DRUG DEVELOPMENT AND INDUSTRIAL PHARMACY, 11(4), 845-854 (1985)

FRICTIONAL PROPERTIES OF TABLET LUBRICANTS

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ABSTRACT

Some frictional properties of tablet lubricants were determined. The friction coefficients and the adhesion forces of six lubricants were evaluated by the method proposed previously. The ejection force against the radial force for each lubricant yielded a straight line through the origin, so that the adhesion forces of these lubricants were estimated to be almost zero. All lubricants had low friction coefficients when they alone were compressed. The value for metal stearate was the smallest and that for talc was the largest. The affinity of the lubricants to the die wall, another important property of the lubricants, was also determined. After the die wall was conditioned by the tabletings of each lubricant alone, the serial tabletings of lactose granulates in the die were carried out. The increasing rate of ejection force in the conditioned die in a serial tableting was different for every pretreatment

0363-9045/85/1104-0845$3.50/0

of each lubricant. The affinity of magnesium stearate to the die wall surface was superior to that of other lubricants.

INTRODUCTION

It has previously been proposed a novel method for estimating the friction between a tablet and a die wall in the tablet ejection process (1). In the tablet formulation lubricants are commonly used to reduce the friction between a tablet and a die wall, and to prevent the adhesion between a tablet and punch surfaces. It is empirically known that magnesium stearate has superior properties, so that magnesium stearate is commonly chosen as a lubricant in the pharmaceutical preparations. However, little information has been presented for the friction properties of magnesium stearate as well as for other lubricants (2,3,4). The purpose of this paper is to determine some frictional properties of tablet lubricants by the method previously proposed.

EXPERIMENTAL

Procedure

The apparatus used was identical with that described in the previous studies (1). Each lubricant was compressed in the die on a physical testing instrument (Autograph IS-5000, Shimadzu Seisakusho Ltd.) at 1500kg/sq.cm of compressional force. The punch and die assembly in which the compact of the lubricant was held stationary was transferred to the space in the apparatus. The ejection force for the compact was measured while the radial force was monitored simultaneously.

The conditions of the operation used were as follows;

The strain rate for compression : 50mm/min.

The maximum compressional force : 1500kg/sq.cm.

The strain rate for ejection : 5mm/min.

The die-punch assembly : 10mm flat-faced.

The weight of sample : 400mg.

In another case, the ejection force was measured without simultaneous measurement of radial force to evaluate the friction between the die wall and lubricants or other materials. In this case, the identical material was compressed at a definite compressional force, so that the radial force developed would be the same.

Materials

Magnesium stearate J.P. (Sakai-kagaku Co.), calcium stearate, stearic acid (Wako Pure Chemical Ind. Ltd.), Lubri-wax (hydrogenated vegitable oil, Freud Ind. Co. Ltd.), talc J.P. (Nihon Talc Co.) and corn starch J.P. (Nihon Cornstarch Co.) were used as lubricants. Lactose granulates were prepared by massing lactose J.P. (D.M.V., Holland) with 15%w/w aqueous solution of hydroxypropyl cellulose J.P. (Nippon Soda Co. Ltd.) and 3%w/w binder was contained in the final granulates. The dried granulates were passed through a 32 mesh sieve. The blended mixtures were obtained by mixing granulates and lubricant in a V-shape mixer (5-litre, Patterson-Kelly, U.S.A.).

RESULTS AND DISCUSSION

Friction coefficients of lubricants

The results of determination of the friction properties for some lubricants are shown in Fig.1. For each lubricant, a graph of the ejection force (Fe) against the radial force (Fr) yielded a straight line through the origin. The friction coef-

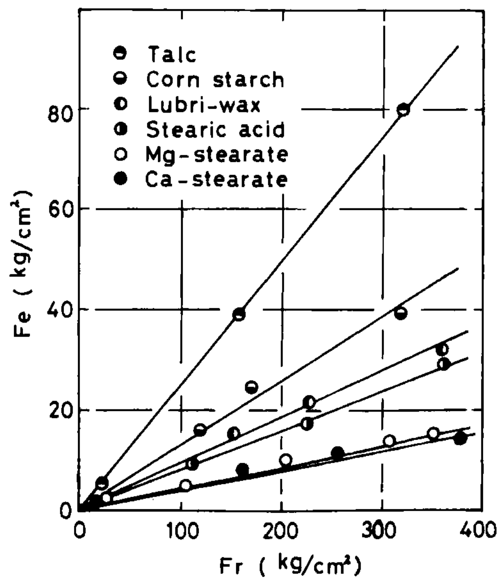

Fig.1 Relationship between the ejection and radial forces for the compacts of some lubricants.

ficient and the adhesion force can be obtained from the equation,

$$Fe = \mu Fr + C$$

where Fe is the ejection force, Fr is the radial force, μ and C are the friction coefficient and the adhesion force respectively (1). The results are listed in Table 1. Since the straight lines were through the origin, the adhesion forces of these lubricants were estimated to be almost zero. Metal stearates such as magnesium stearate have the smallest friction coefficient. On the other hand, the friction coefficient of talc was the largest, followed by that of corn starch. Talc may behave as rather an antiadherent than lubricant (5).

Train reported the relationship between orientation and shear strength for talc (2). And Lewis also measured the shear strength for some metal salts of stearic acid (3). As already mentioned, these measurements were carried out without

Table 1 Friction coefficients of some lubricants.

Lubricant	Friction coefficient
Talc	0.25
Corn starch	0.13
Lubri-wax	0.09
Stearic acid	0.08
Magnesium stearate	0.04
Calcium stearate	0.04

determination of the radial forces. It is noted, however, that the ranking of the values of magnesium stearate, calcium stearate and talc, and the orders of their values in their reports coincide with those of our results. This may be because the very small radial force was developed during the compression of lubricant alone.

Affinity of lubricants to the die wall

Another important property of lubricants is their affinity to the die wall. The rigid adhesion of the lubricant to the die wall should imply an effective lubrication. In order to determine the degree of lubricant affinity to the die wall, the serial tabletings of lactose granulates were carried out in the die conditioned by the preliminary tableting of each lubricant alone. Fig.2 shows the change in ejection force with increasing tableting number, where the relation between the ejection force and tableting number was employed since the identical granulates were compressed. The increasing rates of the ejection force during serial tabletings of unlubricated lactose granulates after tableting of each lubricant were dif-

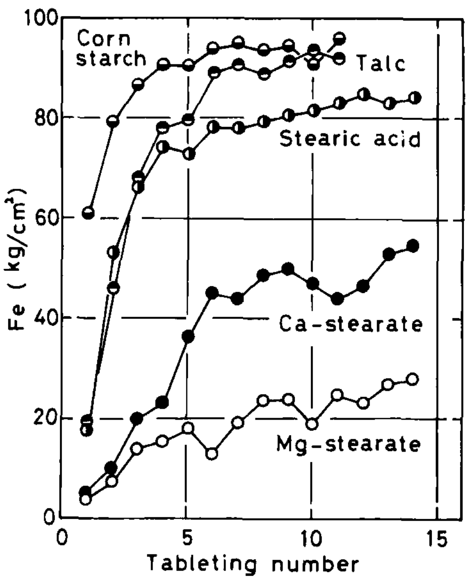

Fig.2 Change in ejection force in serial tabletings of unlubricated lactose granulates after tableting of each lubricant.

ferent for every pretreatment of each lubricant. The lubrication effect of talc or corn starch was lost by several tabletings of lactose granulates. On the other hand, the lubrication effect of magnesium stearate was maintained even after ten or more tabletings of granulates.

The affinity of magnesium stearate to the die wall was superior to that of calcium stearate, where the friction coefficients of both magnesium and calcium stearate were nearly equal to each other as seen in Table 1. The apparent larger affinity of magnesium stearate than calcium stearate to the die wall may be attributed to smaller primary particle size of magnesium stearate than that of calcium stearate. Or magnesium salt itself may have intrinsically greater affinity to the metallic die wall than calcium salt. The strong affinity of magnesium and calcium stearate to the die wall seems to be inconsistent with their frictional properties, that is, a small friction coefficient and practically no adhesion force. The strong affinity of both magnesium and calcium stearate to the die wall must result in the presence of adhesion force in the friction experiment. However, no adhesion force was observed in the frictional experiment. When the compact of the lubricant being tested is ejected, a mono- or multi-layer film of the lubricant will be left on the surface of the die wall. The phenomenon is reasonable since it is thought that magnesium stearate is a bondary type lubricant (5). The adhesion force measured in the experiment must correspond to the adhesion between lubricant particles and not between lubricant and the die wall. The same phenomenon will occur with the compression of not only lubricant alone but granulates as well.

Effect of lubricant on friction properties of blended mixtures

The apparent binding was observed on the surface of the die wall after some serial tabletings of granulates alone. Hölzer reported that lubricants lowered the friction coefficients of NaCl (4). Magnesium stearate was the most efficient lubricant in their studies.

The results of the friction experiments for the blended mixture of lactose granulates with magnesium stearate at 0.1, 0.3, and 0.5% levels are shown in Fig.3. With the unlubricated granulates not only an equilibrium value for ejection force during the serial tabletings was not obtained but also the binding occured after a few serial tabletings. On the other hand, with the blended mixtures an equilibrium state in the ejection force was obtained after several tabletings.

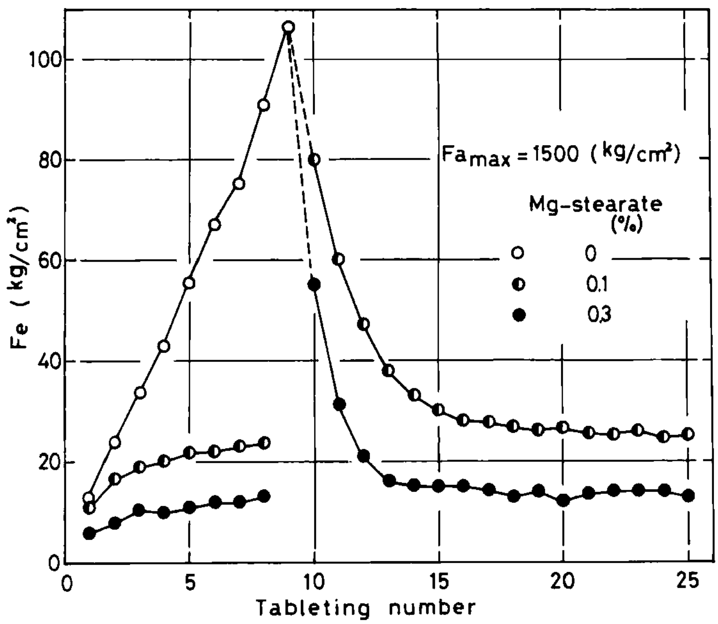

Fig.3 Change in ejection force in serial tabletings of blended mixtures (0.1, 0.3 and 0.5% magnesium stearate) in the clean die or in the conditioned die obtained by nine serial tabletings of unlubricated lactose granulates.

It is clear that the addition of the lubricant lowered the friction coefficient and the adhesion force. Especially the reduction in the adhesion force by lubricant addition was drastic. Not only the lubricant materials but also their concentration and mixing time may affect the frictional properties of blended mixtures. The values of the friction coefficients and the adhesion forces evaluated at sixth tabletings are summarized in Table 2.

When the lactose granulates with different concentrations of magnesium stearate were compressed serially in the conditioned die which was obtained by the nine serial tabletings of lactose granulates alone, the ejection force decreased

Table 2 The effect of lubricant materials, concentration, and mixing time on the friction coefficient and the adhesion force for the blended mixtures.

Lubricant Material	Lubricant Conc.(%)	Mixing Time(min)	Friction Coefficient	Adhesion Force(kg/sq.cm)
Magnesium stearate	0.1	1	0.25	10
		10	0.23	8
		30	0.18	4
	0.3	1	0.22	5
		10	0.17	3
		30	0.12	2
	0.5	1	0.15	3
		10	0.09	1
		30	0.08	0
Stearic acid	0.8	10	0.23	8
	2.0	10	0.17	4
	4.0	10	0.10	1
Talc	1.0	10	0.48	23
	2.0	10	0.38	14
	4.0	10	0.26	8

with the tabletings and approached a constant value as shown in Fig.4. The values depended upon the concentration of magnesium stearate. The values were about 15kg/sq.cm at 0.35% of magnesium stearate and about 25kg/sq.cm at 0.1% which were reached after ten or more serial tabletings. When the blended mixture was compressed in the clean die as

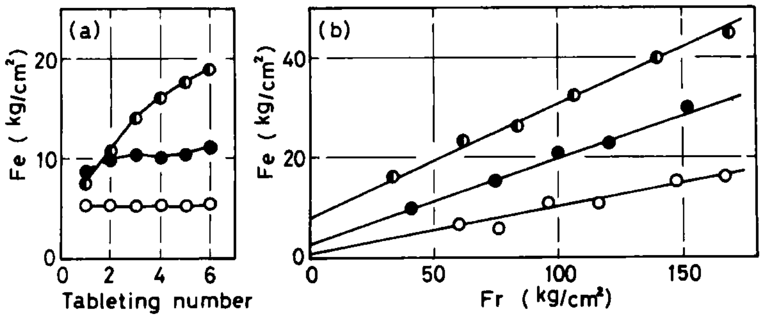

Fig.4 Change in ejection force in serial tabletings of blended mixtures (0.1 and 0.3% magnesium stearate) after nine serial tabletings of unlubricated lactose granulates.

shown in Fig.3, the ejection force also approached a constant value which was obtained by the tableting of the mixture in the conditioned die. These results suggested that the compression and ejection of the blended mixture in a die will be equilibrium phenonena of absorption and desorption of the granulate and lubricant particles to the die wall surface.

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