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Anomalous effect of compression pressure on the brittle fracture tendency of α-cellulose tablets

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Abstract

The effect of varying the compression pressure on the brittle fracture tendency of α-cellulose and lactose tablets has been investigated. Tablet tensile strength, (T), packing fraction, (P_f), and brittle fracture index (BFI) were determined at different compression pressures (0.82, 1.22 and 1.63 MPa). In another aspect of the study, α-cellulose and tapioca powders were mixed in various proportions to obtain powders of varying plastoelasticity. Their tableting characteristics T, P_f and BFI were also determined at the different compression pressures. The polymer, α-cellulose displayed the characteristics of plastic compression—a low BFI but high T and P_f values while lactose displayed the characteristics of elastic compression—a high BFI but low T and P_f values. The degree of plastic compression decreased as the proportion of tapioca in the powder mixture increased from 0 to 50% w/w. Higher concentrations of tapioca >50% produced crumbly tablets at all compression pressures, thus indicating that tapioca is highly elastic. Increase in the compression pressure caused an increase in the BFI values of lactose tablets, 0.39–0.76 but a decrease in the BFI values of α-cellulose tablets, 0.38–0.09. This decrease became less pronounced as the plasticity of the powder mixtures decreased. The difference in the response of the tablets to the change in compression pressure relates to the difference in the plastoelasticity of the materials tested.

Keywords: Compression pressure; Brittle fracture index; Plasto elasticity; Plastic and elastic compression; Stress relief; Crack resistance

1. Introduction

The parameters which determine the tableting characteristics of a pharmaceutical powder are the tensile strength (T), packing fraction (P_f), and the brittle fracture index (BFI) of resulting tablets (Hiestand et al., 1977; Itiola and Pipel, 1986). BFI is a measure of the tendency of the tablet to cap or laminate during decompression (i.e. withdrawal of the upper punch pressure). It is measured by comparing the tensile strength (T_o) of a tablet with a center hole with the tensile strength (T) of a similar tablet without a center hole. Thus (Hiestand

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et al., 1977);

$$\mathrm{BFI} = 0.5\left(\frac{T}{T_o} - 1\right) \quad (1)$$

The center hole is a built-in model defect, which simulates the actual voids formed in the tablets (due to air entrapment) during manufacture. The voids or low density regions in the tablet are weak points from which cracks emanate when stress (due to die wall) is applied on the tablet. Tablet formulations with BFI values ≥ 0.5 are prone to brittle fracture (Hiestand et al., 1977).

The term plastoelasticity refers to the relative elastic to the plastic compression property of a pharmaceutical powder and it is given by the expression ER/PC where ER is the elastic recovery (measured by the percentage increase in tablet thickness after ejection) and PC is the plastic compression (measured by the percentage decrease in tablet thickness while holding the maximum load for 30 s on the tablet in the die). A high ER/PC value therefore indicates that the powder is predominantly elastic (Okor, 1996). Several studies (Ejiofor et al., 1986; Itiola and Pipel, 1986; Esezobo and Pipel, 1987) showed that a high BFI value of tablets is invariably associated with a high plastoelasticity of powders from which the tablets were made. Hence BFI values have been used as measures of plastoelasticity (Okor, 1996; Okor et al., 1998; Eichie and Okor, 2002).

By the Hiestand theory, BFI should be a material constant ($P > 0.9$). However a previous study (Esezobo and Pipel, 1987) showed that an increase in compression pressure will increase the plastoelasticity of most pharmaceutical powders. Based on the relationship between plastoelasticity and BFI noted above, this finding therefore means that an increase in compression pressure is associated with increase in BFI of resulting tablets. More recently, Okor et al. (1998) showed that an increase in the mechanical strength of lactose tablets (by increasing the compression pressure) could be positively correlated to the increase in the BFI values of the tablets. Thus, the present knowledge is that excessive compression pressures aggravate the brittle fracture tendency of tablets. The present study reports on the anomalous effect of compression pressure on the brittle fracture tendency of α-cellulose tablets whereby an increase in the compression pressure ameliorated the fracture tendency against expectation.

2. Materials and methods

2.1. Materials

The α-cellulose used in this study was obtained by sodium hydroxide and sodium sulphite digestion of milled corn cobs (agricultural waste) details of the extraction procedure have been described elsewhere (Okhamafe et al., 1991). It is a white fibrous powder, which is plastic and can be compressed directly without preliminary granulation. Its physical parameters were determined to include a bulk density 0.42 g cm^{-3}, true particle density 1.52 g cm^{-3}, porosity 0.74. Tapioca powder was obtained by extraction from the peeled and rasped root of *manihot utilissima* (cassava) following the extraction procedure described previously by Eichie and Okor, (2002). It is the fibrous residue obtained after a high proportion (>90%) of starch has been removed from cassava roots (Trease and Evans, 1987). It is a highly elastic material and cannot be compacted even after granulation but can be rendered plastic by acid treatment (Eichie and Okor, 2002). Its physical characteristics were determined to include a bulk density 0.34 g cm^{-3}, true particle density 1.43 g cm^{-3}, porosity 0.36 (Eichie and Okor, 2002). The untreated samples were used in this study to vary the plasticity of α-cellulose samples by mixing the two powders in varying proportions (Table 1). The weight ratio (tapioca/α-cellulose) was taken as the index of

Table 1
Composition of the powder mixtures and their plastoelasticity index values

Proportion of each powder (%)		Tapioca/α-cellulose (index of plastoelasticity)
Tapioca powder	α-Cellulose powder	
100	0	α
80	20	4.00
70	30	2.33
60	40	1.50
50	50	1.00
40	60	0.67
30	70	0.43
20	80	0.25
0	100	0

Note: the weight ratio of tapioca (elastic material) to α-cellulose (plastic material) was taken as the plastoelasticity index of the powder mixture.

plastoelasticity of the powders. Both powders were sifted through a fine mesh sieve (aperture size, 212 μm) and dried at 50 °C to a moisture content ≤2.1% ± 0.2% w/w before use. Lactose was used in the granular form because the powder was not compactable without preliminary granulation. The granules were produced by wet-massing with 20% w/v starch mucilage. The dried granules had moisture content, 2.1 ± 0.3% w/w.

2.2. Methods

2.2.1. Tableting

A hydraulic press (Beckman 000–025, Scotland) was employed in the compaction. A sample of the powder or granules (500 mg) was placed in the die (diameter 12.5 mm) and compressed using flat faced punches at different compression pressures, (0.82, 1.22, and 1.63 MPa). Below 0.82 MPa crumbly tablets were formed and above the pressure 1.63 MPa the tablets were too hard and could not be fractured during the tensile strength tests. Hence, these pressures were considered the lower and upper limits. In each compaction the maximum pressure was held on the tablet for at least 30 s before releasing it, so as to allow time for consolidation. The punch and die surfaces were lubricated with a 1% dispersion of magnesium stearate in chloroform before compaction to facilitate tablet ejection.

To form tablets with center hole similar punches but with a center hole (upper punch) and a center pin (lower punch) were used (Roberts and Rowe, 1986; Itiola and Pipel, 1986). Earlier, Hiestand et al. (1977) used a retracting pin to produce tablets with centre hole. Tablets made by the present procedure were used in the determination of BFI values (Eq. (1)).

2.2.2. Determination of tablet tensile strength (T)

The load (P) needed to fracture the tablet was determined by diametric compression with a Monsanto hardness tester (Brook and Marshall, 1968). Ten tablets were used in each determination. T was calculated from the expression (Fell and Newton, 1970);

$$T = \frac{2P}{\pi Dt} \tag{2}$$

where t is the thickness and D the diameter of the tablet. The determination was carried out in triplicate, using different batches of tablets and mean results reported.

2.2.3. Determination of tablet brittle fracture index (BFI)

Tensile strengths of tablets with and without a center hole were determined as described above. The mean values were used to calculate BFI (Eq. (1)).

2.2.4. Determination of tablet packing fraction (P_f)

Thickness (t) and radius (r) of ten randomly selected tablets were determined using a digital micrometer. The tablets were weighed individually using an electronic balance and their mean weight (w) calculated. The apparent particle density (ρ) of each of the powder α-cellulose, varying mixture of α-cellulose and tapioca powder or lactose was determined by a fluid (liquid paraffin) displacement method using a specific gravity bottle (Sugita et al., 1995). Liquid paraffin was used because it is a non-solvent for the test powder. P_f values were obtained from the expression (Itiola and Pipel, 1986);

$$P_f = \frac{w}{\pi r^2 t \rho} \tag{3}$$

The determination was carried out in triplicate and mean values reported.

3. Results

3.1. Effect of compression pressure on the tableting characteristics of α-cellulose and lactose

The values of tableting parameters T, P_f, and BFI for α-cellulose and lactose tablets are presented in Table 2. The T and P_f values for α-cellulose tablets were generally high at all compression pressures while the BFI were low ≤0.38, thus displaying the characteristics of plastic compression (Itiola and Pipel, 1986, 1991). Lactose on the other hand displayed the characteristics of elastic compression—low T and P_f values and high BFI values ≥0.39.

Increase in compression pressure caused a general increase in the T and P_f values of the tablets. The change in BFI values however went opposite directions—a decrease in BFI in α-cellulose tablets but an increase in BFI in lactose tablets (Table 2).

Table 2
Effect of compression pressure on the tableting parameters (T, BFI and P_f) of α-cellulose powder and lactose granules

Compression pressure (MPa)	T (MNm^{-2})	T_0 (MNm^{-2})	BFI	P_f
α-Cellulose powder				
0.82	1.40	0.80	0.38	0.85
1.22	2.03	1.65	0.12	0.86
1.63	2.57	2.19	0.09	0.90
Lactose granules				
0.82	0.32	0.18	0.39	0.67
1.22	0.60	0.33	0.41	0.72
1.63	0.91	0.36	0.76	0.74

3.2. Effect of compression pressure on the tableting characteristics of the powder mixtures

Tapioca alone or its admixture with α-cellulose in concentrations >50% w/w were not compressible as they formed crumbly tablets even at the highest compression pressure, 1.63 MPa. Furthermore, the mixture consisting of α-cellulose and tapioca 50:50 were not compressible at the lowest pressure, 0.82 MPa except at the higher pressures. Powder mixtures with tapioca content ≤40% w/w formed tablets readily at all compression pressures. At a given pressure, T and P_f values of the tablets decreased while BFI values increased with increase in the plastoelasticity of the powder mixtures (Table 3). An increase in the compression pressure from the lowest to the highest level (i.e. 0.82–1.63 MPa) caused a decrease in BFI values to various degrees depending on the plastoelasticity of the powder mixture. The trend was that the decrease in BFI became less pronounced as the plasto elasticity of the powder increased. For instance the ratio BFI_1/BFI_2 (BFI_1 and BFI_2 are the BFI values obtained at the low and high pressures, respectively) decreased from 4.02 to 1.93 as the plastoelasticity index of the powders increased from 0 to 0.67 (Table 4). This means that the less plastic powder mixtures were less responsive to the change in compression pressure.

4. Discussion

Brittle fracture during tableting is a direct consequence of stress (due to die wall pressure) concentrating at the edge of voids or low density pockets which are weak points in the tablet and from which cracks emanate during decompression (i.e. withdrawal of the upper punch pressure). Plastic deformation of particles in the tablet can relieve the stress and thus ameliorate brittle fracture.

Accordingly, the effect of compression pressure on the BFI of the lactose tablets can be explained on the basis that tablets formed at the high pressure were too hard and therefore difficult to deform plastically (Okor et al., 1998). Another consideration is that the

Table 3
Effect of compression pressure on the tableting parameters T, T_o, P_f and BFI of the powder mixture

Compression pressure (MPa)	Plastoelasticity of powder mixture	T (MNm^{-2})	T_0 (MNm^{-2})	BFI	P_f
0.82	0	1.40	0.79	0.38	0.85
	0.25	1.28	0.71	0.40	0.72
	0.43	1.12	0.62	0.40	0.70
	0.67	0.98	0.48	0.52	0.69
	1.0	–	–	–	–
1.22	0	2.03	1.63	0.12	0.86
	0.25	1.88	1.42	0.16	0.78
	0.43	1.71	1.20	0.21	0.74
	0.67	1.16	0.63	0.42	0.72
	1.0	0.52	0.21	0.72	0.65
1.63	0	2.57	2.18	0.09	0.90
	0.25	2.28	1.81	0.13	0.80
	0.43	1.98	1.43	0.19	0.76
	0.67	1.35	0.88	0.27	0.74
	1.0	0.84	0.41	0.52	0.68

Note: formed crumbly tablets.

Table 4
Comparison of the BFI values at low (0.82 MPa) and at high (1.63 MPa) compression pressures for tablets of the powder mixtures

Plastoelasticity index	BFI values of tablets compressed at the pressures (MPa)		Ratio BFI (1) BFI (2)
	0.82	1.63	
1.0	–	0.52	–
0.67	0.52	0.27	1.93
0.43	0.40	0.19	2.11
0.25	0.40	0.13	3.08
0 (100% Plastic)	0.38	0.09	4.0

Note: formed crumbly tablets at this compression load.

plastoelasticity of the powder increased at the high pressure. It was not possible to measure the absolute values of plastoelasticity in this study due to technical constraints. However a previous study (Esezobo and Pipel, 1987) has shown that an increase in compression pressure causes an increase in the plastoelasticity of most pharmaceutical powders. The relevance of this consideration is that an increase in plastoelasticity implies a lesser capacity of the system for plastic deformation and hence aggravation of brittle fracture.

The same explanation cannot be extended to the case of α-cellulose tablets where the hardness of the tablet increased and therefore difficult to deform, yet the BFI values decreased with increase in compression pressure. The decrease in BFI probably resulted from an increase in the plastic compression of α-cellulose powder at the high pressure. Since α-cellulose is a plastic material (See its tableting characteristics in Table 2) an increase in compression pressure will be expected to cause a greater degree of plastic deformation of particles in the tablet and hence an increase in particle–particle bonding which in turn will increase the crack resistance of the tablets. The effect would be that even though stress concentrated at the edge of the center hole (model defect) cracks would not easily emanate from it. It is believed that the increase in crack resistance accounted for the decrease in BFI at the high compression pressure.

The decrease in BFI became less pronounced as the plastoelasticity of the powder mixtures increased (Table 4). This was so because the degree of plastic compression and hence the extent of crack resistance would also decrease in that order. Thus, the less the plasticity of a powder the less the degree of the anomalous effect of compression pressure on BFI.

5. Conclusion

The conclusion therefore is that the observed decrease in BFI at the high compression pressure (as in the case of α-cellulose tablet) is a characteristic of plastic compression while the observed increase in BFI (as in the case of lactose tablet) is a characteristic of elastic compression.

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