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The effect of crystal hardness on radial pressure at the wall of a tabletting die

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The force transmitted to the die wall during the tabletting of seven crystalline pharmaceutical materials has been measured using a photoelastic technique. The Vickers hardness of these materials has also been measured. The compounds examined were aspirin, urea, hexamine, salicylamide, potassium chloride, sodium chloride and sucrose. The pressure on the die wall increases as the hardness value decreases. The Vickers hardness of the resultant tablets was also measured; aspirin and sodium chloride both showed the occurrence of work hardening in that the tablets had a greater hardness value than the parent crystalline substance.

The radial force exerted on the die-wall by a powder under compression in a tabletting die is of great interest in any study of the tabletting process. The larger the radial pressure, the greater will be the amount of friction and shear at the die-wall, and the greater the ejection force needed to remove the tablet from the die. It would be expected that the radial pressure would be greater for softer substances, since they approximate more closely to the idealized case of a liquid, which has zero hardness. Here the pressure would be hydrostatic, so that the radial pressure would be equal to the pressure exerted at the punch face. The purpose of the work reported here was to examine the relation between radial pressure and Vickers hardness by making tablets from a number of substances whose hardness was also measured, the compaction being carried out in a die of a type allowing the radial pressure to be ascertained.

The method of Ridgway (1966) was used. It involves the application of photoelastic stress analysis using the stress patterns observed by means of polarized light in the wall of a Perspex die during compression, as a method of measuring radial pressure.

This method is easy and convenient, and has the advantage that if the radial force should vary over the thickness of a tablet, its value can be found at any point. The other methods described above are only capable of giving the mean pressure over the entire tablet thickness.

Radial pressures have been measured during the application and relaxation of the punch pressure, notably by Leigh, Carless & Burt (1967), who distinguish the behaviour of an ideal elastic material, a constant yield stress material, and a Mohr body, for which the yield stress in shear is a function of the applied normal stress. They were unable to do more, however, than obtain the pressure cycle plots, since they had no data on the mechanical properties of the materials being compressed.

Jaffe & Foss (1959) tabletted a large range of substances and endeavoured to find a factor common to all those which tabletted well, but without success. Varsano & Lachman (1966) measured the compressibilities of beds of potassium chloride and potassium citrate 80 mesh crystals, and also granulations of them using various binders, but did not relate their results to tablet formation or properties. Higuchi, Shimamoto & others (1965) determined both die wall pressure and its rate of decay when the punch pressure was relaxed, but had no mechanical property data for the substances being compressed. They were able to state, but only qualitatively, that softer substances seemed to give higher die wall pressures.

EXPERIMENTAL

Seven crystalline substances were chosen for examination, basing the choice upon pharmaceutical interest and upon relevance to other tabletting work carried out in the Department. The substances examined were aspirin, urea, hexamine, salicylamide, potassium chloride, sodium chloride and sucrose. The sources of supply of the materials were as reported in the previous paper (Ridgway, Shotton and Glasby, 1969).

The surface microhardnesses of the crystals were determined with a microhardness tester (Leitz Ltd., London) in the same manner as reported previously (Ridgway, Shotton & Glasby, 1969). The hardness of the tablets after compression was also determined using the same instrument. The surface of the tablet, which was made using flat-faced punches, was rubbed lightly over graphite powder dusted onto a sheet of paper. This gave the surface a slightly polished appearance, and greatly enhanced the contrast and therefore the ease and accuracy of measurement of the diagonal length of the impressions made by the diamond indentor.

The apparatus for the compression of the tablets and the measurement of die-wall stress was that described by Ridgway (1966). Light passes through a polarizing filter and a quarter-wave plate to give circularly polarized light. This illuminates a Perspex die, the punches for which are compressed by a 100 ton Tangye hydraulic press. Viewing the die through a second quarter-wave plate and an analysing

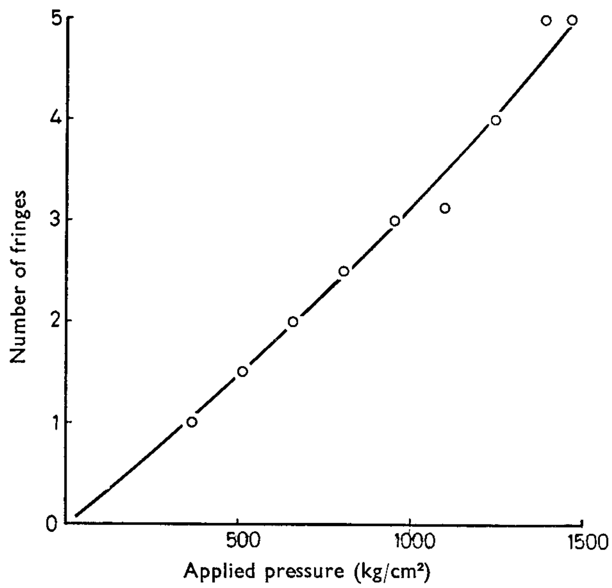

FIG. 1. The calibration curve for the die: fringe order immediately inside the die wall is almost a linear function of applied pressure for an isostatic material under compression.

polaroid filter gives a fringe system within the die which is related to the stress transmitted to the die wall by the compressed tablet. The fringe patterns were photographed at various applied pressures during the compression and relaxation cycles, and the pressed tablets removed for examination. Tablet weights of 1 g were used for all the substances, the die diameter being 12·5 mm. No lubricants were used.

The die was calibrated by compressing a rubber plug as suggested by Windheuser, Misra & others (1963). Rubber under compression gives approximately a hydrostatic pressure so that the die-wall stress is equal to the punch pressure applied. This calibration agreed with that obtained earlier by Ridgway (1966) using silicone putty enclosed in a small chamois leather bag. The fringe order at the die-wall could thus be plotted against applied pressure (Fig. 1).

RESULTS AND DISCUSSION

Surface hardness and transmission ratio

Fig. 2 shows that the force transmitted to the die wall is directly proportional to the compacting pressure exerted on the powder. This is so for all seven substances tested; the constant of proportionality depends on the material being compacted. Because we are interested in the slope of the lines, these graphs are plotted so that the all pass through the origin. The lower the surface hardness of the crystals being compacted, the larger is the force transmitted to the die wall at a given compacting pressure. The relation between the surface hardness of the crystals and the force

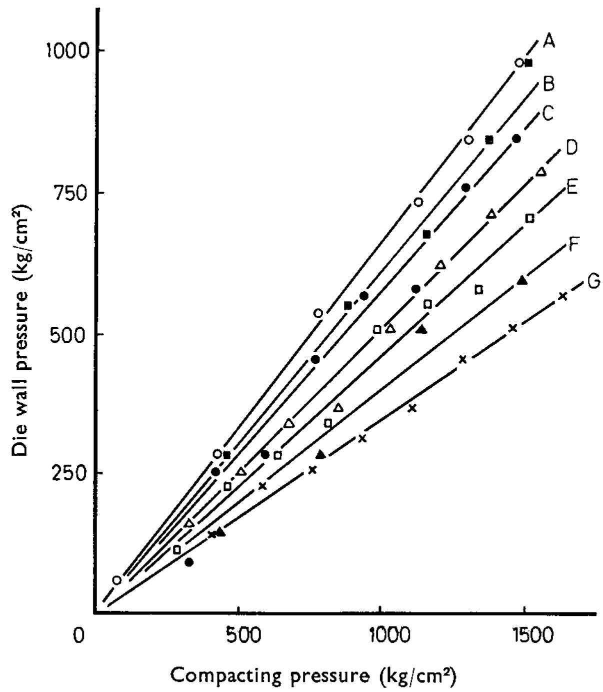

FIG. 2. Radial pressure on the die wall as a function of the pressure applied at the punch face. The lines are plotted so that they pass through the origin. The substances used are as follows: A, Aspirin ○. B, Urea ■. C, Hexamine ●. D, Salicylamide △. E. Potassium chloride □. F, Sodium chloride ▲. G, Sucrose ×.

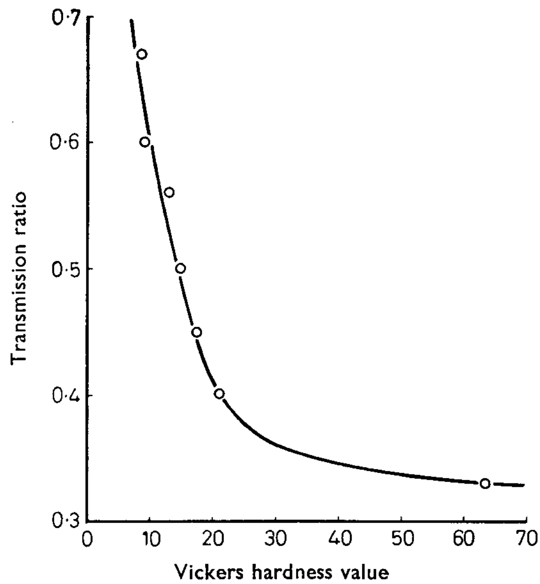

FIG. 3. Transmission ratio, defined as (radial pressure on the die wall) ÷ (axial stress applied by the punches), plotted as a function of the Vickers hardness value of the material being compressed.

they transmit to the die wall at a given compacting pressure, is shown in Fig. 3. The shape of the curve is not unexpected. For softer materials, the transmission ratio (= die wall pressure ÷ applied pressure) is high, and is of course unity at a hardness value of zero. It levels out at about 0·3 for harder materials. This fits in with Nelson's (1955) observation that about 30% of the applied force was transmitted to the die wall by sodium bicarbonate granulation and by sulphathiazole granulations. Nelson also found that lubricants added to the granules increased the transmission ratio, and an important point was made by Windheuser & others (1963) who found that when there was good transmission of forces to the die wall, good tablets were produced.

The results here are in agreement with those of Higuchi & others (1965) who found that the ratio of die wall pressure to upper punch pressure seemed to reflect the hardness of the crystals being compacted, the crystal hardness and the transmission ratio being inversely related to one another. But since these authors did not know the value of the hardness of the crystals used, no quantitative information was derived.

Hardness of tablet in relation to compacting pressure

The surface hardnesses of tablets of the substances tested varied with the compacting pressure used to produce them (Fig. 4). With potassium chloride, hexamine and urea the surface hardness of the tablets showed a slight decrease with increase in compacting pressure. This may be due to incipient failure of the material being compacted, which may occur as plastic deformation or fracture (Train, 1956).

Aspirin and sodium chloride tablets, however, show a tendency to increase in surface hardness as the compacting pressure increases. The increase stops at a certain stage during the compression of sodium chloride crystals and after this point the surface hardness of the tablets remains almost static. An explanation for the behaviour of these materials may be that on compaction, work hardening occurs.

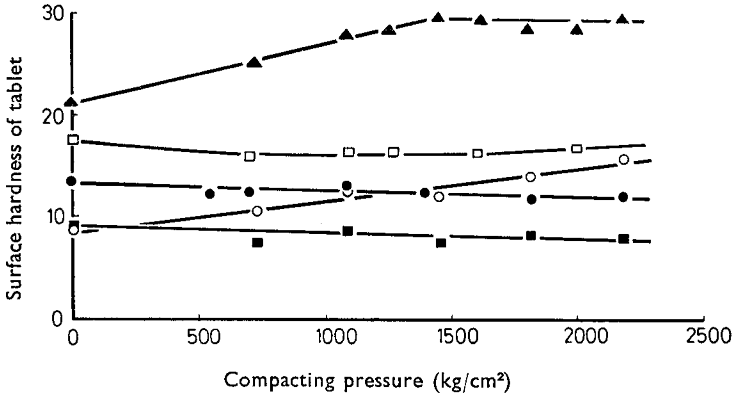

FIG. 4. The surface hardness of the tablets produced at various compacting pressures. Key: ▲ Sodium chloride. ○ Aspirin. □ Potassium chloride. ■ Urea. ● Hexamine.

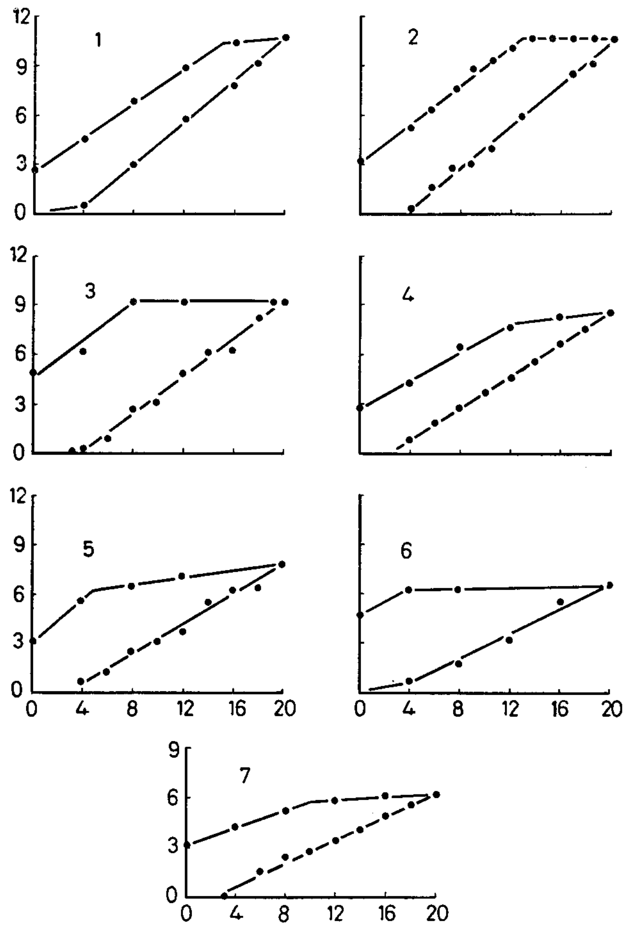

FIG. 5. Variation of radial pressure exerted on the die wall as a function of the punch pressure during the application and relaxation of the compressive force. The units on both axes are thousands of lb/inch^2; radial pressure is ordinate and axial pressure the abscissa. The substances used are as follows: 1. Aspirin. 2. Urea. 3. Hexamine. 4. Salicylamide. 5. Potassium chloride. 6. Sodium chloride. 7. Sucrose.

Compression cycles

In Fig. 5 the "compression cycles" of the materials are plotted, following Long (1960). The scatter of the points is sufficiently large to prevent any conclusions being drawn with certainty, but the cycles have the expected shapes. Those for sodium chloride, aspirin and sucrose agree with the cycles obtained by Leigh & others (1967). In the present work, the cycles tend to be wider and the residual die-wall pressures higher. This may be due to the radial strain being greater in a Perspex die than it is in a steel die. Lateral movement of the material being compacted would allow plastic flow and a greater "permanent set" to occur. The larger lateral strain can be detected on the photographs of the fringe pattern, and work is in hand to produce a glass die which will be photoelastic but, within its limits, as rigid as a steel die.

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