J. Pharm. Pharmacol., 1965, 17, Suppl., 71S–81S Received April 28, 1965

Some studies of friction and lubrication using an instrumented tablet machine†

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Samples of crystalline aspirin, hexamine, and sodium chloride, and a granulation of hexamine were compressed on an instrumented tablet machine. Further samples of these materials, together with samples of sucrose and sucrose granulation, were lubricated with 2% magnesium stearate −100 mesh powder and compacted under similar conditions. The effect of magnesium stearate on die wall friction depends on the ease of deformation of the base particles. For the materials used there is a linear relationship of the form $F_d = k_d(P_m.A)$ and a linear relationship exists between F_e and F_d up to the point where F_e becomes constant. Granulations required larger ejection forces at a given pressure than the corresponding crystalline material, particularly when the materials were lubricated; constant values of F_e were associated with compaction pressures at which the density of the ejected tablets became maximal. Values of F_e for tablets of unlubricated sodium chloride and hexamine granulation depend on particle size; the size of other materials used caused no such effect. Lubrication eliminated this size effect.

WHEN materials are compacted in dies to form tablets by movement of the upper punch only, frictional effects at the die wall cause the force transmitted to the stationary lower punch to be less than the force applied at the upper punch. If both punches move during the consolidation stage, as in rotary tablet machines, the system is equivalent to two single-ended pressings.

Instrumented eccentric tablet machines have been used to investigate force transmission during tabletting (Higuchi, Nelson & Busse, 1954; Markowski, 1958; Hasegawa, 1959; Shotton & Ganderton, 1960; Riad & Zobel, 1962; Fuhrer, 1962) and a rotary machine instrumented with resistance strain gauges on the punches has been reported (Shotton, Deer & Ganderton, 1963). However, most of the recorded work is concerned with the behaviour of pharmaceutical granulations (Nelson, Naqvi, Busse & Higuchi, 1954; Nelson, Busse & Higuchi, 1955; Nelson, 1955; Strickland, Nelson, Busse & Higuchi, 1956; Markowski, 1958; Strickland, Higuchi & Busse, 1960). With the advent of forced-feed devices on tablet machines and the possibility in certain cases of eliminating the granulation process, more information is needed about the behaviour of simple crystalline and powder systems.

Experimental

30–40 mesh samples of aspirin, hexamine crystals, and hexamine granulation, and 40–60 mesh sodium chloride crystals were compacted at mean compaction pressures ranging from 400–2,200 kg/cm². An

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† This work formed part of a thesis accepted for the degree of Ph.D. in the University of London.

instrumented single punch eccentric tablet machine, incorporating a ½ inch plane faced punch and die set, was used (Shotton & Ganderton, 1960). Further samples of these materials lubricated with 2% magnesium stearate (−100 mesh powder) were compacted over this same pressure range. These experiments were repeated with similarly lubricated samples of 30–40 mesh sucrose and sucrose granulation.

The granulations were prepared from −100 mesh powder using only distilled water as an adhesive agent (Shotton & Lewis, 1964). It was not possible to tablet sucrose in any form in the absence of a lubricant.

Samples of sodium chloride, hexamine, hexamine granulation, and aspirin, varying in size from 20–30 mesh to 80–100 mesh were compressed both unlubricated and when lubricated with 2% magnesium stearate −100 mesh powder. Lubricated samples of sucrose and sucrose granulation were also compressed. For any one material the mean compaction pressure was constant, but the pressure level chosen varied with the nature of the base material.

Sample weights tabletted were calculated to give a tablet 0·4 cm thick at zero porosity.

Results and discussion

All results represent the mean of five tablets. From the experimental measurements values were calculated for mean compaction pressure, P_m (the average of the top and bottom punch pressures), ejection force, F_e, force lost to die wall, F_d (difference between upper punch force and lower punch force), and the ratio of lower punch force to upper punch force, R. The relative density, ρ_R, of the ejected tablets was calculated from their weight and dimensions.

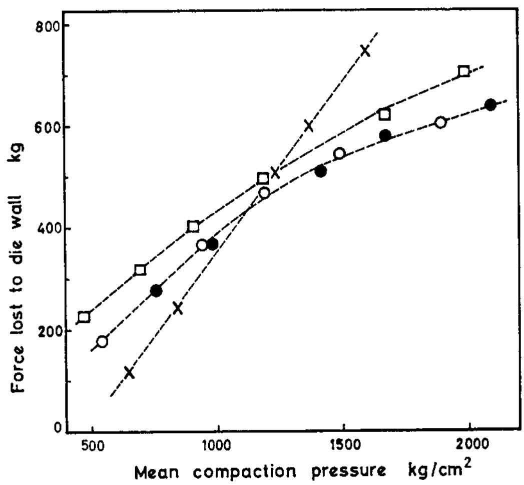

FIG. 1. Relationship between mean compaction pressure and force lost to die walls: unlubricated materials. × Sodium chloride. □ Aspirin. ● Hexamine. ○ Hexamine granulation.

EFFECT OF MEAN COMPACTION PRESSURE

Force lost to die wall. A linear relationship was found between P_m and F_d for unlubricated sodium chloride, but results for aspirin, hexamine, and hexamine granulation are best represented by curves (Fig. 1). The results for hexamine granulation follow closely those for hexamine crystals.

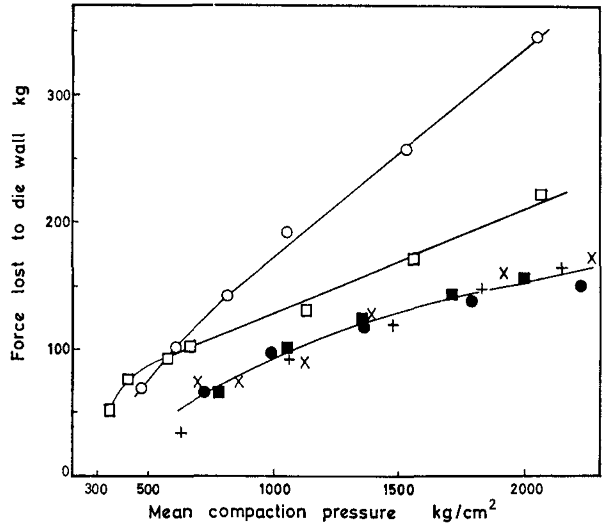

FIG. 2. Relationship between mean compaction pressure and force lost to die walls: materials lubricated with 2% magnesium stearate. × Sodium chloride. □ Aspirin. ● Hexamine. ○ Hexamine granulation. ■ Sucrose. + Sucrose granulation.

The presence of lubricant causes a marked reduction in values of F_d at any one pressure level (Fig. 2); also for any given increment of P_m, the increase in F_d is very much less for the lubricated materials. Results for lubricated samples of sucrose, sucrose granulation, hexamine, and sodium chloride can be represented by a single line, whilst lubricated aspirin and hexamine granulation show quite a different relationship. These latter materials seem to maintain a greater influence on die wall friction during consolidation.

Train (1956) quotes the results of Nelson & others (1954) in support of his own work which indicated an empirical relationship for ejection force,

$$F_e = c_1 (P_a.A)^n \quad \ldots \quad (1)$$

where P_a = applied pressure; A = apparent area of die wall/compact contact; n = a constant, a property of the material; c_1 = a constant, a property of the surface condition of the walls. He suggested a possible linear relationship between the ejection force and the maximum friction at the die wall during compaction.

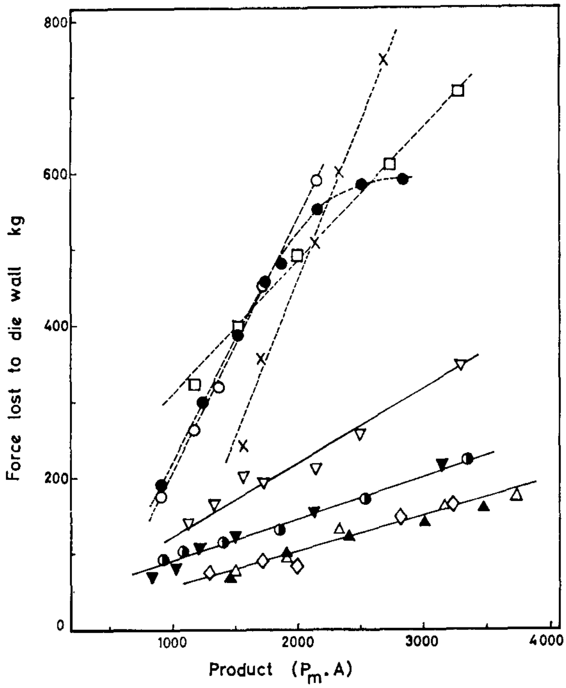

FIG. 3. Relationship between force lost to die wall and the product (P_m.A). × Sodium chloride, unlubricated. △ Sodium chloride, lubricated. ● Hexamine, unlubricated. ▼ Hexamine, lubricated. ○ Hexamine granulation, unlubricated. ▽ Hexamine granulation, lubricated. □ Aspirin, unlubricated. ◑ Aspirin, lubricated. ◇ Sucrose granulation, lubricated. ▲ Sucrose, lubricated.

If such a relationship exists

$$F_d = c_2 (P_a A)^n \quad \text{.. .. .. ..} \quad (2)$$

Using P_m instead of P_a and calculating values of A ($= \pi Dl$) from the length (l) of the ejected tablet it is found (Fig. 3) that there is a simple relationship between F_d and the product (P_m.A), and that n has a value of unity, i.e.

$$F_d = k_d (P_m.A) \quad \text{.. .. .. ..} \quad (3)$$

This relationship is demonstrated by all materials, with the exception only of unlubricated hexamine, over the complete range of compaction pressures studied. Values of F_d for unlubricated hexamine tend to a maximum value at 600 kg, but there is a linear relationship below this value.

The slope of the line, k_d, for unlubricated materials varies with the type of material compacted.

e.g.

	Slope, k_d
Sodium chloride	2·44
Aspirin	1·06
Hexamine crystal and granulation	2·00

When lubricated with magnesium stearate the base materials fall into 3 groups. Group 1: sucrose, sucrose granulation and sodium chloride. Group 2: hexamine and aspirin. Group 3: hexamine granulation.

The slopes of the lines for group 1 and group 2 materials are almost equal, i.e. 0·30 and 0·32 respectively. If the term k_d is a function of the surface condition of the die (cf. Train's constant c_1) it is apparent that the magnesium stearate determines the surface condition to the same extent for all the lubricated materials except hexamine granulation.

Force necessary for ejection, F_e. For tablets of unlubricated hexamine, hexamine granulation, and aspirin, a linear relationship exists between F_e and values of P_m up to 1,200 kg/cm² (Fig. 4). At greater values of P_m for these materials F_e becomes constant. Sodium chloride shows no maximum value of F_e over the range of pressures studied.

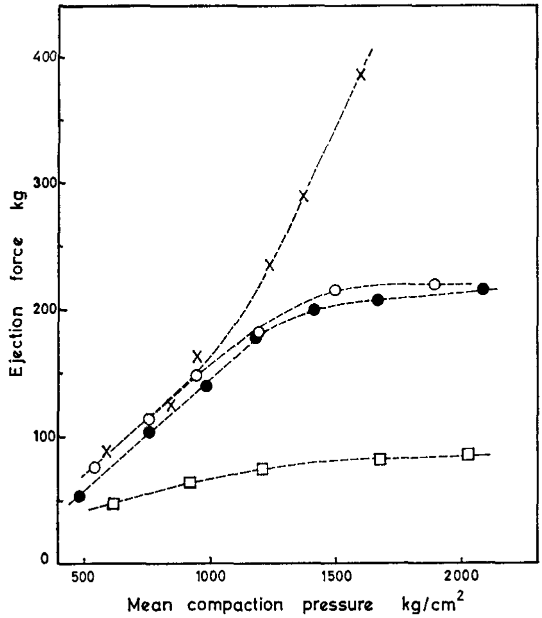

FIG. 4. Effect of mean compaction pressure on ejection force: unlubricated materials × Sodium chloride. ○ Hexamine granulation. ● Hexamine. □ Aspirin.

The magnesium stearate caused a marked reduction in F_e for all materials (Fig. 5). Values of ejection force for tablets of lubricated hexamine and hexamine granulation also became constant at pressures greater than 1,200 kg/cm², but values of F_e for lubricated aspirin tablets reached a constant value at a lower compaction pressure than previously. Ejection forces for lubricated sodium chloride tablets approach a limiting value at the maximum compaction pressure used, and these results are followed very closely by those for lubricated sucrose. There was no evidence of a maximum F_e value when lubricated sucrose granulation was compressed.

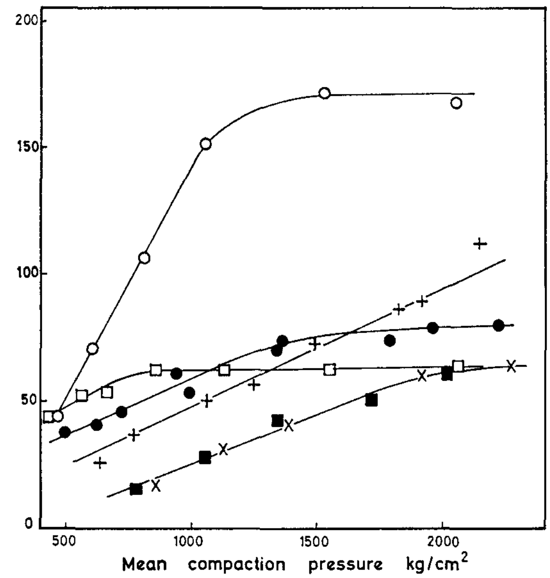

FIG. 5. Effect of mean compaction pressure on ejection force: lubricated materials. × Sodium chloride. ○ Hexamine granulation. ● Hexamine. □ Aspirin. ■ Sucrose. + Sucrose granulation.

Tablets made from the lubricated granulations required greater ejection forces at any one compaction pressure than the respective crystalline materials. Sucrose and sucrose granulation exhibit such large frictional forces that it was not possible to tablet the unlubricated materials, even at low pressures, because the machine jammed at the ejection stage. Both crystalline sucrose and sodium chloride require large ejection forces when unlubricated, yet when lubricated produced the smallest ejection forces over the whole range of compaction pressures.

A linear relationship between F_e and P_m was also found for tablets of lubricated materials until the limiting value of F_e was attained (Fig. 5). With the exception of hexamine granulation the linear portions of the graphs for lubricated materials are almost parallel to each other. In this case it is suggested that in the presence of a lubricant the "frictional condition" of the die surface is the same regardless of the base material, and that in this situation the observed differences in magnitude of F_e, at any one pressure, are dependent on the individual base materials and the residual radial stresses present in the tablet when the compacting force is removed before ejection of the tablet from the die.

The value of P_m at which ejection force for tablets of unlubricated materials approaches a constant value, corresponds to the pressure at which the relative density of the tablet becomes constant at its maximum value (Figs 6–8). The apparent peak value of ρ_R for hexamine is due to the fact that when tablets of this material are compacted at pressures greater than 1,250 kg/cm^2 they exhibit a marked lamination, which

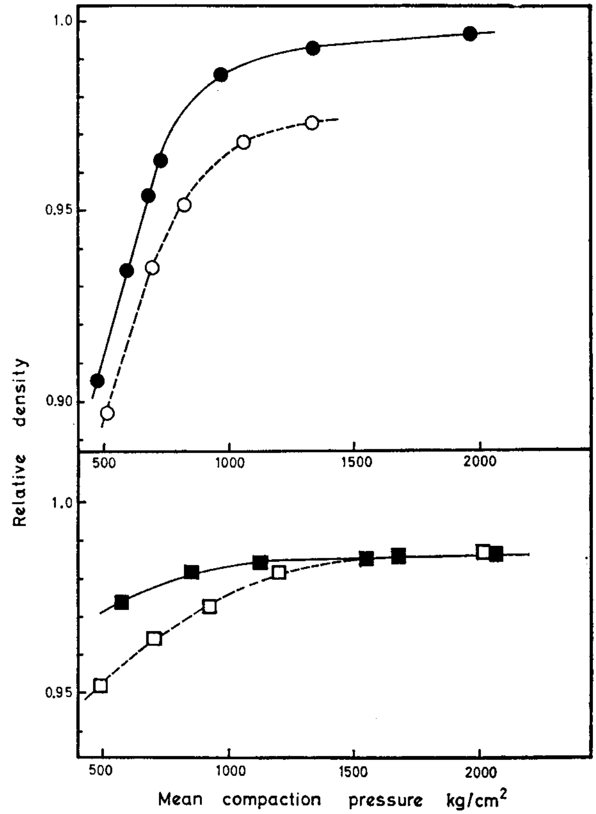

FIG. 6. Effect of mean compaction pressure on density of compact. ● Hexamine granulation, lubricated. ○ Hexamine granulation, unlubricated. ■ Aspirin, lubricated. □ Aspirin, unlubricated.

produces an apparent increase in the measured thickness of the tablet, and consequently a lower value for density.

Similarly, tablets of unlubricated sodium chloride show no sign of attaining a maximum value of F_e because the maximum value of ρ_R is 0·94 (Fig. 7); lubrication facilitates consolidation to a relative density of 0·99, and the ejection force approaches a maximum value. It is notable that at the maximum pressures used (Fig. 8), tablets of lubricated sucrose and sucrose granulation have low ρ_R values compared to the other materials. They also show no terminal value of F_e.

Up to the point where ejection force becomes constant, a linear relationship exists between F_e and F_d (Fig. 9). Such a relationship was reported for sulphathiazole granulation by Nelson & others (1954).

Punch force ratio, R. Three entirely different effects were observed when R was plotted as a function of P_m for the compression of unlubricated materials (Fig. 10). When magnesium stearate was added as lubricant to sodium chloride, sucrose, sucrose granulation, and hexamine,

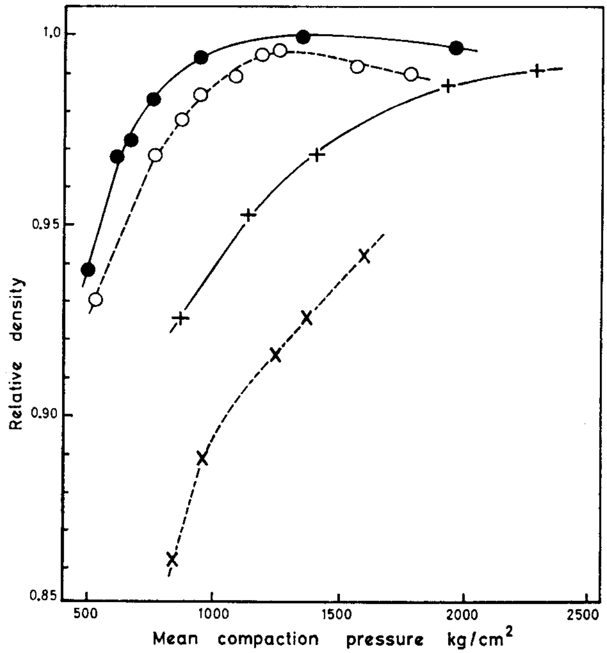

FIG. 7. Effect of mean compaction pressure on density of compact. × Sodium chloride, unlubricated. + Sodium chloride, lubricated. ○ Hexamine, unlubricated. ● Hexamine, lubricated.

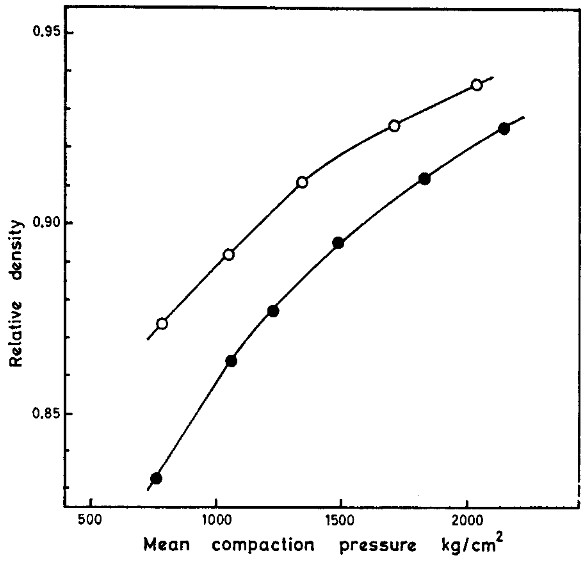

FIG. 8. Effect of mean compaction pressure on density of compact. ○ Sucrose, lubricated. ● Sucrose granulation, lubricated.

the proportion of the applied force transmitted to the lower punch was independent of the magnitude of P_m and the results are best represented by a single horizontal line, R = 0·94. As shown in Fig. 2 lubricated aspirin and hexamine granulation behave in a distinctive manner.

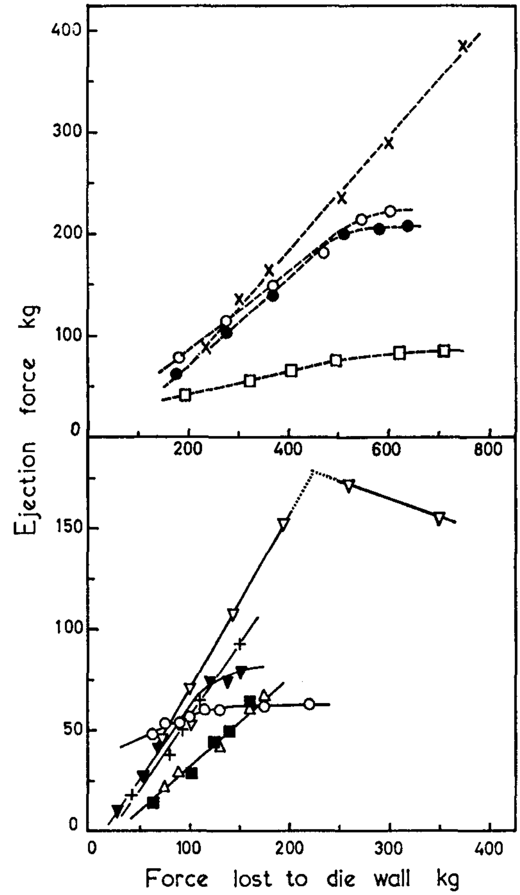

FIG. 9. Relationship between ejection force and the force lost to die wall. Unlubricated materials: × Sodium chloride. ○ Hexamine granulation. ● Hexamine. □ Aspirin. Lubricated materials: ▽ Hexamine granulation. ▼ Hexamine. + Sucrose granulation. ○ Aspirin. △ Sodium chloride. ■ Sucrose.

INFLUENCE OF BASE PARTICLE SIZE

For graphical representation of results the particle size is assumed to be equivalent to the mean of the sieve apertures by which the range is classified (Shotton & Lewis, 1964).

With the exception only of unlubricated sodium chloride and hexamine granulation, the force necessary to eject tablets compressed at a constant pressure is independent of the size of material being compacted (Fig. 11).

The increase in F_e with reduction in particle size of sodium chloride is probably due to the increased area presented to the die wall when the

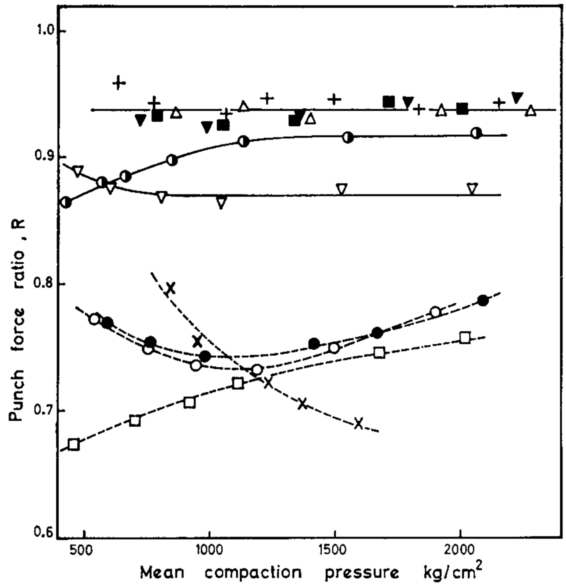

FIG. 10. Effect of mean compaction pressure on punch force ratio. Unlubricated materials: × Sodium chloride. ● Hexamine. ○ Hexamine granulation. □ Aspirin. Lubricated materials: △ Sodium chloride. ▼ Hexamine. ▽ Hexamine granulation. ◑ Aspirin. + Sucrose granulation. ■ Sucrose.

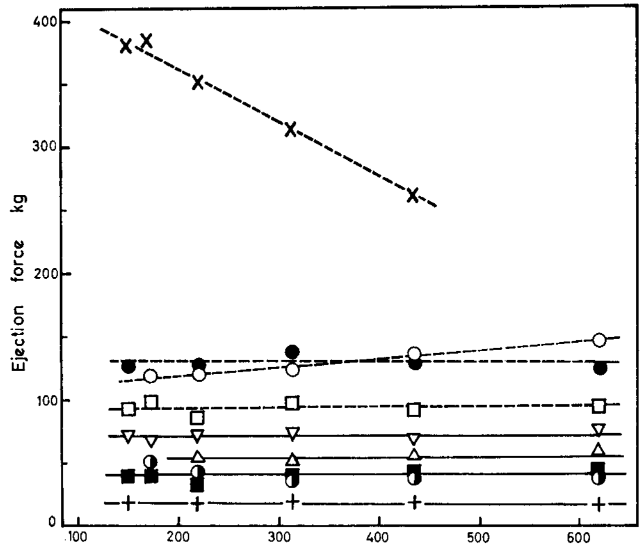

FIG. 11. Effect of base particle size on ejection force. Mean compaction pressures (kg/cm²) as indicated. Unlubricated materials: × Sodium chloride (1485). ○ Hexamine granulation (744). ● Hexamine (802). □ Aspirin (1925). Lubricated materials: △ Sodium chloride (1405). ▽ Hexamine granulation (691). + Sucrose (889). ■ Sucrose granulation (885). ◑ Aspirin (2083). Hexamine (753) results are coincident with those for sodium chloride, lubricated.

smaller material is tabletted. Aspirin, hexamine, and hexamine granulation readily deform so that the area of contact will be approximately constant irrespective of particle size, whereas sodium chloride is harder and more resistant to deformation so that the area of material in contact with the die will depend more on the particle size. The size effect is eliminated by magnesium stearate which subjugates the frictional characteristics of the sodium chloride crystal.

For tablets of unlubricated hexamine granulation F_e values decrease when granule size is reduced. A formed granule undergoes appreciable fragmentation during the compression cycle (Higuchi, Rao, Busse & Swintosky, 1953; Elowe, Higuchi & Busse, 1954; Higuchi, Elowe & Busse, 1954), and the degree of fragmentation will be greatest where the granule is large. It is proposed that the fresh surface produced by compaction of 20–30 mesh granules adheres more strongly to the die wall, and higher ejection force results. Again the effect is eliminated by lubrication.

With the exception of unlubricated sodium chloride, values of R and F_d at a constant compaction pressure were independent of particle size of the base material, whether lubricated or unlubricated. F_d values increased with a decrease in particle size of sodium chloride and there was a corresponding decrease in values of R as shown in Table 1.

TABLE 1. THE INFLUENCE OF PARTICLE SIZE ON FRICTIONAL LOSSES WHEN SODIUM CHLORIDE IS TABLETTED

Mean compaction pressure: 1485 kg/cm²

Particle size μ	Force lost to die wall F_d, kg	Punch Force ratio R
435	550	0·75
315	690	0·69
220	704	0·68
170	712	0·68

References

Elowe, L. N., Higuchi, T. & Busse, L. W. (1954). *J. Amer. pharm. Ass., Sci. Ed.*, **43**, 718–721.
Fuhrer, C. (1962). *Dtsch. Apoth Ztg.*, **102**, 827–832.
Hasegawa, J. (1959). *Chem. Pharm. Bull. Japan*, **7**, 492–500.
Higuchi, T., Elowe, L. N. & Busse, L. W. (1954). *J. Amer. pharm. Ass., Sci. Ed.*, **43**, 685–689.
Higuchi, T., Nelson, E. & Busse, L. W. (1954). *Ibid.*, **43**, 344–348.
Higuchi, T., Rao, A. N., Busse, L. W. & Swintosky, J. V. (1953). *Ibid.*, **42**, 194–200.
Markowski, S. L. (1958). Ph.D. Thesis, Purdue University.
Nelson, E. (1955). *J. Amer. pharm. Ass., Sci. Ed.*, **44**, 494–497.
Nelson, E., Busse, L. W. & Higuchi, T. (1955). *Ibid.*, **44**, 223–225.
Nelson, E., Naqvi, S. M., Busse, L. W. & Higuchi, T. (1954). *Ibid.*, **43**, 596–602.
Riad, M. A. & Zobel, K. F. (1962). *Pharm. Ind.*, **24**, 370–372.
Shotton, E., Deer, J. J. & Ganderton, D. (1963). *J. Pharm. Pharmacol.*, **15**, *Suppl.* 106*T*–114*T*.
Shotton, E. & Ganderton, D. (1960). *Ibid.*, **12**, *Suppl.*, 87*T*–92*T*.
Shotton, E. & Lewis, C. J. (1964). *Ibid.*, **16**, *Suppl.*, 111*T*–120*T*.
Strickland, W. A., Higuchi, T. & Busse, L. W. (1960). *J. Amer. pharm. Ass., Sci. Ed.*, **49**, 35–40.
Strickland, W. A., Nelson, E., Busse, L. W. & Higuchi, T. (1956). *Ibid.*, **45**, 51–55.
Train, D. (1956). *J. Pharm. Pharmacol.*, **8**, 745–761.