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COMPARISON OF METHODS FOR EVALUATION OF FRICTION DURING TABLETING

Arne W. Hölzer and John Sjögren
Pharmaceutical Laboratory, AB Hässle
Fack, S-431 20 Mölndal, Sweden

ABSTRACT

The friction between a tablet and the die wall can be evaluated by comparison of the forces on the upper and lower punches, i.e. force ratio (R-value) or force difference (FD), or by measuring the forces on the lower punch immediately before ejection (REF) or during ejection (EJF). These parameters were compared for different materials using an instrumented single-punch press. The compaction load and the dimensions of the compact had an obvious influence on all parameters studied. By correcting for differences in contact area between tablet and the die wall it appears possible to eliminate the influence on FD, REF and EJF from variation in tablet height. Several compaction loads within the range of interest have to be studied to get a complete picture of the behaviour of a tablet granulate. The different parameters did not always give correlating results and EJF appears to give the best prediction of adhesion problems.

INTRODUCTION

Developments within electronic technology have made it comparatively easy to study the forces exerted during the cycle of tablet compression. One of the obvious uses of this technique is the evaluation of friction between the tablet and the die wall. Such studies may be useful for evaluation of lubricants, optimizing tablet formulations, detection of potential risks of production trouble, etc.

Various principal techniques have been suggested: measuring the degree of force transmission from the upper to the lower punch during compression, the elastic force remaining on the lower punch when the upper pressure has been removed and the force necessary to eject the compact from the die. The energy losses due to friction have also been subjected to direct studies, either by measuring the heat developed (5) or by calculation of the force - displacement integrals (6, 7, 8). The first two principles can be used only on tablet presses where the force is applied by the upper punch moving towards a stationary lower punch.

The degree of force transmission can be calculated either as the ratio between the peak forces on the lower and on the upper punch (R-value) (1) or as the difference between the two (FD) (2). Both methods have been frequently used. Nelson et al. (1) reported as long ago as in 1954 that the compressed tablet exerted a force on the lower punch after the upper punch left contact with the tablet and this remaining force (REF) has been suggested as a measure of the friction between tablet and die (4). The ejection force (EJF),i.e. the maximum force on the lower punch during ejection of the tablet from the die, appears to be the most commonly used parameter in studies on tablet friction (3).

Many papers have been published but few details are given on the influence of experimental factors on the results and on how different methods correlate. The aim of this study was to compare some common methods and to establish suitable experimental conditions for the studies.

EXPERIMENTAL

Equipment

We used a single-punch press[a] with flat punches of 8.0 and 11.3 mm diameter. Calibrated piezoelectric force transducers[b] were inserted between the punch collars and the shortened punch holders. The signals from the force transducers were amplified by a charge amplifier[c] and recorded by 500 Hz mirror galvanometers[d]. To enable better determination of the remaining and the ejection forces the lower punch force signal was also amplified ten times in an expansion amplifier[e]. Each galvanometer was connected with a protective circuit[e] to prevent damage by excessively high signals. A potentiometer, introduced in series with each charge amplifier output and the UV-recorder, enabled adjustment of the signals from the amplifiers. An electronic calibration unit[e] consisting of a high quality capacitor (9 400 pF ± 1 %) was used to calibrate the charge amplifiers and the galvanometers. By giving exactly known voltage inputs from a stabilized rectifier potentiometer unit[e] known charges could be delivered to the amplifier units. The whole charge range of interest was calibrated. An inductive displacement transducer[f] was used to record the upper punch displacement.

Procedure

The punches and force transducers were locked by the punch retaining screws with a very low force and the die was fastened carefully so that the lower punch could move freely. The adjustment of the force transducers was checked before each experiment by pressing the punches towards each other and comparing the galvanometer recordings. At the beginning of each experiment approximately 50 tablets were run at the highest compression load in order to condition the die wall. During the experiment ten tablets were compressed at each force range before five recordings were taken. After each experiment the die and punches were cleaned with hot water and polished with soft paper tissues. Each series was repeated on another occasion and the values given in figs 1-4 represent the mean values. The experiments were performed at an air humidity of 30-40 % R.H. The machine speed appeared to have a negligible effect on the results and we used a speed of 30 tablets per minute. The tablet thickness was measured with a micrometer calliper immediately after compression and the galvanometer recordings were measured with slide callipers.

Materials

Four materials suitable for direct compression were studied: anhydrous lactose U.S.P.[g], acetylsalicylic acid B.P.[h], microcrystalline cellulose N.F.[i] and a granulated corn starch[k]. In some experiments the anhydrous lactose powder was lubricated with magnesium stearate U.S.P.[m].

The pure materials were passed through a 0.7 mm sieve. The magnesium stearate was passed through a 0.2 mm sieve and was mixed into the lactose with a paper card in a bowl for 5 minutes and the mixture was finally passed through a 0.7 mm sieve.

RESULTS

Pressure and Tablet Dimensions

A mixture of lactose and 0.25 % w/w magnesium stearate was compressed at different loads and to different tablet dimensions. The R-value, the force difference (FD), the remaining force (REF) and ejection force (EJF) were determined.

The R-value was clearly dependent on the compression load, as shown in fig. 1. At higher pressures a relatively greater part of the applied force was transmitted to the lower punch. Thicker tablets gave lower R-values but the tablet diameter appeared to be less important. The difference between the upper and lower punch forces (FD) was almost linearly related to the compaction load, see fig. 1. At corresponding pressures thinner tablet gave lower FD-values than thicker ones. To reduce the influence of the tablet

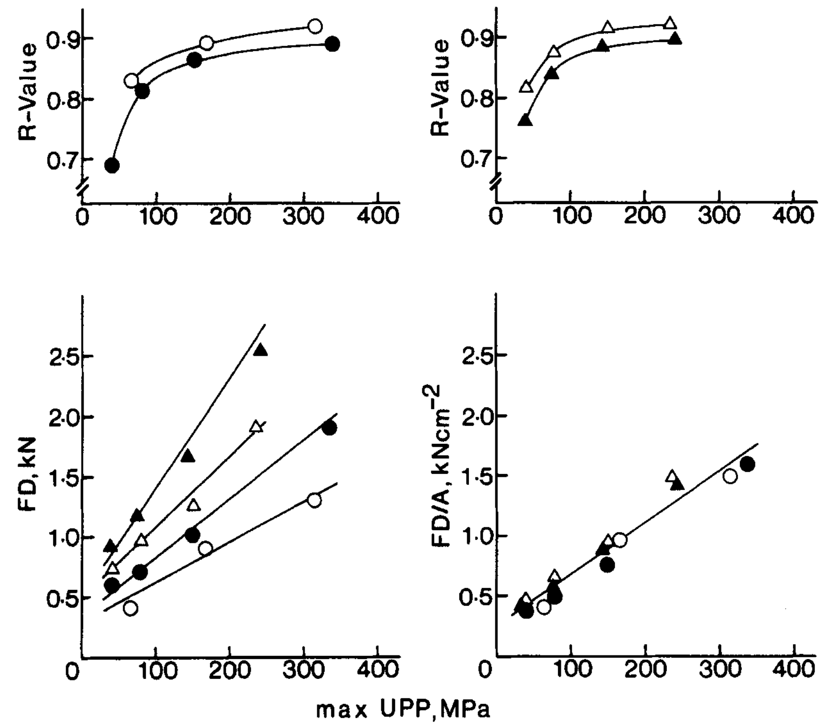

FIGURE 1

Punch force ratio (R-value), force difference (FD) and FD per die wall contact area (FD/A) at various applied pressures (UPP) for tablets of different dimensions.
Material: Lactose + 0.25 % Mg-stearate.

○	Tablet weight 0.25 g	Diameter 0.80 cm
●	" " 0.35 g	" 0.80 cm
△	" " 0.50 g	" 1.13 cm
▲	" " 0.70 g	" 1.13 cm

dimensions, the force difference per contact surface area between tablet and die wall was calculated as suggested by Rees & Shotton (9). Plots of these area-compensated force differences (FD/A) *vs*. upper punch pressure (UPP) yielded almost the same straight line for all tablet dimensions.

The remaining force (REF) and the ejection force (EJF) behaved in a similar way as FD (see fig. 2). The higher the compaction load and the thicker the tablets the greater were the REF and EJF values. Compensation for the contact surface areas in the same way as above reduced the influence of the tablet thickness and when plotted against UPP the results for each diameter could be approximated by a single straight line.

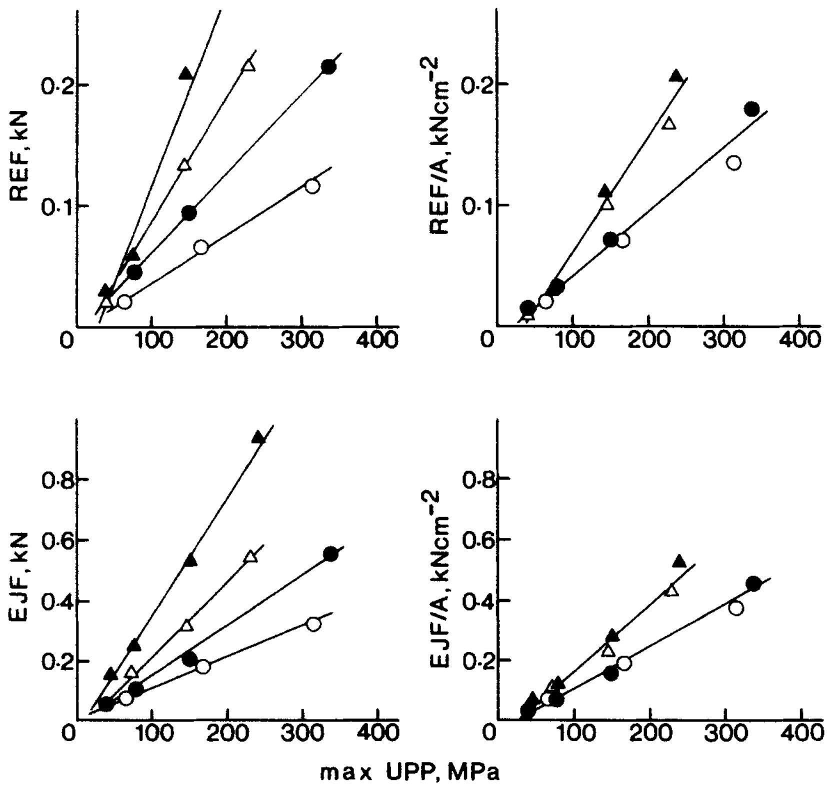

FIGURE 2

Remaining force (REF), ejection force (EJF), REF and EJF per die wall contact area (REF/A and EJF/A resp.) at various applied pressures (UPP) for tablets of different dimensions. Material and symbols see fig. 1.

Lubrication

The effect of addition of magnesium stearate on the frictional properties of lactose was studied in a second series of experiments and fig. 3 summarizes the results. The tablet weight was calculated from the true density of the materials to give tablets 3.05 mm

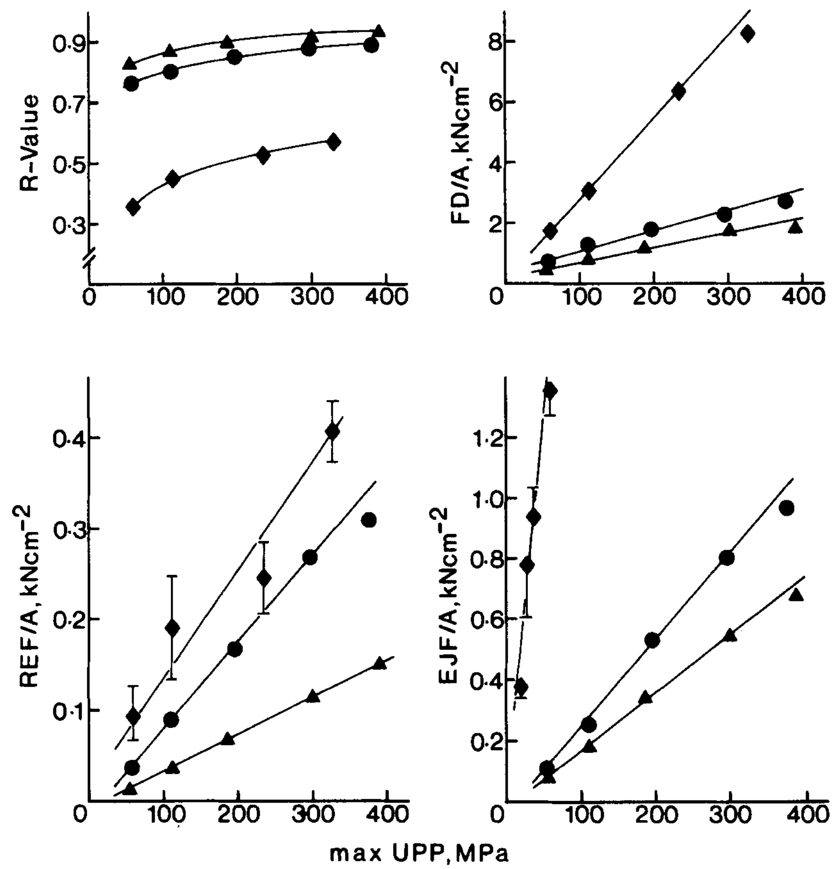

FIGURE 3

Effect of Mg-stearate additions on R-value, FD/A, REF/A and EJF/A at various applied pressures (UPP).

◆ Lactose
● " + 0.10 % Mg-stearate
▲ " + 0.75 % Mg-stearate

thick at zero porosity. Pure lactose gave severe adhesion to the punches and die wall and they had to be cleaned with water and paper tissues after every third tablet. The R-value increased with higher lubricant concentration, as expected. The influence of the pressure was most pronounced for the unlubricated lactose. The area-corrected values of the force difference (FD/A) gave straight lines for the three compositions when plotted against UPP. The slope of the lines was lower at higher stearate concentrations, indicating lower frictional losses. Plots of REF/A and EJF/A vs. UPP were linear over a large range of pressures. Unlubricated lactose showed a remarkably great scatter in the REF/A plot. The addition of only 0.1 % Mg-stearate eliminated this scatter. All the methods of estimating the lubricating effect of magnesium stearate gave the same qualitative picture, i.e. more stearate, less friction.

Substances

In another series of experiments lactose, acetylsalicylic acid, microcrystalline cellulose and granulated corn starch were studied in the same way. The tablet weight was chosen as in the previous series except for microcrystalline cellulose where the zero porosity height was 2.25 mm due to the low bulk density of this material. With the exception of lactose all materials could be tableted without problems. The results are given in fig. 4.

The R-value increased with pressure for all the four materials but less for cellulose than for the other substances. The ranking order of the materials according to their R-values was consequently dependent on the pressure level chosen. At low pressures, e.g. $<$ 100 MPa, cellulose gave the highest R-values while it gave the second lowest at higher pressures. Plots of FD/A against UPP were linear over the whole pressure range for microcrystalline cellulose and lactose but were linear only below about 150 MPa for the other two substances. Surprisingly enough, all four materials gave similar FD/A values at normal tableting pressures (50-150 MPa).

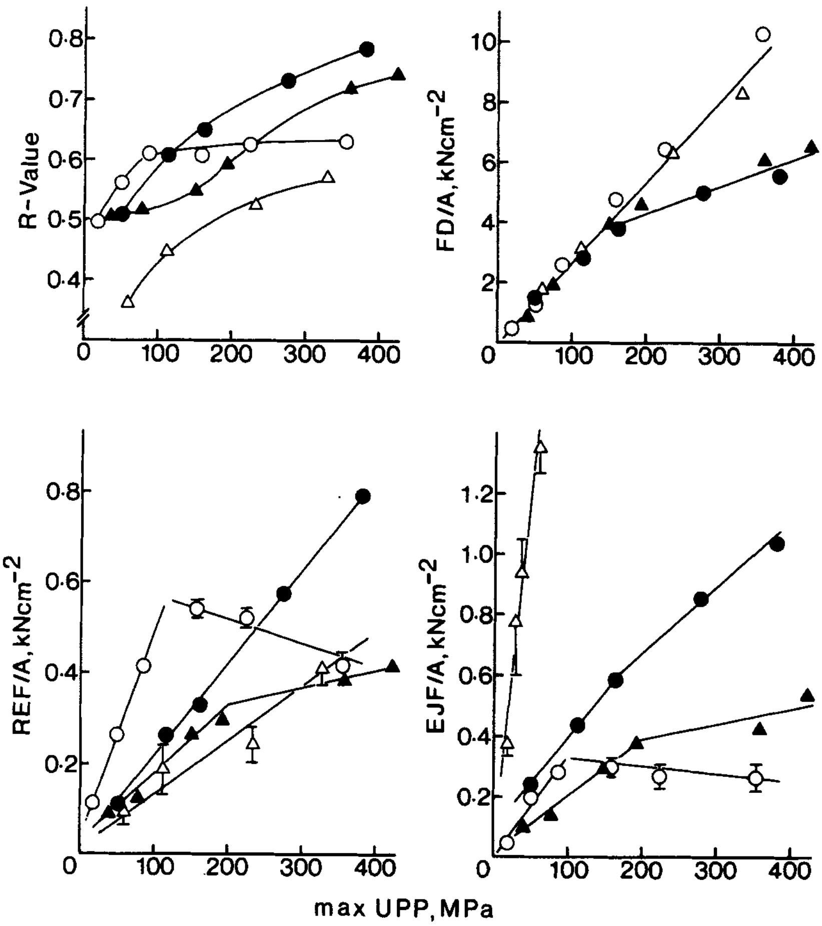

FIGURE 4

R-value, FD/A, REF/A and EJF/A at various applied pressures (UPP) for different substances.

- ○ Microcrystalline cellulose
- ● Acetylsalicylic acid
- △ Lactose anhydrous
- ▲ Corn starch, granulated

Plots of REF/A against UPP were linear up to about 150 MPa for acetylsalicylic acid and starch, and up to about 100 MPa for microcrystalline cellulose and no such scatter as for lactose was seen. At higher pressures, however, the cellulose values scattered considerably and did not increase with pressure. Except for lactose, the results of the ejection force calculations were very similar to those of the REF/A.

DISCUSSION

When a powder mass is exposed to an axial force the pressure is not only transmitted in an axial direction but also radially. The ratio of radial to axial stress during a tablet compression cycle has been studied, for example, by Long (10) and Carless & Leigh (11), who showed a linear relationship between the radial and axial stresses up to a certain limit when the axial pressure was increased. After maximum pressure the radial stress fell more slowly than the axial stress and a certain radial force still remained after the axial force had ceased. This remaining force was related to the maximal load applied but depended on the properties of the tablet material as well (Higuchi et al. 12, Long 10).

According to Amontons´s law (18) the frictional force between a tablet and the die wall should be dependent on the radial pressure exerted by the tablet and the contact surface area with the die. The applied axial load can therefore be expected to have great influence on the friction and all parameters studied in our investigation proved to be clearly pressure dependent. The ratio between lower and upper punch pressure (R-value) might be independent of the applied pressure but our results, as well as previously published data (13), show that the R-value usually increased with increasing pressure. This may partly be explained by the decrease of the contact surface area due to higher pressure (14, 15) but a simple way of compensating for these changes could not be found in our experiments.

The other three parameters studied were approximately linearly correlated to the applied pressure, at least within a limited range.

The contact surface area between tablet and die wall had the expected influence on the results and calculation of the forces per unit area reduced the influence of the tablet dimension considerably, which is in agreement with earlier experiences (1, 9, 19). As long as the same punch and die set is used it appears possible to compare results obtained with tablets of different heights by using this correction.

The linear parts of the plots of FD/A, EJF/A or REF/A _vs_. UPP generally gave intercepts when extrapolated back to zero UPP. Consequently, there is not always a direct proportionality between these parameters and UPP and the results cannot easily be corrected for differences in the applied load. In our opinion, measurements of tablet friction should therefore be performed at several different pressures within the range of interest.

The different parameters did not give a unanimous picture of the frictional properties for all materials investigated and this is in itself not surprising as they measure the friction during different phases of the compression cycle. The R-value and FD/A are measured at the pressure maximum and consequently give an estimation of the friction during the actual compression stage. De Blaey et al. (7) and Juslin & Järvinen (8) have suggested measuring the total work of friction calculated as the integral of FD to upper punch displacement or a function of this integral. In some of our experiments we tested whether these integrals might give another picture than FD but the integrals correlated very well with the maximum force differences and gave no further information. For estimation of the frictional losses during the compression of a tablet granulate, FD/A seems to be the most suitable parameter but it appears to be of limited value for the prediction of other tableting problems. Compare, for instance, the insignificant difference in respect of FD/A between lactose and the other materials in fig. 4. Lactose tablets gave severe adhesion to the punches and die while the other materials gave no great problems.

The remaining force on the lower punch is caused by the elastic expansion of the tablet, and of various machine parts. The reproducibility in REF/A depends very much on the die being closely fastened and being unable to move upwards. Provided that the friction between the lower punch and the die wall is negligible, the elastic force in the axial direction can never exceed the magnitude of friction between the tablet and the die wall. The use of REF/A as a measure of friction is based upon the assumption that the expansion force is equal to or exceeds the friction force so that an equilibrium between these two forces is achieved. This is obviously not always the case. Unlubricated lactose, particularly, gave REF/A values far below the expected ones. Tablet masses which bind to strong tablets are usually characterized by a large remaining radial force (16, 17) and this may explain why the microcrystalline cellulose gave relatively high REF/A values at low pressures. The elastic expansion after compression at higher loads will probably reduce the radial pressure and explain the great scatter in this region. EJF/A also showed the same pressure dependency for cellulose and the correlation between REF/A and EJF/A was very good ($r = 0.984$). In this case REF/A was always higher than EJF/A, in contrast to the other materials. Of the parameters studied, EJF/A seems to give the best prediction of risk for adhesional problems.

CONCLUSIONS

All parameters used for studies on tablet friction are influenced by the compaction load and by the dimensions of the compact. To compare results from different materials or from different studies it is consequently necessary to standardize carefully the experimental conditions or to correct for the variations. Differences in the tablet height can be corrected for by calculating the friction per unit area. As the relationship between the studied friction parameters and the applied load may vary for different materials, the studies have to be performed at several levels in the range of interest.

The studied parameters correlate to each other only to a certain extent. FD/A or R give estimation of the frictional losses during the compaction of the materials and EJF/A of the losses during the ejection of the tablet from the die. EJF/A appears to give the best prediction of the tendency to stick to the die wall. REF/A seems to be less suitable for this purpose.

ABBREVIATIONS

EJF	Ejection force
EJF/A	Ejection force per unit area
FD	Force difference
FD/A	Force difference per unit area
MPa	Pressure unit, 1 MPa = 1 MNm^{-2} or 145 Psi
REF	Remaining force
REF/A	Remaining force per unit area
R-value	Punch force ratio
UPP	Upper punch pressure

FOOTNOTES

a Single punch press type KS, Kilian & Co GmbH, Köln-Niehl

b Kistler type 902A. Kistler Instrumente AG, CH-8408 Winterthur, Switzerland

c Kistler type 5001, address see above

d UV-recorder Ultralette 5656. Mirror galvanometers type 5831/N 500 Hz. Atlas Copco ABEM AB, S-121 20 Bromma 20, Sweden

e Made at the workshop of AB Hässle, Fack, S-431 20 Mölndal, Sweden

f Transducer PR 9314/10, Phillips Industry, Eindhoven, Holland

g Lactose anhydrous U.S.P., Sheffield Chemical Co., Norwich, New York, USA

h ASA Monsanto 7023 B.P., Monsanto Chem. Rouabon, U.K.

i Avicel[R] PH101 N.F., FMC Corp. American Viscose Division, Markus Hook, Pa., USA

k STA-Rx[R] 1500, A.E. Staley Mfg Company, Decatur, Ill., USA

m Magnesium stearate U.S.P., Unem 4850, Unilever-Emery Gouda, Po box 2, NV, Holland

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