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Work of friction and net work during compaction

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Series of tablets were compressed in a reciprocating tablet machine with a gradually increasing die wall friction. The force needed on the upper punch to maintain the tablet dimensions constant increased with the die wall friction while the lower punch force decreased. The change in punch forces due to differences in die wall friction had no effect on the tablet strength. The net work of compaction should be constant under these circumstances. The net work calculated by subtracting the work of friction and the expansion work from the gross work input was constant when the frictional work was calculated according to one of the two equations proposed in the literature (Järvinen & Juslin 1974) while the other appears to give an overestimation of the work of friction.

For the compaction of materials and formation of strong compacts, energy is needed and it seems logical to correlate the properties of the compact with the energy input rather than the compression pressure. The energy used during compaction can be calculated from punch forces and displacements. The total energy input is used for particle rearrangement, interparticle friction, friction with the die wall, elastic deformation, plastic deformation, fragmentation and formation of bonds. The work needed for particle rearrangement and to overcome interparticle friction has been considered negligible (de Blaey & Polderman 1971). The friction against the die wall is one of the factors which should be known in studies of the compaction process. The measurement of energy input has been used by several workers, as recently reviewed by Krycer et al (1982), and attempts have been made to derive a net energy input which does not include the work needed to overcome friction and elastic deformation.

De Blaey & Polderman (1971) have suggested that the work of die wall friction could be calculated according to equation 1.

$$\int_{D_S}^{D_M} (\mathrm{UPF} - \mathrm{LPF}) \quad \mathrm{dD} \qquad (1)$$

D = the displacement of the upper punch, measured relative to the lower punch,

D_S = the point at which the force rises from zero,

D_M = the maximum displacement of the upper punch,

UPF = the upper punch force,

LPF = the lower punch force.

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This method of calculating the work of friction was criticized by Järvinen & Juslin (1974), who pointed out that the point of action of the frictional forces does not coincide with the movement of the upper punch. Only particles in the upper layer of the compact will move the same distance as the upper punch while, for example, particles at the lower punch surface will be stationary.

The following equation for the work of friction was derived:

$$\int_{D_S}^{D_M} \mathrm{UPF} - (\mathrm{UPF} - \mathrm{LPF})/\ln(\mathrm{UPF}/\mathrm{LPF}) \, \mathrm{dD} \qquad (2)$$

The same abbrevations are used as in equation 1. The question regarding the validity of the two equations has been the subject of further discussion (Lammens et al 1980; Järvinen & Juslin 1981). The confusion still remains, however, as few experimental data are available.

If in a series of tablet compressions, it were possible to keep all experimental factors except the die wall friction, constant, the true net work should be constant even though the friction varies. Calculation of the net work, by subtracting from the upper punch work the work of expansion and the work of friction, should consequently yield constant results over a range of friction if the frictional work is calculated correctly. It should be possible to get very close to constant compaction conditions with a variation only in the die wall friction, by prelubricating the die with magnesium stearate, which forms a rather resistant lubricant layer. When a series of tablets of an unlubricated material are compressed, the layer will gradually be worn off, giving a gradual increase in die wall friction (Hölzer & Sjögren 1981).

The aim of this work was to investigate which of the two proposed equations for work of friction gives the best fit when used in net work calculation.

MATERIALS AND METHODS

Materials

Sodium chloride (99·6% NaCl, 0·2% Na_2SO_4, AKZO Zout Chemi, Hangelo, Holland), cubic crystals, sieve fraction 0·250–0·315 mm. Anhydrous lactose U.S.P. (Sheffield Chemical, New Jersey, U.S.A.), sieve fraction 0·250–0·315 mm. Saccharrose (Svenska Socker AB, Sweden), sieve fraction 0·400–0·500 mm. Magnesium stearate U.S.P. (Unem 4850, Unilever Emery, Gouda, Holland).

Methods

The materials were compressed with flat 1·13 cm circular punches in a reciprocating tablet machine. The machine was equipped as described by Hölzer & Sjögren (1979), with piezoelectric load washers on the upper and lower punches and strain gauges at the die wall. Inductive displacement transducers (Hottinger Baldwin Messtechniks W 10, amplifier KWS 3073) were used to measure the punch movement.

The tablet weight for each material was chosen so as to give a tablet height of 4·0 mm at an upper punch pressure of 150 MPa when compressed in a prelubricated die. The tablet weights were 0·819 g for sodium chloride, 0·551 g for anhydrous lactose and 0·583 g for saccharrose.

Before each series, the die wall was prelubricated by compressing a tablet of the test material mixed with 25% magnesium stearate at an upper punch pressure of about 150 MPa. The compression was repeated 5 times to build up a uniform lubricant layer on the die wall and excessive amounts of powder materials were removed with a soft brush and a vacuum cleaner.

The unlubricated test materials were weighed (±0·5 mg), filled by hand into the prelubricated die and compressed. Series of about 15 tablets were compressed without any further adjustment of the equipment. The series were ended when the friction coefficient at the pressure maximum, μ_1 (Hölzer & Sjögren 1981), reached a high and constant level or the tablet machine emitted a groaning noise at the ejection of the tablets.

Upper and lower punch force, upper punch displacement and die wall force were measured during each compression. The analogue signals given each millisecond from the measurement devices were transformed into digital form by an AD-converter (Digital AD8-A, Digital Equipment Corporation). The data were transformed into force and displacement units and stored in the memory of a computer and were thereby available for further calculation, e.g. according to equations 1 and 2.

The upper punch work was calculated by integration (trapezoidal method) of upper punch force versus upper punch displacement. The integral of lower punch force versus upper punch displacement, which was termed 'lower punch work' by de Blaey & Polderman (1970), was also calculated. The term 'lower punch work' is inadequate since the lower punch cannot produce any physical work as long as it is stationary but it is used in this paper to facilitate comparison with earlier studies. The work of expansion was estimated from the decompression curve after a single compression. The tablets were stored for at least 3 days at about 35% RH before diametral crushing strength was measured and the tensile strength was calculated according to Fell & Newton (1968).

RESULTS AND DISCUSSION

When a series of tablets of the unlubricated materials was compressed in a previously lubricated die the friction coefficient increased as the lubricant layer wore off. The maximum upper punch force (UPF) increased at the same time and the maximum lower punch force (LPF) decreased (Fig. 1a–c). For sodium chloride, which can be compressed without lubrication, the friction coefficient tended to reach a plateau level after about 12 tablets, as did UPF and LPF. The saccharrose adhered to the die wall as the lubrication decreased and this may have altered the conditions to some extent but the results were similar to those obtained with the other two materials.

As the die wall friction increases, more force will be needed on the upper punch to compress the material to the same tablet height. The force on the lower punch is, however, reduced and less force is transmitted to the punch in spite of increasing upper punch force. This is in accordance with the results reported by Järvinen & Juslin (1974). They used lubricated and unlubricated dies but changed the maximum forces to get approximately the same compressional work. This change in the experimental conditions has been stated to be a possible source of error by Lammens et al (1980), who found constant LPF when changing the die wall friction by using different punch diameters. In our study no changes in the machine settings were made.

In Fig. 2 two examples of force-displacement curves are given. The examples show the first and the last tablet in the series with anhydrous lactose. The

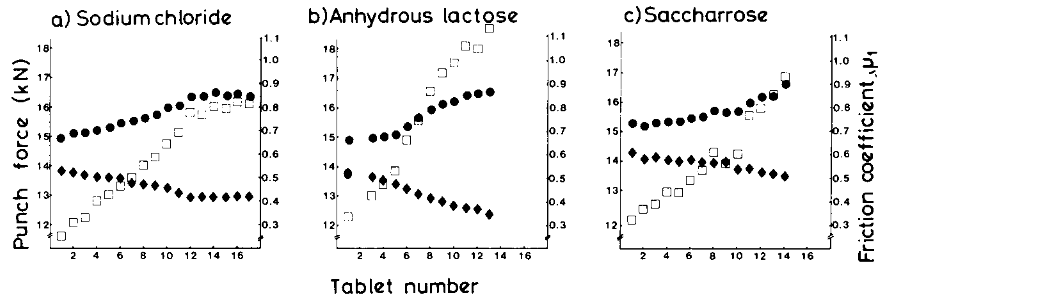

FIG. 1a–c. Punch forces and friction and coefficients of consecutive tablets. ●, maximum upper punch force; ◆, maximum lower punch force; □, friction coefficient, μ_1.

area under both the upper punch force curve (UPW) and the lower punch force curve (LPW) changed when the die wall friction was increased. UPW increased and LPW decreased for all materials, as can be seen in Fig. 3a–c. These results differ from the findings of de Blaey & Polderman (1970). They obtained the same LPW when compressing a lactose granulate in a prelubricated and an unlubricated die. In a later paper (1971), however, the authors have stated that the precision of the method used to calculate the work was fairly low.

The work of expansion (EXW) of sodium chloride was less than 0·04 Nm and these low figures were negligible in the calculation of net work. EXW of saccharrose and anhydrous lactose was higher but decreased slightly within each series, probably due to the friction against the die wall, which will retard the expansion of the tablet (Fig. 3b–c). De Blaey & Polderman (1970) have suggested that the work of expansion should be measured by using a double compression technique. The tablets will thereby be given more time to expand and the influence of die wall friction will be less. When the die wall friction is high, neither the single nor the double compression technique will give the complete EXW, due to incomplete expansion of the tablet. With prelubricated dies or lubricated materials, on the other hand, only small differences between the two methods have been obtained (de Blaey & Polderman 1970). There therefore seems to be little reason to use the more complicated double compression technique, especially as a second compression might also include some plastic deformation (Krycer et al 1982) and the properties of the compact may change during repeated compression (Armstrong et al 1982). Comparison of the tablet height at the end of the decompression with the tablet height after ejection, showed that only about 55 and 65% of the total axial expansion of saccharrose and of anhydrous lactose respectively took place in the die when the die wall was well lubricated. These values decreased to about 40% when the die wall friction increased. Since the tablets will also expand in the radial direction after ejection, it seems reasonable to conclude that the calculated EXW will underestimate the expansion work, especially when the friction is high.

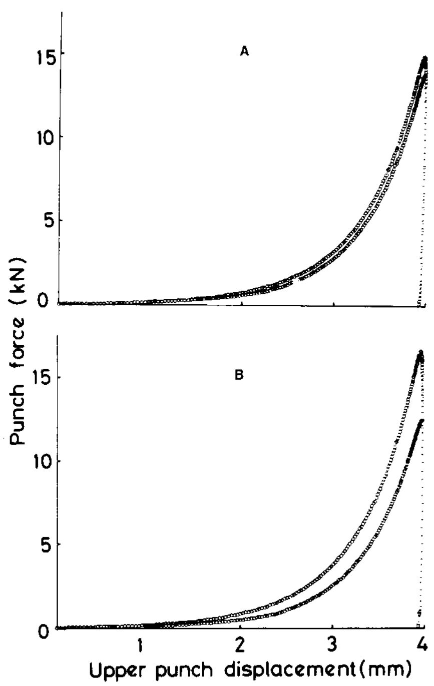

FIG. 2. Upper punch force (upper points) and lower punch force versus upper punch displacement for the first (A) and last (B) tablet in the series of anhydrous lactose tablets.

The inaccuracy in the net work calculation caused by the influence of die wall friction on the EXW (see Fig. 3b–c) was reduced by using the EXW from the first well-lubricated tablets (μ_1 <0·5) throughout the series.

The net work of compression obtained by subtracting from the UPW both EXW and the friction

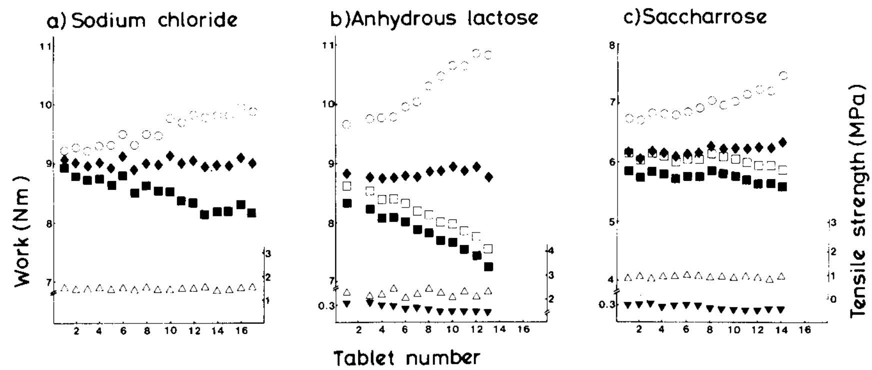

FIG. 3a–c. Work of compaction and tensile strength of consecutive tables. ○, upper punch work; □, 'lower punch work', ◆, net work according to Järvinen & Juslin (1974); ■, net work according to de Blaey & Polderman (1971); ▼, expansion work; △, tensile strength. The expansion work in (a) was negligible and hence □ and ■ coincide.

work, calculated either according to Järvinen & Juslin (1974) or according to de Blaey & Polderman (1971), is given in Fig. 3a–c. The former calculation yielded an approximately constant net work of compression in spite of increasing friction, while the latter gave a decreasing net work. Since the experimental set-up was chosen so as to maintain all factors, except die wall frictions, contant, the net work should be constant within a series. The tablet strength for each material was unaffected by the increase in die wall friction (Fig. 3a–c), which supports the assumption that all conditions except the friction were practically constant.

All tablets in a series expanded to approximately the same tablet height after ejection, which indicates that the expansion work was independent of the die wall friction. Even though the method of measuring EXW underestimates the true expansion work, the magnitude of the calculated net work should not be affected by increasing friction.

Our conclusion is therefore that the work of friction calculated according to Järvinen & Juslin (1974) gives a more correct estimate than the equation proposed by de Blaey & Polderman (1971). It also seems reasonable from a theoretical point of view to expect that the latter method overestimates the work of friction. Since the friction is caused by the contact between the particles in the compact and the die wall, the movement of these particles and not the movement of the upper punch should be used in the calculation of the work of friction.

The Järvinen & Juslin method is based on a simplified model concerning the movement of particles during compaction but our results strongly support the conclusion that it gives a better estimation of the work of friction. The frictional work values in this study were very close to half of those calculated according to equation 1, and this approximation would be a reasonably accurate estimate of the work of friction for most purposes. The approximation seems to be valid not only at low friction (Järvinen & Juslin 1974) but also at high friction levels. The deviations from the values obtained according to equation 2 were only about 3% in the low friction range ($\mu_1 < 0{\cdot}5$) and about 4% in the higher friction range ($\mu_1 = 0{\cdot}5$–$1{\cdot}2$).

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