This is because the lowest porosity or highest solid fraction is reached, which probably results in a high elastic modulus of the tablets. A high elastic modulus causes the force to fall rapidly during decompression (Dwivedi et al 1992). At approximately 15 kN, the curves in Fig. 10 bend towards lower absolute Sl_c values, indicating that at higher compression forces the elastic recovery of the compacts is enhanced. The differences between the lower three curves are not, however, significant. At the highest force level, differences between all the curves are insignificant. It seems that differences in compact recovery might hardly be distinguished from machine deformation at higher forces. With decompression times of 20 to 35 ms for the actual experiment, decompression is the shortest of the three phases passed during tableting. This time seems to be too short for a proper analysis of the differences between the starch batches based on force-time curves only. It is, therefore, assumed that elastic properties of compacts are distinguished more adequately on mechanical testing machines after tableting. To determine elastic compact properties on a rotary press during tableting, accurate and precise displacement information is needed in addition to well-established force instrumentation.

Conclusions

Table 2 qualitatively summarizes the effect of compression force on the plastic and elastic behaviour of Starch 1500 compacts at a given moisture content. With increasing force, plastic flow increases during the compression phase and decreases during the dwell phase. At a very high force, there is no further plastic flow during dwell time, elastic compression alone occurs. This is reflected in the elastic recovery during decompression phase. The total compression is the sum of plastic flow during the densification phase and of the elastic recovery during the decompression phase. It increases from medium to high force, but remains constant from high to very high force.

The discussed parameters of the force-time curve illustrate the tableting process on a rotary tablet press. There are two types of force-time curve parameter, general parameters, which might be applied on both eccentric and rotary press profiles, and specific parameters related to rotary press profiles exclusively. The total area A_{tot}, the peak offset time t_{off} and the compression slope Sl_c are examples of the former whereas A_1 and A_4, the area ratio AR and the decompression slope Sl_d are examples of the latter.

A comprehensive interpretation cannot be obtained from a single parameter. One parameter per tableting phase should be

Table 2. Qualitative effect of the compression force on plastic flow (+) and elastic recovery (−) in Starch 1500 compacts at a given moisture level. 0 = no further plastic flow.

	Compression force		
	Medium	High	Very high
Compression phase	+	+ + + +	+ + + + + +
Dwell phase	+ +	+	0
Decompression phase	−	− −	− − −
Total compression[a]	+ +	+ + +	+ + +

[a] The total compression is the sum of plastic flow and elastic recovery.

considered together with tablet strength, in-die bulk porosity and tablet porosity. Punch displacement data are not absolutely essential for proper interpretation of compression and stress relaxation phases, because the porosity data might provide sufficient information about irreversible densification.

For Starch 1500, the plastifying action of water is reflected by the parameters of the force-time curve which characterize the densification phase. The area ratio AR, the peak offset time t_{off}, the compression slope Sl_c and the compression area A_1, the last with some limitations, however, enable specific detection of the stress relaxation phase.

The area ratio is a specific and robust parameter enabling description of stress relaxation during dwell time on a rotary tablet press. Because it is a relative parameter, it is independent of the absolute signal height. When the punch pressure passes the yield pressure of the powder merely at the end of the compression phase, significant stress relaxation will be detected during dwell time. At punch pressures much higher than the yield pressure of the powder, stress relaxation occurs mainly during compression phase, hence the porosity limit is reached before the dwell phase begins. In this instance there is virtually no further plastic flow, but elastic compression below the porosity limit takes place during dwell time. This leads to a higher elastic recovery during decompression and to reduced tablet strength. The AR might therefore be used as a parameter for in-process control of plastically flowing formulations to prevent overpressing: As long as the AR reveals significant stress relaxation during dwell time, the formulation does not show excessive elastic recovery. Thus, reduced tablet strength should not be a problem. It should, however, be noted that generalized conclusions should not be drawn from this experiment, because the significance of the parameters can change with the subject studied and the experimental approach.

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