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A comparison of tablet lubricant efficiencies for a sucrose granulation using an instrumented tablet machine†

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Thirteen materials have been assessed as lubricants when added to a sucrose granulation and compressed on an instrumented tablet machine. Values of ejection force, force lost to die wall, and punch force ratio for each lubricated sample were compared, and the order of efficiency as lubricants was compared with the shear of lubricant powder compacts. The most efficient lubricants are those materials of lowest melting point, and not those with the smallest shear strength value as measured in a punch penetration test.

IN the production of tablets, substances are usually added to the materials to be compressed to improve the flow of granules and to reduce the friction between tablet material and the die wall. Those materials facilitating flow have been termed "glidants", and those reducing friction, "lubricants" (Munzel & Kagi, 1954).

Evaluations of various materials as lubricants have been made by Wolff, DeKay, & Jenkins (1947), Nelson, Naqvi, Busse & Higuchi (1954), Patel & Guth (1955), Markowski (1958), Strickland, Higuchi & Busse (1960) and Maly (1961). More importance is attached to the quantitative results of Nelson and Strickland and their colleagues and Markowski, which were obtained using conventional tabletting machines instrumented with strain gauge equipment. Although there is general agreement between workers that the best tablet lubricants are found in that class of materials normally called "boundary lubricants" in the general field of lubrication, e.g. salts of stearic acid, there are discrepancies between the various orders of merit. Only Markowski (1958) attempts an explanation of this variation and points out that his rating of the stearates seems to be related to the valency of the metal involved; he suggests that the "size of the molecule" may influence lubricant efficiency. Thus of all materials examined by this worker sodium stearate reduced the ejection force to the greatest extent and also had the smallest molecular size.

The magnitude of the die wall friction will depend on the shear strength of the friction junction and the area of contact (Bowden & Tabor, 1954). Good lubricants would be expected to have low shear strength values and the shear strength of a number of materials has been measured by Lewis & Train (1965).

The present work is an attempt to evaluate the lubricating efficiency of these same materials using a tablet machine instrumented with strain

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gauges as described by Shotton & Ganderton (1960). The relative efficiency of a lubricant is then compared with its shear strength value.

Experimental

MATERIALS AND APPARATUS

A granulation of sucrose was prepared from material ball-milled until all passed a 100 mesh sieve. The powder was moistened with distilled water and pressed through a 30 mesh sieve. The granules were dried at room temperature (2 hr) then at 60° (2 hr). They were sieved on an Inclyno machine for 15 min and the 30–40 mesh fraction used throughout this work.

To samples of 30–40 mesh granulation, 2% of the following materials was added as –100-mesh powder: stearic acid, palmitic acid, lithium stearate, sodium stearate, potassium stearate, magnesium stearate, zinc stearate, zinc oleate, calcium stearate, "synthetic wax,"* boric acid, and talc.

Hard paraffin was applied to the granulation as a solution in light petroleum (b.p. 60°–80°). The granules were coated with this in a coating pan, using a jet of hot air to remove the solvent; they were then dried (2 hr) at 60°/20 mm.

METHODS

Preliminary experiments indicated that the sucrose granulation possessed desirable pressing characteristics and high frictional resistance, making it suitable as a base material. When lubricated with –100-mesh magnesium stearate powder, this material gave a linear relationship between ejection force and mean compaction pressure over the range 500—2000 kg/cm². The proportion of the applied pressure transmitted to the lower punch over this range was constant. Values of the punch force ratio, R, force lost to die wall, F_d, and ejection force, F_e, were unaffected by variations in base granule size, and of all the lubricants examined 2% –100-mesh magnesium stearate affected these values to the maximum extent.

Samples weighing 700 mg were filled into the die with the aid of a funnel, levelled, and were compacted at a mean compaction pressure of 1033 kg/cm². The die was conditioned by compressing five tablets from the material in question; these were rejected and a further five tablets prepared. Measurements were made of upper punch force, lower punch force, ejection force, weight of tablet, and length of tablet.

Between the evaluation of each lubricated sample the die and punches were removed from the machine, cleaned with metal polish, polished, and then degreased with a mixture of equal parts of carbon tetrachloride and acetone before conditioning with the next sample.

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* Synthetic wax flake WC 5956: Wilkins, Campbell and Co.

Results and discussion

Nelson & others (1954), Strickland, Nelson, Busse & Higuchi (1956), Strickland & others (1960) and Markowski (1958) favoured the ratio (R) of force transmitted to the lower punch to that applied by the upper punch as a means of comparing lubricant efficiencies. The better the lubricant, the greater the proportion of the applied force transmitted to the lower punch: values of R thus tend to unity.

The use of this parameter was criticised by Maly (1961) as being insufficiently sensitive to distinguish between good lubricants, and a lubricating factor based on ejection forces was proposed.

$$\text{Lubricating factor M} = \left(1 - \frac{T_2}{T_1}\right) \times 100$$

where

T_2 = ejection force for a given pressure for lubricated sample

T_1 = ejection force at same pressure for unlubricated sample of base material

For a given base material this factor (M) offers no advantage over the use of the ejection force F_e. Comparisons based on ejection force were also made by Patel & Guth (1955), Smilek, Cosgrove & Guth (1955), Appino, Banker & De Kay (1959) and Munden, De Kay & Banker (1960), but the method of instrumentation was not as sensitive as the strain gauge equipment used by other workers.

Present results for ejection force, F_e, punch force ratio, R, and force lost to die wall, F_d, are presented in Table 1.

TABLE 1. EVALUATION OF TABLET LUBRICANTS

Material	Ejection force, Fe, kg	Punch force ratio, R	Force lost to die wall, Fd, kg	Melting point °C.	Shear strength,* kg/cm²
Hard paraffin	15	0·94	80	50	—
Stearic acid	22	0·94	78	54	13·7
Palmitic acid	24	0·95	68	57	12·3
Synthetic wax	26	0·84	224	105	50·5
Sodium stearate	38	0·93	93	240–243	33·9
Zinc oleate	40	0·93	93	170–174	—
Lithium stearate	41	0·95	63	215–218	6·0
Potassium stearate	43	0·94	89	252–255	31·3
Zinc stearate	45	0·94	82	120	9·3
Calcium stearate	48	0·93	103	140	15·0
Magnesium stearate	50	0·93	104	186	20·0
Boric acid	346	0·63	584	—	73·0
Talc	353	0·59	664	—	—
Unlubricated	371	0·55	682	—	—

* Lewis & Train (1965). Spread of results ± 7%. Values measured under zero load conditions.
Base material: 30–40 mesh sucrose granulation
Lubricant concentration: 2%
Mean compaction pressure: 1033 kg/cm²
Sample weight: 0·7g

Most values of R fall in the close range 0·93–0·95 making the use of this factor unsatisfactory as a means of differentiating between the lubricating efficiencies of most of the substances tested. This parameter is insufficiently sensitive to distinguish between the soap lubricants but could serve as a first classification to separate "efficient" lubricants, such as magnesium stearate, from "inefficient" ones such as talc.

Ejection forces allow of slightly sharper differentiation. The most efficient lubricants in this respect are the fatty acids and waxes, those materials with the lowest melting point. Salts of stearic acid melt with decomposition at a much higher temperature than the acid itself (see Table).

Bowden & Tabor (1954) have shown that considerable temperatures are generated even in the lightest frictional contacts, and that in addition, where boundary layer lubrication conditions exist, the efficiency of soap lubricants and fatty acids drops appreciably when the surface temperature reaches the value of their melting-points.

The addition of fatty acids to metals with which they react, results in the formation of soap films possessing strong lateral cohesion and a melting-point much higher than that of the acid. It is clear that the application of a metallic soap need not necessarily be as effective a lubricant as the soap formed *in situ.* The present results suggest that 2% lubricant is sufficient to make boundary layer conditions unimportant, so that the greatest efficiency of lubrication is not dependent on the high melting-point of a lubricant. A low melting-point probably facilitates the formation of wax or fatty acid films on the die wall.

Table 1 shows that the ratings of lubrication efficiency, as assessed by comparison of ejection forces, are not explained by the values of shear strength measured by the punch penetration method (Lewis & Train, 1965). Lithium and zinc stearates, which have the lowest shear strengths, gave ejection forces nearly twice those for hard paraffin, stearic and palmitic acids. The absence of such correlation may well be due to the properties of the sucrose granulation substrate. Jones (1960) reports for metal powders that the best lubricant and optimum concentration depends on the powder being pressed, and Daoust (1960) presents evidence that the efficiency of lubricants used with pharmaceutical granulations may vary with the type of granulating agent used. Such interdependence between substrate and lubricant may well account for different workers producing different orders of efficiency.

On the other hand the shear strength of compacted lubricants under zero load conditions may not be the important value for comparison purposes. When the compressive force has been removed and before ejection from the die, the tablet exerts a radial force on the walls of the die. The lubricant film will therefore be subjected to a constraining load and it is possible that the shear strength of the lubricant under an applied compressive load is the value that is needed.

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