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Research Papers

FRICTION COEFFICIENTS OF TABLET MASSES

ARNE W. HÖLZER and JOHN SJÖGREN

Department of Pharmaceutics, AB Hässle S-431 83 Mölndal (Sweden)

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SUMMARY

In order to obtain a better understanding of friction during tabletting, the possibility of determination of friction coefficients was investigated using a reciprocating tablet machine instrumented with piezoelectric load washers on the punches and strain gauges at the die wall. By suitable amplifications and calibration it was possible to determine the friction coefficient at the pressure maximum (μ_1) and during ejection of the tablet (μ_2) with acceptable precision. Careful cleaning of the die was not sufficient to provide reproducible values. Conditioning with about 10 tablets was required before the determination. The applied pressure affected μ_1 for some materials but not μ_2. The friction coefficient for stearic acid and magnesium stearate was 0.1, for well-lubricated tablet masses 0.2–0.4, for non-lubricated materials 0.7–2 if non-sticking, and above 2 if adhesion to the die wall occurred.

INTRODUCTION

In previous studies on friction properties of tablet masses we have used the force difference (FD), the punch force ratio (R-value), the remaining force on the lower punch (REF) and the ejection force (EJF) (Hölzer and Sjögren, 1977, 1978, 1979a and b). We suggested the use of FD and EJF after corrections for differences in circumferential area to evaluate friction properties of tablet masses. For some tablet masses, e.g. microcrystalline cellulose (Avicel PH 101), the linear relationship held only in a very limited pressure range (Hölzer and Sjögren, 1977).

A friction coefficient can be calculated by simultaneously measuring both the axial and radial forces during tabletting where, using friction terminology, the tablet is the "slider" and the die wall the "support". Friction coefficients have been calculated in metallurgical compaction studies (e.g. Bockstiegel and Svensson, 1971; Dangerfield et al., 1977) but not in tabletting of pharmaceutical materials. Al Shammat et al. (1979, 1980) have reported apparent friction coefficients of some direct compression bases but have not measured the radial force during tabletting. Therefore the "apparent" coefficients included the stress ratio and were based on the assumption that the force distribution equation proposed by Unckel (1945) was valid for the materials.

The aim of this study was to investigate the possibility of determining the friction coefficient (μ) during compression (μ_1) and ejection (μ_2) with the aid of an instrumented tablet press. We also wanted to evaluate whether μ is a parameter less dependent on the tabletting pressure than FD or EJF.

MATERIALS AND METHODS

Materials

Acetyl salicylic acid BP (ASA 7013, Monsanto Chem., U.K.) *Alprenolol hydrochloride* (alprenolol, Astra Pharmaceutical Production, Södertälje, Sweden, 5 random batches from 3 years production. *Ascorbic acid* USP (Hoffman-La Roche, Switzerland). *Microcrystalline cellulose* NF (Avicel PH 101, FMC Corp., U.S.A.), 2 different batches. *Dibasic calcium phosphate dihydrate* NF, granulated with polyvinylpyrrolidone (PVP K25, BASF, G.F.R.) in water. The granulate containing 3% PVP, was passed through an 0.5 mm sieve and had a loss on drying of 2.2% (105°C, 15 min). *Anhydrous lactose* USP, (lactose, Sheffield, U.S.A.). *Corn starch* USP (Maizena, Krefeld, G.F.R.). *Magnesium stearate* USP (Unilever Emery, Holland). Specific surface area by permeametry 6.3 m^2 g^{-1} (Fisher Sub-Sieve Sizer, porosity 0.60). Loss on drying 3.6% (105°C, 2 h). *Paracetamol crystalline* BP (Bayer AG, Leverkusen, G.F.R.). *Cubic sodium chloride* USP (NaCl, KNZ, Holland). Arithmetic mean sieve diameter 0.27 mm and 0.43 mm (Allen Bradley Sonic Sifter). *Granular starch* (Sta-Rx 1500, A.E. Staley, U.S.A.). *Stearic acid* USP (Crodacid PE3180, Croda Chemicals, U.K.). Specific surface area by permeametry 0.089 m^2 g^{-1} (Fisher Sub-Sieve Sizer, porosity 0.47).

Equipment

Tablets of 1.13 cm diameter were compressed in a reciprocating tablet press equipped with load washers (punch forces), strain gauges (die wall force) and inductive displacement transducers (punch positions and tablet height during compression) as described by Hölzer and Sjögren (1979b).

The punch force signals were calibrated as described by Hölzer and Sjögren (1977). The charges could be reproduced better than 0.1% and the standard deviation of repeated punch force measurements was 75 N and of FD about 100 N at an applied force of 15 kN. The relative error depends on the absolute value of the forces involved. The signals during the ejection phase were increased by expansion amplifiers (workshop of AB Hässle) and the standard deviation in EJF was less than 8 N.

The die wall signals were expanded during the ejection phase and separate calibration curves were made as previously described (Hölzer and Sjögren, 1979b). The relative standard deviation in the die wall force was less than 3% at maximum pressure and about 6% at ejection.

Procedure

The materials were filled by hand into the die. The weight was calculated from the density (Beckman Air Comparison Pycnometer 930) to provide a tablet of 0.3 cm height at zero porosity unless otherwise stated. The tablet speed corresponded to 30 rpm which gave a punch speed during ejection of about 45 mm/s. Mixtures with magnesium stearate (0.2 mm sieve) were made in a 2 litre Turbula mixer (Willy Bachhofen, Switzerland) for

200 revolutions at 42 rpm. The relative humidity was between 45 and 55%.

The results presented are the mean of 5 tablets after conditioning the die wall with 10 tablets unless otherwise stated. The die and punches were cleaned with water, paper tissues and a mixture of acetone and carbon tetrachloride. Materials with great adhesion to the die wall, e.g. unlubricated anhydrous lactose or calcium diphosphate granulate could not be tabletted with the above-mentioned conditioning of the die wall. The die had to be cleaned before every second tablet. The tablet height after ejection was used for die wall force and μ_2 calculation as well for the measurement of the circumferential area. FD and EJF were calculated per unit contact area between the tablet and the die wall (Hölzer and Sjögren, 1978).

RESULTS

Variation of the friction parameters with pressure

Figs. 1 and 2 show the friction forces per contact area FD/A and EJF/A and the radial forces DWFM and DWFE plotted vs. UPP for 4 materials. NaCl and lubricated lactose are examples of "normal" materials while Avicel (batch A) and alprenolol (batch E) did not give ejection forces proportional to UPP.

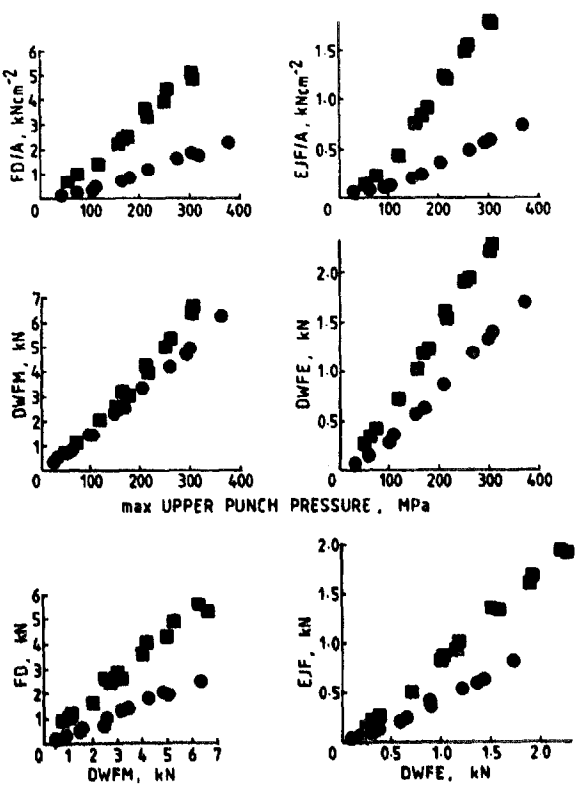

Fig. 1. Area compensated force difference (FD/A) and ejection force (EJF/A) vs upper punch pressure (UPP), die wall force at pressure maximum (DWFM) and during ejection (DWFE) vs UPP, and FD and EJF vs DWFM and DWFE, respectively. ■, NaCl; ●, lubricated lactose.

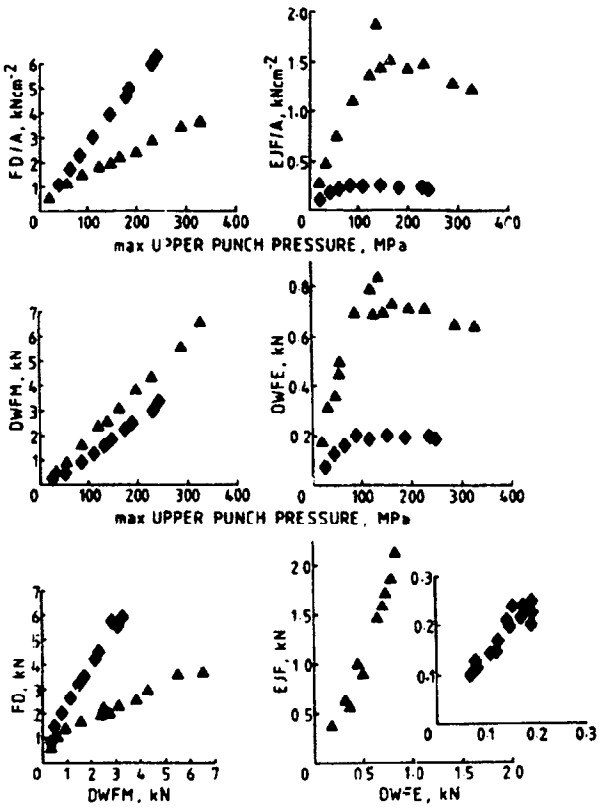

Fig. 2. FD/A, EJF/A, DWFM and DWFE vs UPP and FD and EJF vs DWFM and DWFE respectively. ▲, alprenolol; ◆, Avicel batch A.

DWFM increased nearly proportionally with UPP for all 4 materials and a yield point could be seen for NaCl and Avicel in accordance with reported results of other materials (e.g. Summers et al., 1976; Obiorah, 1978). DWFE increased proportionally to UPP for NaCl and lactose but not for Avicel and alprenolol. These two materials showed a relaxation of the radial stresses above a certain UPP level. In the case of alprenolol this was due to capping.

If the friction coefficient (μ) is reasonably constant over the pressure range tested a plot of fractional force vs. die wall force should yield a straight line without an intercept. The slope of this line will be μ. For NaCl and lactose FD vs. DWFM gave straight line plots and reasonably constant μ_1 values in the range studied. Avicel and alprenolol, on the other hand, gave decreasing μ_1 with increasing UPP. Plots of EJF vs. DWFE were linear for all 4 materials and μ_2 was constant in the range studied, see Fig. 3.

The two calculated friction coefficients, μ_1 and μ_2, were in good agreement for NaCl and lactose but differed considerably for alprenolol where μ_2 was higher than μ_1. As μ_2 is determined during the ejection phase it is a "kinetic" friction coefficient and should therefore be lower than μ_1 which is measured under "static" conditions (Bowden and Tabor, 1971, p. 323). The reason for this discrepancy in the coefficients for alprenolol may be adhesion of material to the die wall.

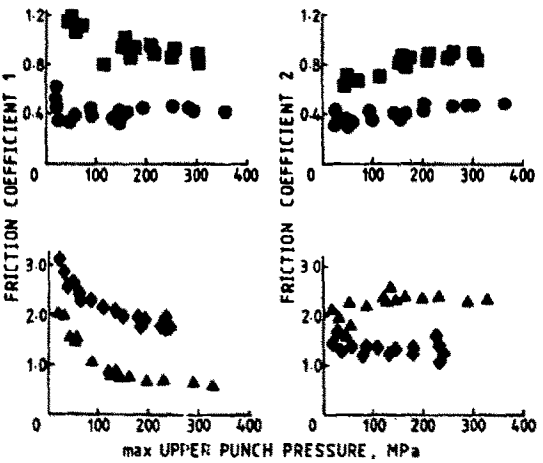

Fig. 3. Variation of μ_1 and μ_2 with UPP for the 4 materials shown in Figs. 1 and 2.

3. Conditioning the die wall

The die wall was cleaned as described above and when 20 tablets of NaCl were tabletted at 115 MPa UPP the "static" coefficient, μ_1, increased to a constant value of 1.4 (Fig. 4). Thereafter 20 tablets of lubricated NaCl (1% magnesium stearate) were compressed and μ_1 decreased rapidly to 0.4 and reached a constant value of 0.3 after a few tablets. When changing to unlubricated NaCl after the lubricated NaCl, μ_1 increased again but more than 30 tablets were needed before the starting value of 1.4 was reached. The change in μ_1 was due to the change in FD and not in DWFM. Magnesium stearate apparently forms a film on the die wall which is very resistant to abrasion by NaCl.

Unlubricated Avicel (batch B) was tabletted at 2 different pressures after cleaning the die wall as before (Fig. 4). 20 tablets were compressed at 60 MPa and μ_1 increased for each tablet. This indicates a lower friction at the die wall from the beginning. After the first 20 tablets the die wall appeared to be sufficiently conditioned as the following 30

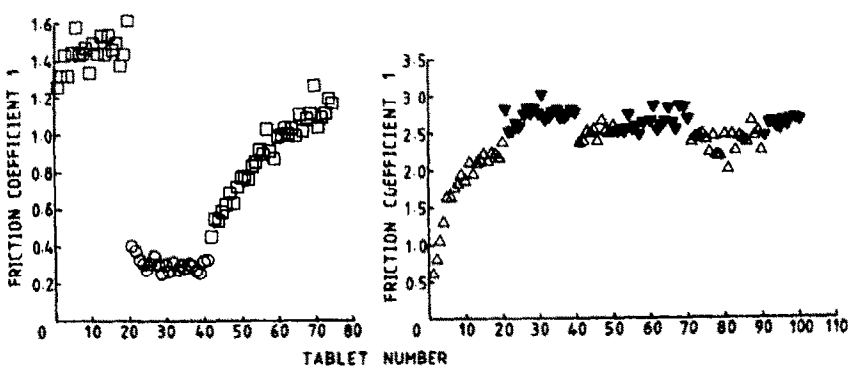

Fig. 4. Friction coefficients of consecutive tablets of: □, sodium chloride (0.43 mm) at 115 MPa; ○, sodium chloride (0.43 mm) + 1% magnesium stearate at 115 MPa; △, Avicel batch B at 60 MPa; ▼, Avicel batch B at 150 MPa.

tablets, compressed at 150 and 60 MPa, gave fairly constant μ_1 values. Cleaning after 50 tablets did not affect the μ_1 values as shown in Fig. 4. The DWFM values did not change and the change in μ_1 was due to the change in FD.

The die must be carefully cleaned before each new material but this procedure is obviously not sufficient to remove traces of lubricating film on the die wall. In general, conditioning the die wall with at least 10 tablets appears to be necessary.

Tabletting of unlubricated materials

In Table 1 the results from tabletting at 150 MPa UPP are given. According to Bowden and Tabor (1968, p. 399) the friction coefficient of stearic acid on bulk stearic acid varies between 0.5 and 0.1 depending on the normal load used. At the normal forces used in our study the friction coefficient of 0.1–0.2 is reported and consequently our results are in agreement with the literature values. The μ_2 values for stearic acid and magnesium stearate are very uncertain due to the very low die wall forces.

NaCl had μ_1 and μ_2 of 1.4 and 0.8 respectively. Bowden and Tabor (1968, pp. 118 and 126) give the friction coefficients 0.7–0.8 for NaCl (rock salt on steel).

The "static" μ_1 varies between 0.1 and 2.44 and the "kinetic" μ_2 between 0.35 and 3.15 for the different materials (Table 1).

It is interesting to note that different batches of a substance (alprenolol batches A–E) may have very different friction properties although all batches complied with the manufacturer's specifications. Two of the 5 alprenolol batches had considerably higher μ_2 values than the others but μ_1 was the same for all 5. These two batches also were more prone to capping.

TABLE 1

UNLUBRICATED MATERIALS TABLETTED AT 150 MPa UPPER PUNCH PRESSURE

Materials	μ_1	FD/A (kNm^{-2})	DWFM (kN)	μ_2	EJF/A (kNm^{-2})	DWFE (kN)
Alprenolol HCl batch A	0.72	1.92	2.82	1.47	0.68	0.51
B	0.72	1.90	2.76	2.14	0.90	0.47
C	0.72	2.00	2.87	1.54	0.64	0.46
D	0.75	2.01	2.77	1.68	0.54	0.36
E	0.75	2.22	3.11	2.36	1.50	0.73
Avicel PH 101, batch A	1.97	3.98	1.77	1.29	0.26	0.19
B [a]	2.61	4.65	0.98	1.83	0.31	0.10
Acetyl salicylic acid	0.88	2.29	2.80	1.15	0.14	0.14
Corn starch	0.84	1.94	2.40	0.81	0.07	0.10
Lactose anhydrous	2.25	4.19	2.35	2.68	2.52	1.19
Magnesium stearate	0.11	0.45	5.31	0.35	0.01	0.03
Paracetamol crystals	1.55	2.81	1.98	1.72	0.85	0.65
Sodium chloride (0.27 mm)	1.37	3.18	2.65	0.83	0.75	1.04
Stearic acid	0.10	0.43	6.60	0.37	0.01	0.06

[a] Height at zero porosity 0.15 cm.

Both Avicel batches had high μ_2 values though the ejection forces are low due to rapid relaxation of the radial force. For batch B, both μ_1 and μ_2 were higher than for batch A but no other obvious difference regarding tabletting was found. Avicel is probably a better antiadhesive than lubricant.

Paracetamol had rather high friction coefficients, probably due to adhesion to the die wall. ASA, corn starch and NaCl did not adhere to the die wall and had intermediate friction coefficients. NaCl gave a large FD value due to a high DWFM. Lactose adhered to the die wall and gave high μ_1 and μ_2 values.

Effect of lubricants on friction parameters

The friction forces, radial forces and friction coefficients for some materials with and without magnesium stearate are given in Table 2.

Addition of magnesium stearate reduced the friction for the materials and increased the radial force transmission during compression but reduced the remaining radial force after compression. Therefore the effects of magnesium stearate on the ejection force was due to both reduced radial force and reduced friction coefficient.

Addition of 0.1% magnesium stearate was not sufficient to prevent the adhesion of lactose to the die wall, although the tablets could be compressed without cleaning the die before every second tablet as was necessary with lactose alone. This probably explains the increased friction values obtained after the addition of the lubricant. Higher concentrations prevented adhesion and the friction was reduced as expected.

Well-lubricated materials gave friction coefficients between 0.2 and 0.4. Lubrication of

TABLE 2

MATERIALS TABLETTED AT 150 MPa UPPER PUNCH PRESSURE

Materials	μ_1	FD/A (kNm^{-2})	DWFM (kN)	μ_2	EJF/A (kNm^{-2})	DWFE (kN)
ASA 7013 + Sta-Rx1500 (9 : 1)	0.89	2.83	3.42	1.35	0.19	0.16
+ 1.0% Mg-Stear.	0.22	0.62	3.14	0.99	0.05	0.05
Avicel + ascorbic acid (1 : 1)	2.35	4.06	2.18	2.39	1.29	0.65
+ 1.0% Mg-Stear.	0.21	0.46	2.57	0.26	0.05	0.23
Ca-phosphate + Sta-Rx1500 (9 : 1)	2.16	4.07	2.53	1.34	1.58	1.69
+ 0.1% Mg-Stear.	0.67	1.35	2.66	0.73	0.17	0.33
+ 1.0% Mg-Stear.	0.29	0.64	2.86	0.30	0.06	0.28
Lactose anhydrous	2.25	4.19	2.35	2.68	2.52	1.19
+ 0.10% Mg-Stear.	2.44	4.39	2.23	3.15	3.98	1.62
+ 0.25% Mg-Stear.	0.34	0.68	2.39	0.38	0.18	0.61
+ 1.00% Mg-Stear.	0.26	0.55	2.60	0.42	0.21	0.63
Sodium chloride (0.27 mm)	1.37	3.18	2.65	0.83	0.75	1.04
+ Mg-Stear. (0.25%)	0.33	0.93	3.11	0.23	0.10	0.53
+ Mg-Stear. (0.50%)	0.31	0.87	3.13	0.23	0.09	0.47
+ Mg-Stear. (1.00%)	0.17	0.49	3.27	0.23	0.05	0.37
Sta-Rx1500 + anh. lactose, 7 : 3	1.98	4.36	2.58	2.23	0.66	0.38
+ 1.00% Mg-Stear.	0.19	0.52	3.15	0.35	0.02	0.07

ASA had a surprisingly small effect on μ_2 but the ejection force was very low due to the relaxation of the radial stress.

DISCUSSION

When the slider is repeatedly dragged over the support along the same path, an identical frictional force and a reproducible friction coefficient should be found. According to Bikerman (1976) there are 4 friction mechanisms in this group: (1) hydrodynamic lubrication in which the distance between the two solids is so large that they have no effect on the behaviour of the fluid passing between them; (2) boundary lubrication when the distance between the solids is so small that the surface roughness affects the fluid (lubricant) between them; (3) dry friction when the above-mentioned distance is very small, the viscosity of the "lubricant" is very low and the resistance to sliding follows the "law of friction", i.e. the friction coefficient is independent of the geometrical contact area, independent of the normal force and is reproducible; (4) the case in which elastic deformation changes the areas of at least one of the "bodies", either the support or the slider. The law of friction is not valid but the friction force is reproducible and often depends on the mechanical properties of the materials. Additional mechanisms when μ cannot be reproduced properly are mentioned in the literature (Bikerman, 1976); (5) when the slider or the support is plastically deformed and the friction coefficient changes every time the slider passes the support; (6) the support or the slider not only deforms but also ruptures during sliding (Bikerman, 1976); and (7) the situation may be complicated as the frictional heat may cause deformation or chemical changes and the friction force will be very difficult to reproduce.

Materials lubricated with magnesium stearate, e.g. lubricated lactose or NaCl which gave reproducible friction forces, probably fall into the 3rd category. Polymers, e.g. Avicel, after conditioning the die wall probably belong to group 4, The friction coefficient of Avicel alters with UPP probably because of elastic deformation of the tablet when the latter slides across the die wall. Avicel PH 101 consists of cellulose fibers the friction properties of which is reported to vary with the direction of the fiber (Bowden and Tabor, 1971, p. 170). Materials with adhesion problems such as unlubricated lactose, calcium, phosphate and alprenolol belong probably to group 6. Seizured surfaces on the lactose, calcium phosphate and alprenolol tablets could be seen. For such materials it is generally difficult to determine the friction coefficients and μ_2 is often higher than μ_1. Our results with unlubricated lactose and alprenolol indicate that the friction is measured between different surfaces during compression and ejection.

For most tablet masses the friction coefficient could be measured with acceptable precision at compression and ejection. The "static" friction coefficient varied with the tabletting pressure for some materials but the "kinetic" friction coefficient was almost independent of the pressure for the materials investigated.

Determination of friction coefficients contributes to a better understanding of lubricating mechanisms and it may also be useful in the characterization of the tabletting properties of substances or tablet masses.

ABBREVIATIONS

UPF = max upper punch force;
LPF = max lower punch force;
UPP = max upper punch pressure;
FD = force difference, (UPF–LPF;
DWFM = max die wall force at compression;
REF = remaining force on lower punch before ejection;
EJF = ejection force when lower punch is moving smoothly;
DWFE = die wall force at ejection;
μ_1 = friction coefficient at compression (FD/DWFM);
μ_2 = Friction coefficient at ejection, (EJF/DWFE).

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