The tensile strength and consolidation of lactose coated with non-ionic surfactants II. Tablets

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Summary

The tensile strengths of tablets made from fine lactose powder coated with increasing amounts of non-ionic surfactants (Spans) have been measured using the diametral compression test, and their compression characteristics have been investigated. The tensile strengths of the tablets decreased, and their packing fractions increased at any compression pressure as the amount of the coating material was increased. The results have been analyzed using Cheng's equation for the tensile strength and the compression equation of Heckel. The terms in these equations varied systematically with the amounts of the surfactant in the powders used for tabletting.

Introduction

Measurements of the tensile strengths of compressed powders are widely used for providing basic information on the materials employed in the preparation of pharmaceutical tablets (Newton et al., 1971; Ridway et al., 1972).

In the preceding paper, an account was given of the results obtained when powdered lactose coated with increasing amounts of a series of non-ionic surfactants (Spans), was compressed by tapping or by the application of pressure to form relatively loosely packed beds. It was shown that, when the coating was monomolecular, both the tensile strength and the resistance of the powder to consolidation were decreased to minima and then increased when sufficient surfactant was present to form pendular bonds between the particles. It was shown that the packing fraction of the powder after 100 taps increased to a maximum and then decreased as the

amount of the coating material present was increased. Cheng's equation for the tensile strength was shown to apply to the coated powders.

Commercial tablets normally have packing fractions in excess of 0.75 and it was therefore considered desirable to see to what extent the changes produced by the surfactants on the mechanical properties of rather loosely packed beds would be reflected in the tensile strengths (as measured by the diametral compression test (York and Pilpel, 1973; Esezobo and Pilpel, 1976) of lactose tablets containing the same surfactants.

Besides measuring the mechanical strength of the tablets, an investigation has also been made of how the densities of the coated and the uncoated lactose powder varied with applied pressure. Many workers (Heckel, 1961; Cooper and Eaton, 1962; Hersey and Rees, 1970; York and Pilpel, 1973) have proposed that consolidation of powder takes place by the following mechanisms: (i) at a relatively low pressure, rearrangement of particles leads to closer packing, the applied energy being dissipated mainly in overcoming interparticle friction; (ii) at higher pressures, elastic or plastic deformation may occur, causing the particles to fill the voids and thus increase the area of interparticle contact. For materials of low thermal conductivity, and low melting points (e.g. Spans) the frictional heat generated may cause a rise in temperature, resulting in increased plasticity or even in melting (York and Pilpel, 1974); and (iii) brittle powders may fracture, and rearrangement of the fragments leads to an increase in packing fraction.

The mechanisms may occur sequentially or simultaneously. The order in which they occur depends on the properties of the materials, and the consolidating condition employed.

The aim in the present work has been to study the tensile strengths and compression behaviour of tablets made from coated and uncoated lactose.

Materials and methods

The materials, and coating procedure have already been described (part I).

Preparation of tablets

600 mg samples of lactose coated with different amounts of the surfactants were compressed under standard conditions (York, 1973) in a hand press to form 10.5 mm diameter flat-faced tablets. The packing fractions of the tablets were determined in quintuplicate from their measured weights and dimensions.

Tensile strength

The tensile strengths of the tablets were measured by diametral compression using a CT 400 tester (Engineering Systems, Nottingham), which works on the principles described by Esezobo and Pilpel (1976), and York and Pilpel (1973). The expression for the tensile strength of flat-faced tablets is given by (see York, 1973):

$$T = \frac{2P}{\pi D t_t} \tag{1}$$

where P = applied stress in MN; D = tablet diameter in cm; and t_t = tablet thickness in cm.

Results

Fig. 1 is representative of the way in which the tensile strengths of the tablets varied with their surfactant contents and packing fractions. The graphs were rectilinear, and in every case increasing the surfactant content caused a decrease in tensile strength.

Fig. 2 shows that, at a selected packing fraction of 0.85, increasing the amount of surfactant produced a decrease in the tensile strengths of the tablets (similar results were obtained at other packing fractions).

Cheng's expression for the tensile strength, T, of a powder bed or compact, and the method of using it for calculating the functions F and G have been given in part I.

Representative graphs of F versus G for tablets containing Span 60 are shown in Fig. 3. It is seen that, instead of the continuous straight lines usually obtained with these type of plots on loosely packed beds of powders (see part I; and York, 1973), there are abrupt changes in slope, and all the graphs show two distinct regions (see Esezobo and Pilpel, 1977).
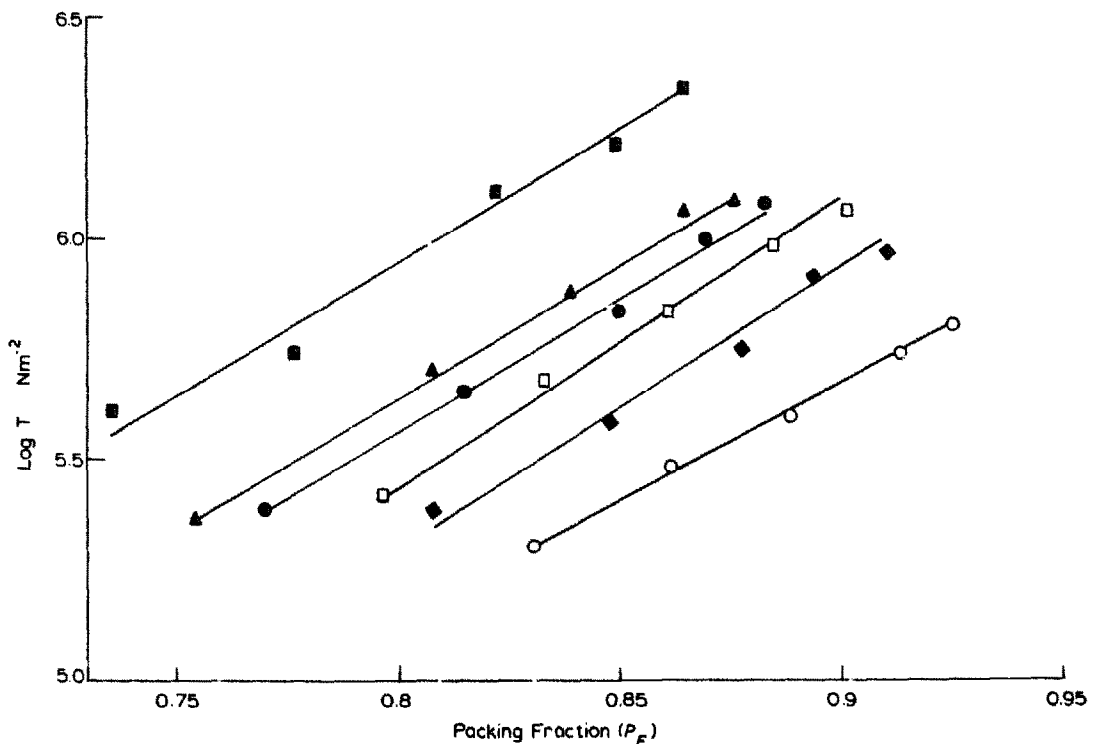

Fig. 1. Log tensile strength $MN \cdot m^{-2}$ vs packing fraction (p_f) for tablets of lactose coated with Span 85: ■, no surfactant; ▲, 0.5; ●, 1; □, 2; ◆, 3; and O, 10 (all $mol/g \times 10^5$).
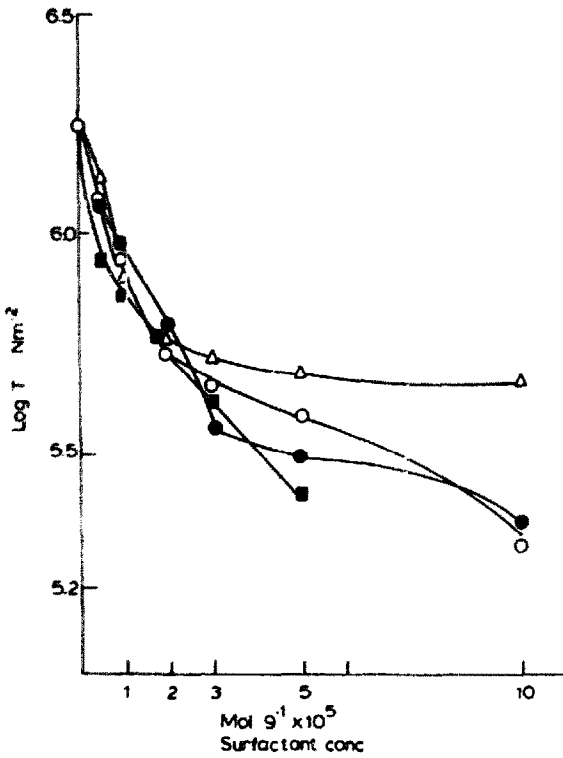
Fig. 2. Effect of surfactant concentrations on the tensile strengths of lactose tablets: ●, Span 20; △, Span 60; ○, Span 80 and ■, Span 85.
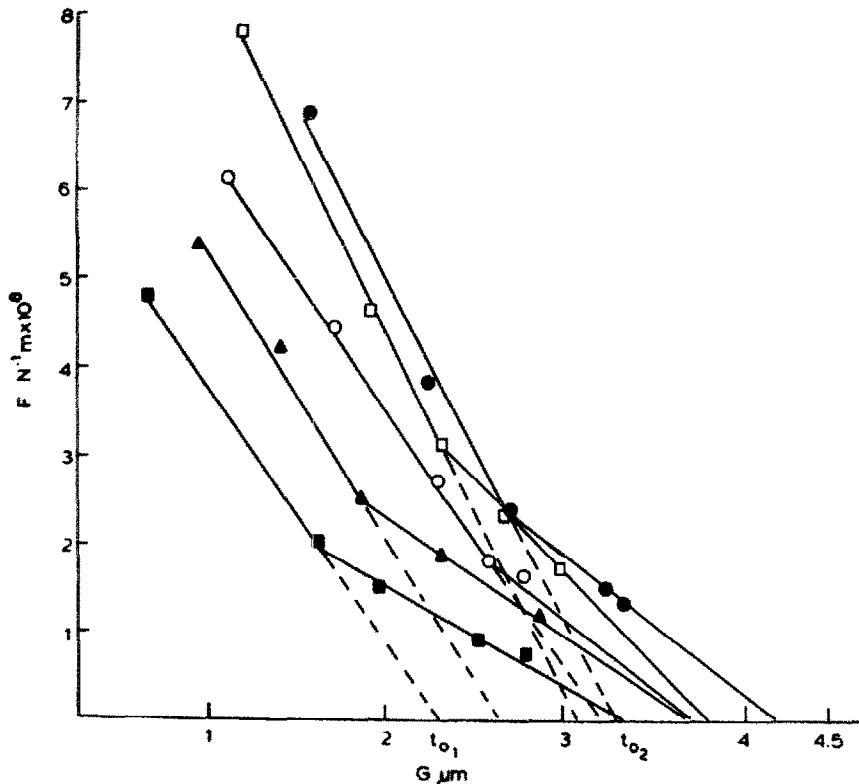

Fig. 3. Relation between $F = (1/2 \, s/v \cdot \rho_f/T)$ and $G = \overline{d}/3(\rho_f/P-1)$ at concentrations of ■, 0.5; ▲, 1; ○, 2; □, 5; and ●, 10 (all in $mol/g \times 10^5$ Span 60).
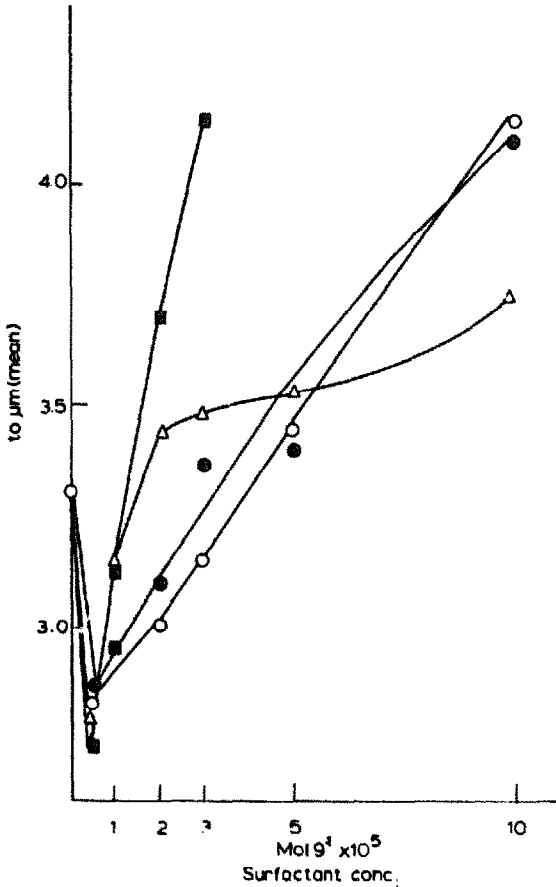
Fig. 4. $t_{0(mean)}$ versus surfactant contents. The symbols: ●, Span 20; △, Span 60; ○, Span 80; and ■, Span 85.

In order to obtain the values of the average interparticle forces, t_0, the two sections of each graph were extrapolated independently to F equals zero, and $t_{0(mean)}$ was calculated as:

$$t_{0(mean)} = (t_{0_1} + t_{0_2})/2 \qquad (2)$$

Values for $t_{0(mean)}$ are given in Table 1, and are plotted versus surfactant contents in Fig. 4.

The interparticle separations, t, at a packing fraction of 0.85, were obtained from $t_{0(mean)}$ by substituting into:

$$t = t_{0(mean)} - \frac{\overline{d}}{3}\left(\frac{\rho_f}{\rho_{f_0}} - 1\right) \qquad (3)$$

Values for the energy of the particle surfaces, Σ_0 (Cheng, 1968, 1970), were calculated using Eqn. 11 in part I. The values of t and of Σ_0 initially decrease and then increase with increasing surfactant content, while t increases uniformly.

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TABLE I

DATA OBTAINED FROM CHENG'S EQUATION AND THE COMPRESSION PLOTS OF HECKEL

Surfactant	Concentration $(mol \cdot g^{-1} \times 10^5)$	t_0 (μm)	t (μm) (at ρ_f = 0.85)	Σ_0 (at ρ_f = 0.85)	$K \times 10^4$ $((MN \cdot m^{-2})^{-1})$	A	ρ_f at P = 100 $MN \cdot m^{-2}$
None	None	3.30	0.19	1.00	47	0.05	0.833
Span 20	0.5	2.87	0.51	0.85	50	1.15	0.836
	1	2.95	0.59	1.33	50	1.17	0.849
	2	3.10	0.74	2.33	52	1.22	0.850
	3	3.37	1.01	5.21	57	1.38	0.860
	5	3.40	1.04	6.15	62	1.47	0.871
	10	4.10	1.70	15.23	71	1.53	0.895
Span 60	0.5	2.80	0.44	0.77	50	1.06	0.836
	1	3.15	0.79	1.86	–	–	–
	2	3.45	1.09	3.30	50	1.17	0.850
	3	3.48	1.11	3.85	50	1.17	0.854
	5	3.50	1.16	4.31	60	1.36	0.859
	10	3.75	1.39	5.58	72	1.46	0.883
Span 80	0.5	2.82	0.46	0.94	–	–	–
	1	2.95	0.59	1.42	55	1.14	0.842
	2	3.00	0.64	2.48	57	1.20	0.847
	3	3.15	0.79	3.46	66	1.42	0.864
	5	3.45	1.09	5.34	69	1.47	0.876
	10	4.17	1.81	17.98	77	1.53	0.892
Span 85	0.5	2.73	0.37	1.15	54	1.17	0.845
	1	3.53	1.17	2.98	61	1.24	0.855
	2	3.70	1.34	4.98	64	1.41	0.871
	3	4.13	1.77	8.33	68	1.47	0.882
	5	–	–	–	75	1.57	0.896

The pressure density relationships for the various samples were next analyzed using the Heckel (1961) equation (an attempt to employ the Cooper and Eaton (1962) equation proved inconclusive). The Heckel equation is:

$$\ln\left(\frac{1}{1 - \rho_f}\right) = KP - A \tag{4}$$

where ρ_f is the packing fraction of the tablet, and P is the applied pressure, the constants K and A are determined from the slope and intercept respectively of the extrapolated linear portion of the plot of $\ln(1/1 - \rho_f)$ versus P.

Fig. 5 shows representative Heckel plots for lactose coated with different amounts of surfactant, while the values of K and A are listed in Table 1.

Finally Fig. 6 shows plots of packing fractions achieved by a selected compression pressure of 100 MN m^{-2} obtained from the linear portions of the Heckel plots in
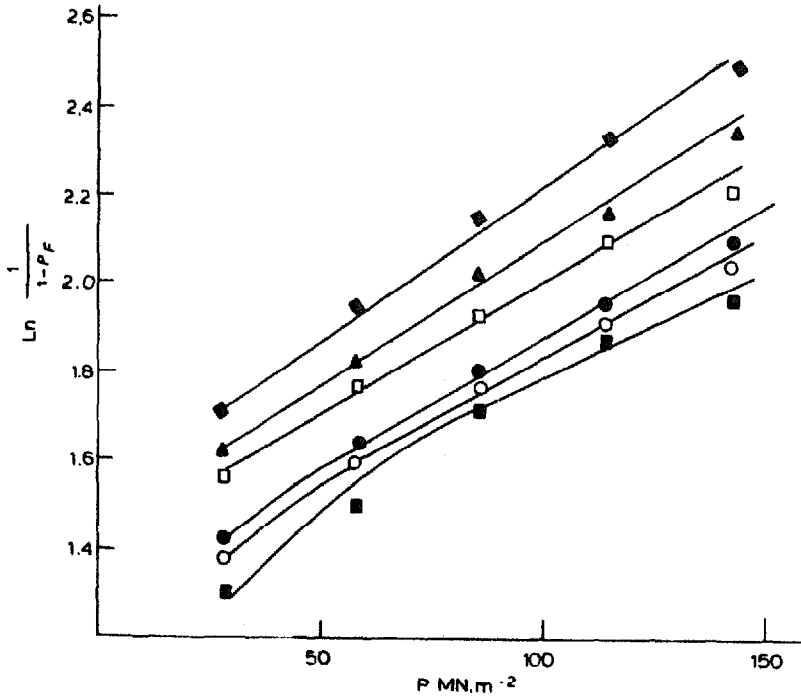
Fig. 5. ln $1/1 - \rho_f$ versus P at different surfactant contents. ■, no surfactant; ○, 1; ●, 2; □, 3; ▲, 5; and ◆, 10 (all mol/g × 10^5 of Span 80).

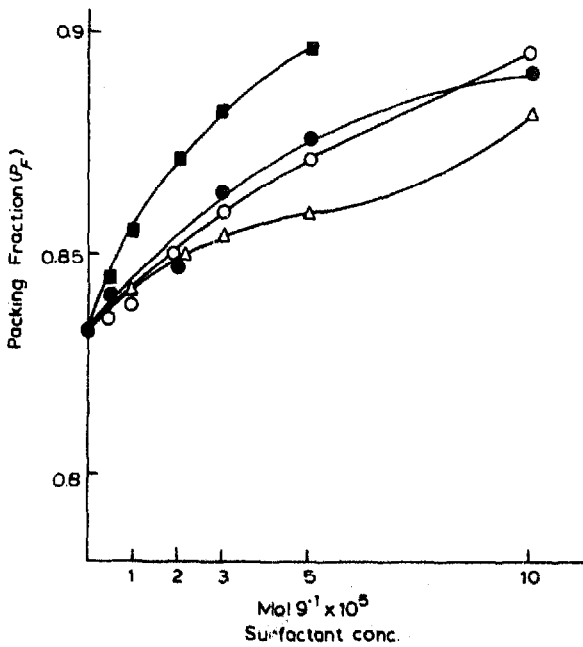Fig. 6. ρ_f achieved by a compressive force of 100 MN m^{-2} versus surfactant content. ●, Span 20; △, Span 60; ○, Span 80; and ■, Span 85.

Fig. 5 versus the amounts of the surfactants employed. It is seen that the packing fractions produced by a fixed pressure increased with increasing surfactant content.

Discussion

It can be seen that, at any particular packing fraction above 0.7 (Figs. 1 and 2), the tensile strengths of the compressed samples decrease with their surfactant content. This is in contrast to the results obtained at a lower packing fraction of 0.54 (part I).

At relatively low packing fractions, the particles of the powder bed are relatively loose and mainly held together by pendular, funicular or capillary bonds of surfactant, whose contribution to the tensile strength of the bed can be expressed in quantitative terms (Rumpf, 1962; Derjaguin, 1961). However, at higher packing fractions the surfactants begin to act as lubricants or dispersing media, smoothing and softening the surfaces of the particles, preventing them from interlocking with each other, also decreasing the strength of the solid bonds between them after their plastic deformation, and hence reducing the tensile strengths of the tablets.

It is seen from the positions of the graphs in Fig. 2 that the decrease in tensile strength produced by Span 60 at concentrations over 2×10^{-5} g/mol is smaller than those produced by the other surfactants (Span 20, 80 and 85), indicating that tablets containing solid coatings are stronger than those containing liquid coatings.

On the basis of Cheng's equation, it is seen from Figs. 3 and 4 and Table 1, that the average interparticle forces $t_{0_{(mean)}}$, and the surface energies, Σ_0, initially decreased at low concentrations of the surfactants, but then started to increase. This behaviour can be attributed to a reduction in the Van der Waal's forces between the lactose particles at low concentrations of surfactant. However, once a monolayer has formed, further addition of surfactant leads to the development of enhanced surface tension, and mechanical binding forces between the particles even though the distance between them (interparticle separation, t), increases as seen in column 4, Table 1.

Referring to the compaction behaviour of the samples, the graphs in Fig. 5 for tablets containing different amounts of Span 60 have an initial curved portion followed by a rectilinear portion up to a surfactant concentration of 2×10^{-5} g/mol. but at higher surfactant concentrations the curved portion disappears. The curved portions represent particle rearrangement (i), and the rectilinear portions, plastic or elastic deformation and fragmentation of the particles (ii, iii). It appears that at surfactant concentrations above about 2×10^{-5} g/mol, particle rearrangement is being inhibited.

The quantity A derived from the Heckel equation represents the degree of packing achieved at low pressures as a result of particle rearrangement. The second constant, K, depends on the material involved. For example, Heckel (1961) showed that soft ductile powders have higher K values than hard powders. Similarly, oxides within powder particles and in the form of surface films reduce the values of K, and high values indicate the onset of plastic deformation at relatively low pressure.

It is seen from Table 1 that increasing the surfactant contents resulted in increases in the values of both A and K indicating softness and greater plasticity of particles at higher pressures. This would account for the increased packing fractions of the tablets at a selected compressive force of 100 $MN \cdot m^{-2}$ in the presence of the surfactants (Fig. 6). The effect is least when the coating is solid, i.e. Span 60.

Conclusions

The tensile strengths of lactose tablets decrease when the powder is coated with increasing amounts of non-ionic surfactants.

The values of t from the Cheng's equation increase, while those of $t_{0_{(mean)}}$ and Σ_0 initially decrease then increase as the surfactant content is increased.

On the basis of the Heckel equation, lactose powder coated with non-ionic surfactants is softer and more readily compressible than the uncoated powder. However, the effect is least when a solid coating of Span 60 is employed.

Acknowledgement

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