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Some aspects of the failure of sucrose tablets

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The effects of compression force, amount of magnesium stearate, carbowax and talc on the failure characteristic of tablets of sucrose granulation with gum acacia as binder have been examined. The results show that breakage of the tablets during ejection is accompanied by high ejection forces and low ratio of lower to upper punch force. Tablets with an upper bevelled edge capped at high applied forces unlike those prepared with a flat upper face. This may be due to high radial forces in the bevelled top at the end of the compression cycle, caused by the bevelled edge, resulting in break-down of interparticulate bonds formed during compression. At very high compression forces all tablets showed lamination because of axial expansion.

Sucrose is widely used in buccal and sublingual tablet formulations which are usually designed to release the active ingredient over 15 to 30 min. To obtain slow dissolution, these tablets generally contain large proportions of water soluble filler(s), such as powdered sucrose, and water soluble binder(s), such as gum acacia, and are compressed at high pressures to give high crushing strength (hardness).

One of the difficulties encountered in the compression of sucrose tablets, especially at high pressure, is capping, lamination or breakage of the tablet. Ritschel (1966) has listed many and various possible causes of capping, Train (1956), Long (1960) and Führer (1966) explain capping as a result of relaxation of elastic strains in the tablet. Long & Alderton (1960) demonstrated that trapped gas could produce cracks. Shotton & Ganderton (1961) explain capping by the mechanism of failure under recovery stress because of strong interparticulate bonds. Führer (1966) mentions formation of a crystallographic unstable mass during expansion of the compact, resulting in cracks.

We have examined the effects of compression force, type of punchface, and type and amounts of some glidants and lubricants on the compression characteristics of a sucrose - gum acacia granulation. Special attention has been paid to the failure phenomena.

MATERIALS AND METHODS

The sucrose used was saccharum pulv. Ned. Pharm. grade from the Amsterdam Chemie Farmacie (ACF) Holland. Of the sucrose 90% passed a 40 mesh sieve, 75% passed a 140 mesh sieve and 50% passed a 200 mesh sieve. The magnesium stearate and polyethylene glycol 4000 were supplied by ACF and were Ned. Pharm. grade. The talc and gum acacia were Ned. Pharm. grade from Reese & Beintema, Holland. The specific surface of the glidants and lubricants was 13 m^2/g for magnesium stearate, 3 m^2/g for talc and 0·1 m^2/g for carbowax 4000.

The sucrose granulation was prepared by mixing in a planetary mixer 1140 g sucrose with a solution of 30 g gum acacia in 60 g water, passing the mass through an oscillating granulator (Frewitt; 2 mm screen), drying for 30 min at 50° in a tray drier, and screening the granulation through a 0·7 mm screen (Frewitt). The glidants or lubricants (or both) were added by blending for 10 min in a 2 litre cubic tumbling mixer. The sucrose tablets were made in a power driven eccentric tablet machine (HOKO–KJ) operating at 15 cycles/min with automatic die filling. The punches were plane-faced, or plane-faced bevel-edged punches of 9 mm. The tablet machine was instrumented with strain gauges to measure upper and lower punch forces in a manner similar to that of Shotton & Ganderton (1960). The strain gauges were bonded, according to De Blaey & Polderman (1970), to small load cylinders, axially located inside the guide blocks of the punches to measure high responses, especially of the ejection forces. The displacement of the punches was measured with inductive displacement transducers (Philips type PR 9309). The forces and displacements were recorded on a four channel Mingograf 3440. Force against displacement curves were registered on an oscilloscope with a camera. Percentage of the total energy input recovered as elastic energy was calculated from the force displacement curves.

The crushing strength of the tablets was determined using a motorized Heberlein instrument. The mean rate of loading of this instrument is about 5 kg s^{-1} according to Rees, Hersey & Cole (1970).

RESULTS AND DISCUSSION

Fig. 1 shows the crushing strength of tablets compressed at different upper punch forces from sucrose granulation with 1·5% talc and 0·6% magnesium stearate. In each case the result given is the mean of ten determinations. The tablets were made either with flat-faced, bevel-edged upper and lower punches or a bevel-edged lower punch and a flat-faced upper punch respectively. To check the reproducibility of the granulation two batches were tested.

Fig. 1 also shows the effect of bevelled edges on the strength and capping behaviour of the tablets. Up to a compression force of about 2100 kg there is no difference in strength between the tablets with flat-faces and those with bevelled edges. At higher forces the bevel-edged compacts all show separation (capping) of the top of the tablet at the bevelled edge, the resulting tablets having lower strength than the tablets with a flat upper face. At the highest upper punch force of about 4300 kg all tablets show lamination.

To exclude the effect of a bevel-edged upper punch on the capping tendency of the tablets, all other compression experiments were made with a flat-faced upper punch and a flat-faced, bevel-edged lower punch.

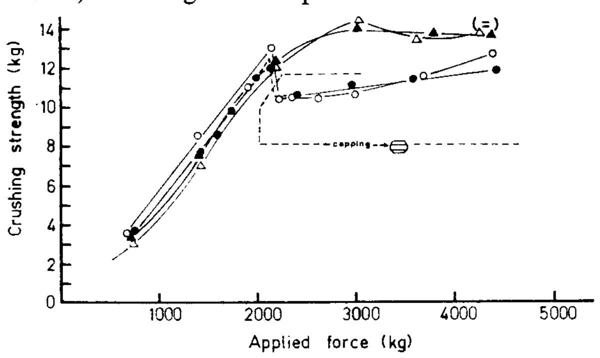

FIG. 1. Effect of bevelled edges on top of the tablets on the strength and capping tendency of sucrose compacts (1·5% talc + 0·6% magnesium stearate). Punch size 9 mm. Each coordinate represents the mean of 10 determinations. Two granulation patches (○, △ and ●, ▲) were compressed either with an upper punch having a flat face (△, ▲) or an upper punch with a flat face and a bevelled edge (○, ●).

Fig. 2 (I–III) shows the influence of carbowax 4000 on the compression of a sucrose granulation with 1·5% talc. The compression behaviour is represented by the relation between upper punch force and the ratio (R) of lower to upper punch force, the ejection force, and the strength of the tablets respectively. The ratio (R) of lower to upper punch force increases with upper punch force, except the series with 0·3% carbowax (see later), and increases with amount of carbowax (Fig. 2I). The ejection force increases with upper punch force, and decreases with increasing

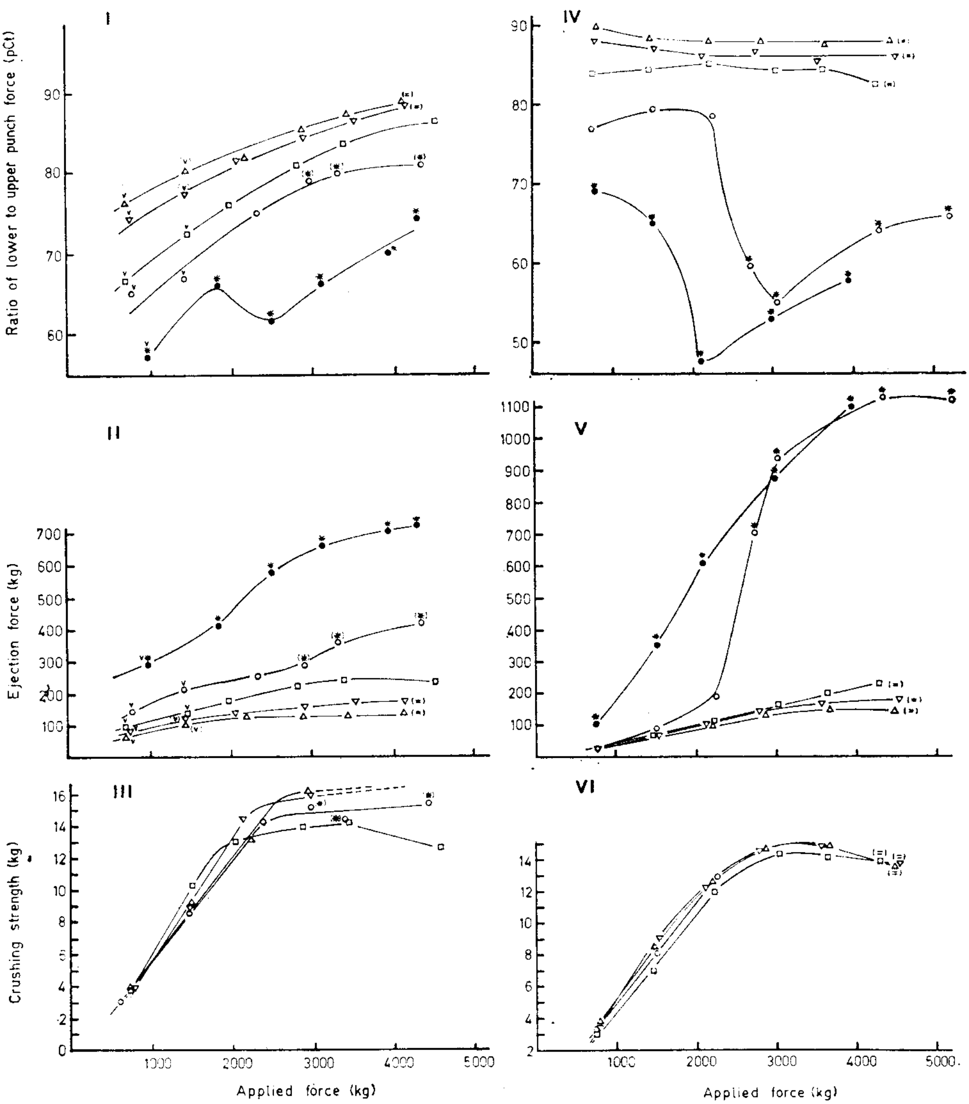

FIG. 2I–III. Effect of percentage carbowax 4000 on the relation between applied compaction force and ratio of lower to upper punch force (I), ejection force (II) and crushing strength (III) for sucrose tablets with 1·5% talc. Carbowax (%) ●, 0·3; ○, 0·5; □, 1·0; △, 1·5; ▽, 2·0.

IV–VI. Effect of percentage magnesium stearate on the relation between applied compaction force and ratio of lower to upper punch (IV), ejection force (V), and crushing strength (VI), for sucrose tablets with 1·5% talc. Magnesium stearate (%): ●, 0·2; ○, 0·4; □, 0·6; ▽, 0·8; △, 1·0. Failure characteristic: ↓ capping; = lamination; * breakage; v sticking; ( ) some of tablets show failure.

amount of carbowax (Fig. 2II). The strength of the tablets increases with upper punch force. The amount of carbowax shows little effect on the strength (Fig. 2III). Most characteristic is however the failure of the tablets. The tablets compressed from the granulation with 0·3% carbowax all broke during ejection. These compactions are marked by low R-values (Fig. 2I) and high ejection forces (Fig. 2II). The tablets with 0·5% carbowax show higher R-values and much lower ejection forces, did not break at low compression forces and showed only a slight tendency to break at higher forces. The tablets with 1·0, 1·5 and 2·0% carbowax showed no failure, except a slight tendency to laminate at the highest compression force. At 4000 kg compression force, 35% of the total energy input is recovered as elastic energy. It would seem that the lamination at high compression forces is due to strong axial relaxation of the compact. All compacts showed some sticking to the punches at low compression forces.

The influence of magnesium stearate on the compaction of sucrose granulation with 1·5% talc, is represented in Fig. 2IV–VI. These results better illustrate that breakage of the tablets during ejection coincides with very high ejection forces, as would be expected (Fig. 2V). The ratio of lower to upper punch force shows for the broken tablets a characteristic decrease and subsequent increase with compaction force (Fig. 2IV). Compressions with 0·1% magnesium stearate and lower were not possible, because of extremely high ejection forces.

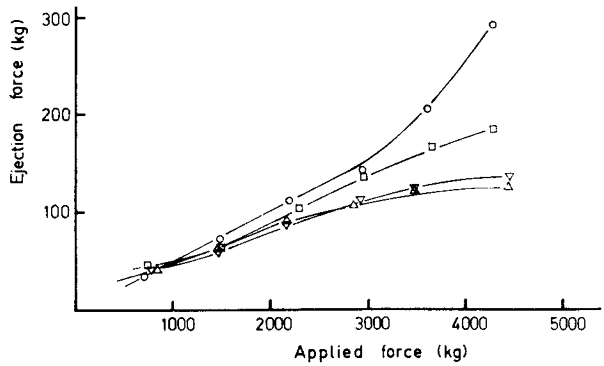

FIG. 3. Effect of talc on the lubricating action of magnesium stearate (0·6%) during ejection. Talc (%): ○, 1·5; □, 1·0; ▽, 0·5; △, 0·0.

The lubricating action of magnesium stearate, represented by the ejection force, increases with a decreasing amount of talc (Fig. 3) which masks the lubricating action of magnesium sterate. This finding supports the conclusion of Lewis & Shotton (1965), that talc is an inefficient lubricant. Consequently, by decreasing the amount of talc, it could be expected that breakage of the tablets containing 0·4% magnesium stearate would be eliminated. The results (Fig. 4) for the compacts without talc showed that breakage did not occur over the entire compression range, and for the compactions with 0·5% talc breakage only occurred at the highest applied force. The punch force ratio for the compressions with 1·0 and 1·5% talc shows the phenomenon of a *minimum*. The decrease in punch force ratio with increasing compression force would be consistent with the findings of an increase of shear strength of talc under a constraining load (Train & Hersey, 1960). The increase in punch force ratio at higher forces may be due to the approach of a maximum die wall interface. The standard deviation for the weight of ten tablets was not influenced by the amount of

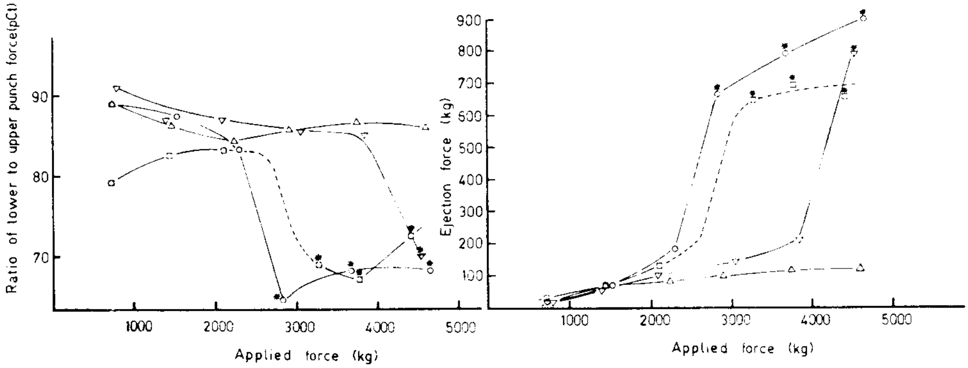

FIG. 4. Elimination of the breakage of the tablets during ejection by decreasing the amount of talc. Percentage magnesium stearate 0·4%. Talc (%): ○, 1·5; □, 1·0; ▽, 0·5; △, 0·0. Failure: * breakage of the tablets.

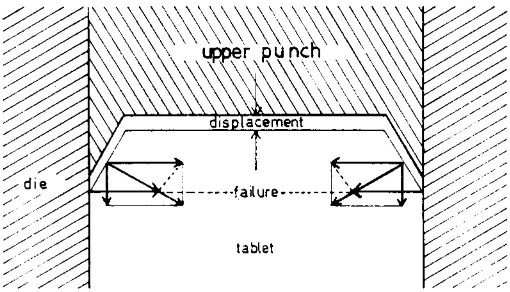

FIG. 5. Capping of tablet compressed with bevel-edged upper punch.

talc. This indicates that magnesium stearate is as good a glidant as the talc.

The observed compression characteristics of the sucrose granulation under examination indicate three types of tablet failure: breakage during ejection cycle because of high ejection forces, capping at high compaction forces caused by bevel-edged upper punches, and lamination at extreme high compression forces because of strong axial relaxation of the tablet. It is not known if these laminations are present after removal of the applied compression force and before ejection of the tablet.

An explanation for the capping at high compression forces of tablets with bevelled edges on the upper side may be that, at the bevelled top of the tablet, interparticulate bonds formed during compression are broken down at the end of the compression cycle by high radical forces in the bevelled top caused by the displacement of the bevelled edge on the upper punch (see Fig. 5).

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