J. Pharm. Pharmacol., 1965, 17, 577–583 Received April 26, 1965

The compaction of some solid lubricant materials‡

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Samples of −100 mesh powders of stearic acid, palmitic acid, sodium stearate, potassium stearate, lithium stearate, calcium stearate, magnesium stearate, zinc stearate, zinc oleate, boric acid and a synthetic wax, were compacted and the die reaction determined using a "moving-die" technique. The shear strength of the compacts measured in a punch penetration test, with the exception of boric acid and zinc stearate, was independent of the compaction pressure above 500 kg/cm^2. Solid discs of stearic acid and palmitic acid possessed the same shear strength as the powder compacts; discs of synthetic wax and zinc stearate gave higher values than the corresponding powder compacts. Values of shear strength calculated from measurements of die reaction were higher than values obtained in the shear strength test, but show good agreement with the results calculated by other workers from sliding friction experiments. Shear strength values for compacts of boric acid and discs of synthetic wax, talc crystal, and graphite, indicate that these materials are unlikely to be such good lubricants as stearic acid or its salts.

It has been shown (Hersey, 1960) that friction theory as propounded by Bowden & Tabor (1954) can be applied to a compacting system. Hence the force lost to the die wall, F_d, will be equal to the product of the true area of compact-die interface, A, and the shear strength of the friction junction, S.

$$F_d = A.S \quad \ldots \quad (1)$$

Perfectly clean surfaces of similar or dissimilar materials will adhere with a strength equivalent to the bulk strength of the material (Biewend, 1842; Tomlinson, 1927; Bowden & Hughes, 1939). Almost any contaminant at the interface, e.g., an oxide layer, moisture or grease, will reduce the adhesional forces and hence the sliding friction.

Lubricants, as distinct from glidants, are used in compaction processes to facilitate consolidation under pressure and reduce ejection forces, the shear strength of the friction junctions being effectively reduced by interposing materials of low shear strength between the sliding surfaces. Talc, stearic acid, boric acid, paraffin wax and salts of stearic acid, are solid materials listed (Little & Mitchell, 1963) as being in frequent use.

In the concentrations normally used in pharmaceutical tabletting, sufficient lubricant is present to maintain a film on the surface of the die. The effectiveness of the film will depend on its shear strength, the force with which it adheres to the metal of the die, its resistance to penetration by the material of the compact, and its resistance to wear.

The object of the present work was to study the consolidation of some solid lubricant powders into compacts, the shear strength of the compacts being measured in a punch penetration test and correlated with friction measurements.

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‡ This work formed part of a thesis (C.J.L.) accepted for the degree of Ph.D. in the University of London.

Experimental

MATERIALS AND APPARATUS

The materials examined were stearic acid, palmitic acid, sodium stearate, potassium stearate, lithium stearate, calcium stearate, magnesium stearate, zinc stearate, zinc oleate, talc, boric acid and synthetic wax. All materials were sieved for 15 min using B.S. sieves on an Inclyno machine, and −100 mesh fractions were used.

Where possible the materials used were of B.P. or B.P.C. quality, but in the absence of official standards the best quality technical grade material was used.

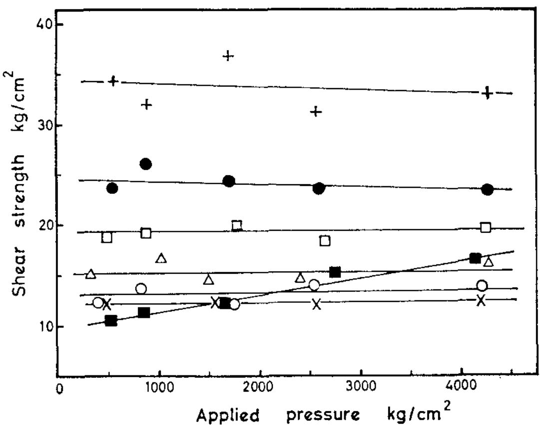

FIG. 1. Effect of applied pressure on shear strength of compacted lubricant powders. + Sodium stearate. ● Potassium stearate. ◻ Zinc oleate. △ Calcium stearate. ■ Zinc stearate. ○ Magnesium stearate. × Stearic acid.

Synthetic wax (Synthetic wax flake W.C. 5956; Wilkins, Campbell and Co.) consists of straight chain hydrocarbons of average molecular weight 750, and has a melting point of 105°. The material was reputed to have the pressing characteristics of a brittle substance, and was supplied in large flakes; a sample was ball-milled to obtain −100 mesh powder.

The apparatus used for compressing the powders has been described (Lewis & Train, 1965a). The shear strengths of the compacts were measured using a punch penetration test originally used with solid homogeneous discs (Train & Hersey, 1960), and subsequently applied to compacts of crystalline materials (Lewis & Train, 1965b).

METHODS

Samples of boric acid powder (5 g) and samples of the other materials (4 g) were compacted at various pressure levels up to 4,300 kg/cm^2 using the moving-die technique under standardised operating conditions (Lewis & & Train, 1965a). The applied pressure, P_a, the die reaction, F_d, and change in length of compact with pressure were recorded for each pressing. The compacts were ejected from the die and the shear strength measured.

To assess more accurately the shear strength of boric acid compacts, five replicate determinations were made on material pressed to 4,000 kg/cm^2. A similar number of replicate measurements were made on compacts of the other materials pressed to 1,725 kg/cm^2. Values of die reaction were not required from these pressings and the die remained stationary during the compaction.

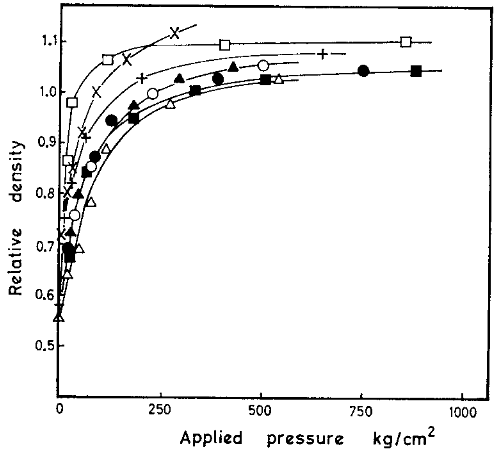

FIG. 2. The relationship between applied compaction pressure and relative density of compact. + Sodium stearate. ● Potassium stearate. □ Palmitic acid. △ Calcium stearate. ○ Magnesium stearate. ■ Zinc stearate. × Stearic acid. ▲ Lithium stearate.

Stearic acid, palmitic acid, synthetic wax, hard paraffin and zinc stearate, were melted with the minimum of heat and cast into cylindrical blocks approximately 3·5 cm diameter. Solid blocks of talc crystal and graphite were available. From these blocks of materials, discs 2·42 cm diameter and approximately 0·6 cm long were cut on a lathe. The shear strengths of these solid discs were estimated under the same conditions as those used for the compacts.

Results and discussion

The shear strengths of compacts made from powdered stearates of magnesium, sodium, potassium and calcium are virtually unaffected by the magnitude of the compacting pressure above approximately 500 kg/cm^2 (Fig. 1). Stearic acid and zinc oleate behave in a similar manner but zinc stearate shows a small but steady increase in shear strength with pressure. It was observed that all compacts of stearic acid and the stearates tended to laminate as they were ejected from the die. The measured length of the compact thus depended on the way the micrometer gauge was handled during the measurement, the laminated compact possibly being compressed. This led to some variation in measurements of length of compact, which probably accounts in part for the observed variations in strength.

That shear strength values are essentially constant over a pressure range 500–4,250 kg/cm^2 is explained by the fact that these materials attained zero porosity at pressures less than 500 kg/cm^2 (Fig. 2), so that an increase in pressure above that figure does not produce any greater

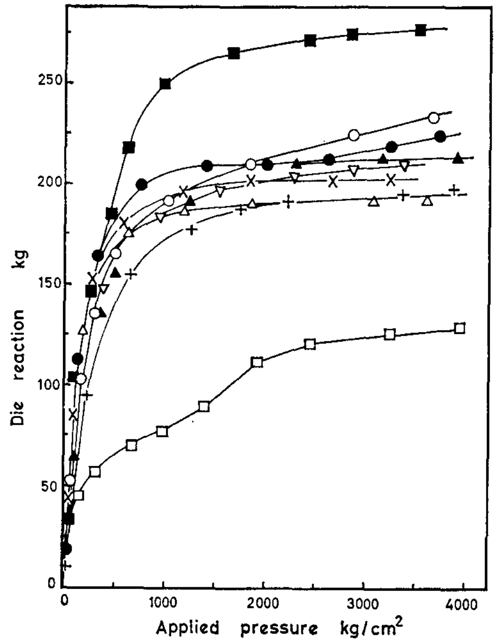

FIG. 3. Effect of applied compaction pressure on die reaction. + Sodium stearate. ● Potassium stearate. ▽ Palmitic acid. △ Calcium stearate. ■ Zinc stearate. ○ Magnesium stearate. ▲ Lithium stearate. × Stearic acid. ◻ Zinc oleate.

densification and only serves to aggravate lamination of the compact. Apparent values of relative density ρ_R (ratio of apparent density of compact to density of solid) greater than unity may be explained by the extrusion of material past the punch tips, this being particularly noticeable with stearic acid and palmitic acid. The difficulty of containing the experimental material was so great with zinc oleate that calculated values of ρ_R were meaningless.

Frictional losses for the above materials increase with pressure initially (Fig. 3) and then approach a constant value at pressures greater than 1,500 kg/cm^2. Except for zinc stearate and zinc oleate the maximum values of die reaction, F_d, lie within the range 190–225 kg.

Boric acid produces a greater die reaction (Fig. 4) than the soap lubricants, and it is found that the shear strengths of the compacts are also higher. In addition the shear strength of the compact increased with increased compaction pressure to a maximum at a pressure of 4,000 kg/cm^2 (Fig. 5). Although of a waxy nature, synthetic wax produced die wall frictional forces similar to boric acid (Fig. 4) indicating the unsuitability of these two materials as die wall lubricants when a constraining load is applied.

Table 1 lists values of shear strength for the compacted powders and the solid discs. Using values of shear strength for powder compacts, and calculating the area of compact-die wall interface from experimental

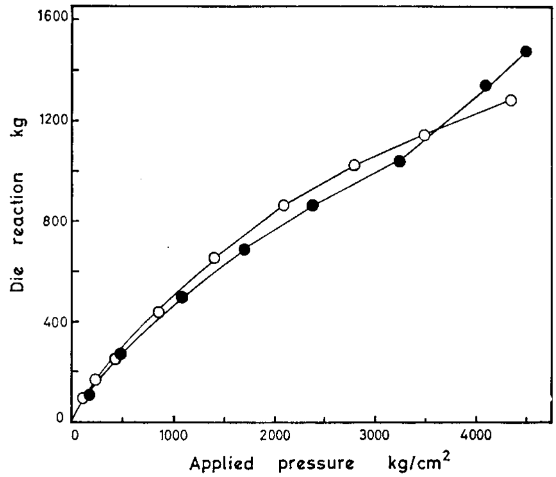

FIG. 4. Effect of applied compaction pressure on die reaction. ● Synthetic wax. ○ Boric acid powder.

measurements of length of compact at a given pressure, values for F_d were calculated from equation (1) and are compared with the maximum experimental values, also in Table 1. Experimental and calculated values were only compared for those materials attaining an approximately constant value of F_d as the compacting pressure increased.

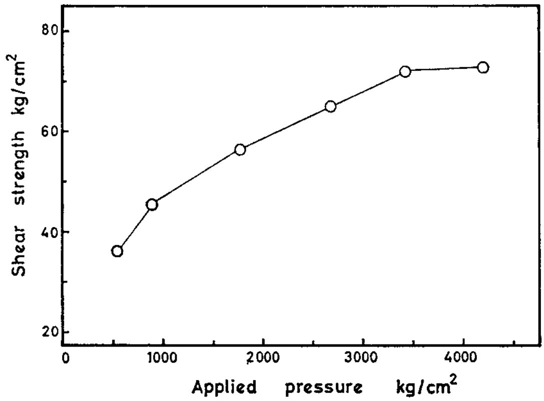

FIG. 5. Effect of applied pressure on shear strength of compacts of boric acid powder.

Most of the salts of stearic acid investigated decompose at high temperatures without melting, so that it was not possible to manufacture solid discs. Compacts of talc powder suffered gross lamination and fracture on ejection from the die so that the shear strength of a compact could not be measured for comparison with solid discs.

Only the calculated values for the sodium and potassium stearates are comparable with the observed practical results. Maximum die reaction values in the range 190–225 kg would require a material possessing a shear strength of 28–34 kg/cm², i.e., approximately twice the measured value for compacts of stearic acid, palmitic acid, calcium stearate and magnesium stearate, three times larger in the case of zinc stearate, and five times larger in the case of lithium stearate.

TABLE 1. THE SHEAR STRENGTH OF LUBRICANT MATERIALS AND VALUES OF DIE REACTION, F_d

Material	1 Shear strength powder compacts* kg/cm²	2 Shear strength solid discs** kg/cm²	3 Max. exptl. die reaction F_d, kg	4 Calculated die reaction† F_d, kg
Lithium stearate	6·0	—	212	41
Zinc stearate	9·3	20·2	275	61
Palmitic acid	12·3	12·7	208	89
Stearic acid	13·7	13·3	200	99
Calcium stearate	15·0	—	190	101
Magnesium stearate	20·0	—	230	135
Potassium stearate	31·3	—	210	212
Sodium stearate	33·9	—	195	229
Synthetic wax	50·5	65·0	—	—
Boric acid	73·0	—	—	—
Hard paraffin	—	19·0	—	—
Talc, with grain	—	63·2	—	—
Talc, cross grain	—	80·0	—	—
Graphite	—	75·0	—	—

* Mean of five determinations. ** Mean of three determinations. † From data in column 1.

Shear strengths calculated from F_d values show good agreement with the results of Bailey & Courtney-Pratt (1955), who found that bimolecular films of calcium stearate possessed a shear strength of 25 kg/cm², and that multi-layers gave a lower value. Wilson (1955) reported shear strength values for stearic acid in the range 27–35 kg/cm², and indicated that the friction observed was mainly due to shearing of the lubricant film. Correlation is probably the more remarkable when it is realised that the results of Wilson, and of Bailey and Courtney-Pratt, were obtained in sliding friction experiments using very refined techniques.

Comparison of the present results with those of other workers gives rise to three considerations.

(1) The punch penetration shear test produces shear in multilayers and not bimolecular layers; the low values of shear strength obtained can then be explained (cf. Bailey & Courtney-Pratt, 1955).

(2) Experimental values of die reaction, F_d, are due to shear of bimolecular, or very thin films.

(3) If the shear strength of the compact is the true value for the materials used, the higher values calculated from F_d measurements may be due to the material possessing a higher shear strength when it is subject to a

compressive load. This would be consistent with the findings of Bridgman (1946), Boyd & Robertson (1945), and Cameron (1960).

Further work on the measurement of shear strength of lubricant powder compacts subjected to a compressive load will help to clarify the matter.

Solid discs of zinc stearate have twice the shear strength of the powder compacts (Table 1), although the two sets of values for stearic acid and palmitic acid demonstrate single values within the limits of experimental error. It may reasonably be assumed that the film of compacted lubricant powder on the die wall will behave in the same manner as the solid material under conditions of shear. The results for zinc stearate seem to indicate that for salts of stearic acid the shear strength of the solid material may be greater than that of a powder compact. This would account for the difference between calculated and experimental values of F_d (columns 3 and 4, Table 1).

Shear strength values for talc and graphite discs show how less desirable these materials may be as lubricants compared to the fatty acid and soap materials, especially when subject to a constraining load (Train & Hersey, 1960). Present results for the shear strength of talc discs are higher than those obtained by Train & Hersey, and are almost certainly due to the increased rate at which the shearing load was applied.

If the shear strength of the lubricant is the main factor affecting its efficiency during normal pelletting and tabletting procedures, then the material with the lowest shear strength should be the most efficient. The materials used in this study have been evaluated as lubricants on an instrumental tablet machine and it is hoped to publish the results elsewhere.

References

Bailey, A. I. & Courtney-Pratt, J. S. (1955). *Proc. roy. Soc.*, **A227**, 500–515.
Biewend, E. (1842). *Ann. Physik.*, **57**, 164–165.
Bowden, F. P. & Hughes, T. P. (1939). *Proc. roy. Soc.*, **A172**, 263–279.
Bowden, F. P. & Tabor, D. (1954). *The Friction and Lubrication of Solids*, Oxford: Clarendon Press.
Boyd, J. & Robertson, B. P. (1945). *Trans. Amer. Soc. Mech. Engrs.*, **67**, 51–56.
Bridgman, P. W. (1946). *Rev. Mod. Phys.*, **18**, 1–93.
Cameron, A. (1960). *Amer. Soc. Lubric. Engrs. Trans.*, **2**, 195–198.
Hersey, J. A. (1960). Ph.D. Thesis, University of London.
Lewis, C. J. & Train, D. (1965a). *J. Pharm. Pharmacol.*, **17**, 33–41.
Lewis, C. J. & Train, D. (1965b). *Ibid.*, **17**, 1–9.
Little, A. & Mitchell, K. A. (1963). *Tablet Making*, 2nd ed., Liverpool: Northern Publishing Co.
Tomlinson, G. A. (1927). *Proc. roy. Soc.*, **A115**, 472–483.
Train, D. & Hersey, J. A. (1960). *J. Pharm. Pharmacol.*, **12**, *Suppl.* 97*T*-104*T*.
Wilson, R. W. (1955). *Proc. Phys. Soc.*, **B65**, 625–641.