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The application of photoelastic techniques to a rotary tabletting machine

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Powders have been compacted on a rotary tabletting machine, using a Perspex die mounted above the die table, with an extended lower punch and a shortened upper punch. By high speed cine photography in polarized light, the fringes due to the radial stress in the die wall were recorded during compaction. At the same time, the upper and lower punch forces were monitored by instrumented pressure rollers coupled to a recording oscilloscope. The total information thus obtained was sufficient to enable compression cycles to be determined for the first time on a rotary machine.

Much fundamental work on the compaction of powders has been carried out since the development of the instrumented single punch tablet machine, and most of the available data relate to such machines, or to compression in a hydraulic press. Shlanta & Milosovitch (1964) pointed out that non-equilibrium data alone have physical meaning and relevance to tablet production, where the compaction process is much more rapid. Because of this commonly recognized fact, various workers have instrumented rotary tabletting machines to obtain data at normal operating speeds.

A Manesty type D3 machine was instrumented by Shotton, Deer & Ganderton (1963) by bonding strain gauges to the punches. The strain gauge signals were transmitted by a radiotelemetric system to the chart recorder. A comparison between the applied pressure and the tablet crushing strength was made for rotary and single punch machines. The upper and lower punch pressures were much more nearly equal in the rotary machine, the lower punch force being the smaller because the lower roller was sprung, the upper being rigidly supported. Sodium chloride tablets when produced by the rotary machine were stronger than when made by the single punch machine at the same mean pressure, but this difference was not found with aspirin tablets.

Knoechel, Sperry & others (1967) fitted strain gauges to the compression screws interposed between the arm holding the moveable axis of the pressure wheel and the spring used to adjust the compression force applied. Deflection measured by the gauges was proportional to the force applied to the punch. Modification of one of the supporting bolts for the ejection cam allowed the ejection force to be measured. The compression force was approximately linearly related to the tablet weight, when all other machine settings were held constant. The apparent density of the tablets produced increased to an asymptotic value as the compaction force was increased, at constant tablet weight. In correlating the physical properties of the tablets with the forces applied to them, Knoechel & others (1967) distinguished two types of property:

(i) thickness, apparent density, ejection force and compression forces were thought to depend on the machine rather than upon the material being compressed.

(ii) hardness, friability, disintegration and dissolution rates varied with compaction pressure, but this variation depended on the material being pressed. These properties were thought to be affected by the formulation at least as much as by the compressional forces used in the manufacture of the tablets.

Similar studies have been carried out by Wray (1967, 1969) who improved the measurement of the ejection force, finding it to be linear with percentage of added lubricant over a narrow range. He confirmed the equality of upper and lower punch forces to within about 3%.

In the work presented here the photo-elastic technique of Ridgway (1966) has been developed for use on a Manesty type D3 rotary tablet machine. The axial loads applied to the compact were monitored using instrumented compression rolls as described by Deer, Ridgway & others (1969) and the radial pressures transmitted to the die-wall were determined photoelastically as described by Ridgway (1966). Compression cycles were thus obtained for seven substances of pharmaceutical interest. These were the same seven used for compression cycle measurement using a Perspex die in a static hydraulic press (Ridgway, Glasby & Rosser, 1970). These static measurements showed the radial force to be dependent upon the surface hardness of the crystals in the die, confirming quantitatively the qualitative suggestion of Higuchi, Shimamoto & others (1968) that such a relation appeared to hold. They found that the lowest radial pressure was given by lactose and the highest by stearic acid, which were the hardest and softest materials which they compressed.

The work done by a punch during compression is $\int F.dx$ where F is the force and x is the distance moved by the punch face. Higuchi, Nelson & Busse (1955) measured this force in a single punch tablet machine as a function of punch displacement. The area under the curve of displacement against applied load for a sulphathiazole granulation then gave the work done. We have been able to make similar calculations, and have used the energy input to calculate the expected temperature rise for comparison with the results of Travers & Merriman (1970). Their method, implanting a thermocouple in a tablet during compression, cannot be extended easily for measurements on a rotary machine.

MATERIALS AND METHODS

A Manesty D3 rotary tabletting machine, capable of producing 500 tablets per minute in normal operation, was modified to enable the required measurements to be made. The general arrangement of the measuring equipment fitted to the machine is shown in Fig. 1 (for fuller details of technique and results, cf. Rosser, 1970). Fifteen of the stations were blanked off, and the Perspex die fitted at the remaining station on the die table with its associated shortened upper punch and lengthened lower punch. Brackets were bolted to the machine frame to carry the equipment for photoelastic measurement. Light from an Atlas 100 W projector bulb was passed through a 10 cm diameter condenser lens, focal length 25 cm, to give a parallel beam. This beam then passed through a polaroid filter, a first quarter-wave plate, the Perspex die, second quarter-wave plate, analysing polaroid and into the camera, a Hycam (Red Lake Laboratories, Inc., California) capable of a framing rate of up to 10 000 pictures s^{-1} on 16 mm film (Ilford Mk V, using a green filter for maximum fringe definition).

The die had a lower section which fitted into the die table and was held in position by two 4 B.A. screws. The upper section, 57 mm diameter and 32 mm deep, had a

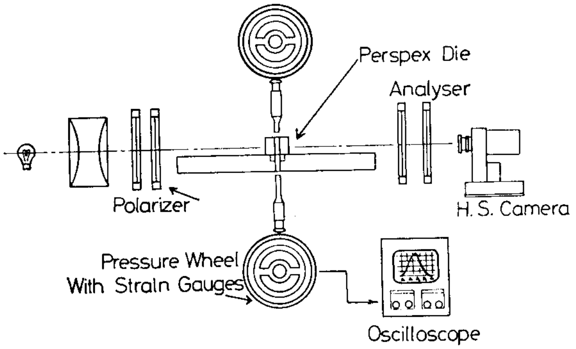

FIG. 1. General arrangement of the apparatus. Light passes through the condenser lens and is polarized by the polaroid filter A. It passes through the die and the second polaroid B to the camera. Upper and lower punch pressures are measured by the instrumented pressure rollers, the values being recorded on the oscilloscope or the chart recorder. The camera and oscilloscope are triggered by the contact wire, with timing marks being derived from the stroboscope and an initiating flash from the flash bulb.

12 mm bore. Two flat surfaces were cut at the front and rear for viewing purposes, and a graticule was ruled on the front surface.

The normal pressure rolls were replaced by a pair of the type mentioned earlier (Ridgway, Deer & others, 1969). These had two central spokes which deflect slightly under load. The original method of using two moire fringe plates to measure the deflection, and hence the applied force, was changed; strain gauges were fitted to the spokes as they gave greater precision. Four C6-121 foil gauges (Automation Industries Ltd., Camberley) were fitted to each pressure wheel. The wheels were mounted with their spokes horizontal, and a strain gauge was cemented to the upper and lower side of each spoke. By suitably connecting the four gauges into a Wheatstone bridge network, it was possible to make the assembly respond only to forces in the vertical direction, horizontal forces and twisting couples exerted on the wheel giving changes in the gauges which cancelled out.

The bridge output was fed to a differential D.C. amplifier (ZLD 2U. RC silicon integrated circuit, Ferranti Ltd.) and could be recorded by a Tektronix type 564 double-beam storage oscilloscope. The wheels and force-measuring equipment were calibrated by pressing in a hydraulic press along with a standardized load pillar of known characteristics.

For a tabletting run, in addition to the camera and the oscilloscope, a stroboscope (1209B, Dawe Instruments Ltd.) was operated so that it flashed light at 6000 flashes/min through a mirror to one part of the camera field of view, and also sent an electrical pulse at the same time to the second channel of the oscilloscope. This synchronizes the time for film and oscilloscope, and also gives a time interval calibration. As the die comes round to the compression station, it makes an electrical contact which fires a flash bulb as well as triggering the single sweep of the oscilloscope. The starting instant of the oscilloscope sweep can thus be correlated with the light flash appearing on the film. The filming speed was usually 1000 frames s^{-1}.

The net result of the technique is that a photographic record of the fringe pattern in the die wall during a compression is obtained, the film carrying markers which enable each frame to be calibrated for time and correlated exactly with the oscilloscope deflection which measures the tabletting force applied. Normally about 20 separate frames during a compression were enlarged and printed for scrutiny. The calibration factor between radial load and the number of fringes obtained was determined by compression of a rubber plug which gives a hydrostatic pressure distribution under applied punch pressure. Punch displacement during a compression could also be obtained from the film or from a large scale drawing of the pressure roll and punch head profiles.

Weighed amounts of powder were loaded by hand into the die, since the feed frame had to be removed as it would have fouled the Perspex die. Ejection force was not measured because (a) the tablet was gripped more firmly for a given residual stress than it would have been in a steel die, because of the yielding of the wall and (b) the die was held in position by two small screws fitting into tapped holes in the die base, and the strength of these fixings was only just adequate to withstand the greater ejection force.

RESULTS AND DISCUSSION

Since the major remaining difference in compaction conditions between the present work and normal industrial practice is that the die is made of Perspex instead of steel, two sets of aspirin tablets were made at a range of compaction pressures in two dies of the same diameter, one steel and the other Perspex. Their diametral crushing strengths are shown in Fig. 2. Tablets made in the Perspex die are some 30% stronger on average than those of the tablets made in the steel die. This is because the wall of the Perspex die yields more than that of the steel die; this allows more shear to occur, which increases the tablet strength. The important point for the purpose of the present work is that it does not appear that compaction in Perspex

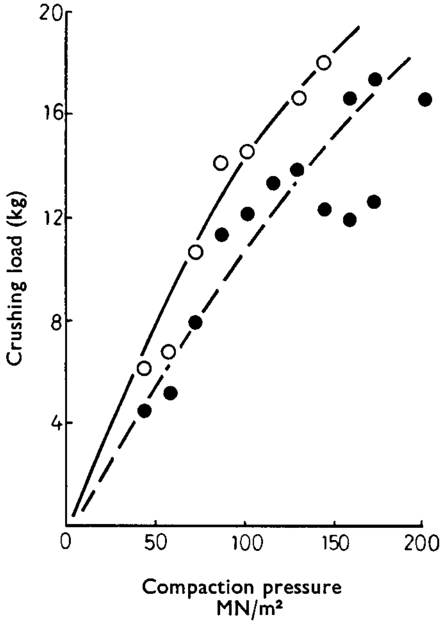

FIG. 2. Crushing load for aspirin tablets as a function of applied compaction pressure ○ tablets made in Perspex die. ● tablets made in steel die.

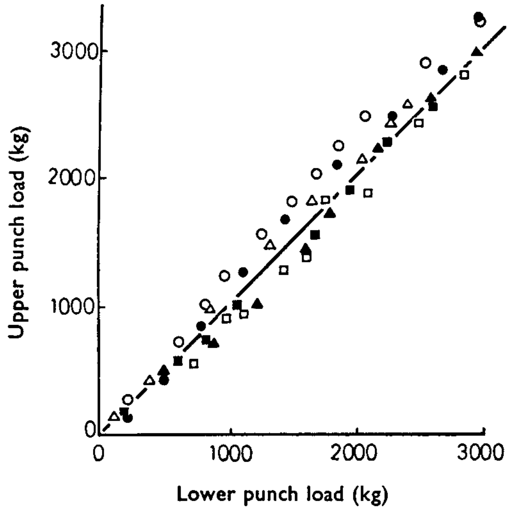

FIG. 3. Upper punch force as a function of lower punch force for a number of substances: ○ aspirin, ● hexamine, △ sucrose, ▲ urea, □ salicylamide, ■ sodium chloride.

is fundamentally different from compaction in steel, so that photoelastically-obtained results should be valid generally.

In Fig. 3 the experimentally-determined relation between upper and lower punch load is plotted for seven substances. From elementary statics, upper and lower punch forces should be equal. The only force which has a vertical component, other than those applied by the punches, is the shear between the tablet and the die wall, and even this should be fairly small. Indeed, for loading equally from top and bottom, it should be zero by symmetry. Inequality of upper and lower punch forces is thus

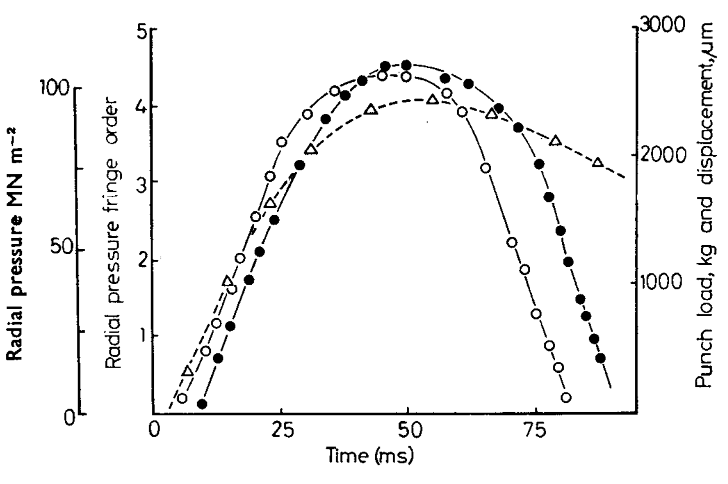

FIG. 4. Applied pressure, radial pressure and punch displacement as a function of time as derived from the photographic and electrical records. ○ punch load, ● radial pressure, △ punch displacement.

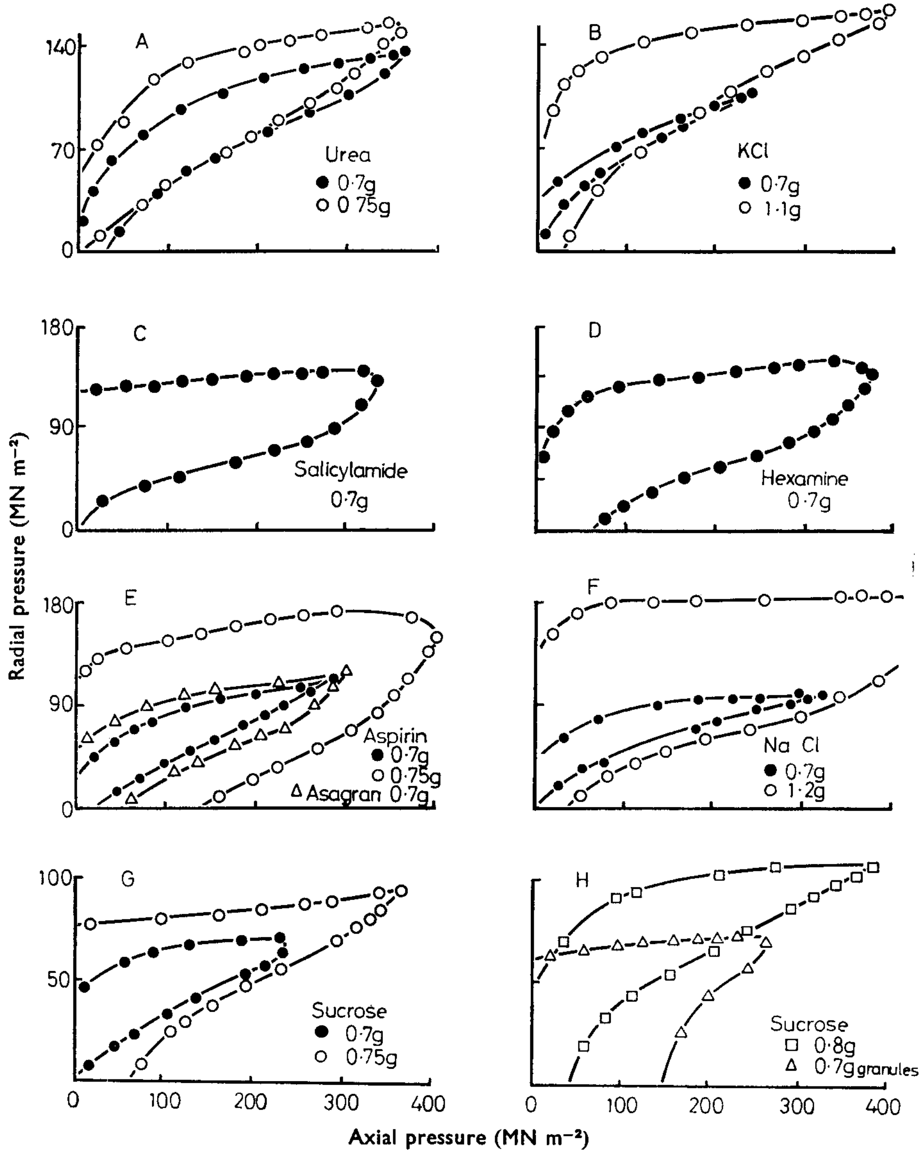

FIG. 5. Pressure cycles, derived from plots of the type shown in Fig. 4, for seven substances.

caused by dynamic factors, is probably machine-dependent, and is relatively unimportant. The greatest average difference in this work between upper and lower punch forces is 8%, for aspirin, but with no apparent correlation with substance properties. Shotton & others (1963) found a 10% difference, but in the opposite sense to that found in the present work (on the same machine). It seems certain that such force differences are due to different accelerations of the punches, either due to one being heavier and therefore more sluggish, or to slight relaxation of the sprung lower roller.

A typical result of a compaction run is shown in Fig. 4, where the applied pressure, the radial pressure on the die wall, and the punch displacement are shown as a function of elapsed time from the zero marked by the flash bulb and the electrical contact. All three quantities increase to a maximum and decrease again, the radial pressure lagging behind the other two. The curve of punch load is a flat-topped parabola, the flatness reflecting the "dwell time" due to the shape of the punch head. The applied pressure is above 80% of its peak value for more than a third of the compaction event.

If the applied punch load is plotted against the radial force exerted at the same instant, pressure cycles may be obtained, and these are shown, for each of the substances examined, in Fig. 5. This method of plotting is due to Long (1960). The cycles in Fig. 5 may be compared to those resulting from slow compression in a hydraulic press (Ridgway & others, 1969). The chief difference is that in the static case, the force transmitted to the die wall is proportional to the hardness of the material being compacted, whereas in the rotary machine, no such correlation is immediately apparent, and it seems that under fast dynamic loading the radial force is more machine-dependent than substance-dependent.

However, some regularities can be seen which do reflect substance properties. Sodium and potassium chlorides both have elongated narrow cycles, pointed at the upper right extremity, indicating good recovery from the applied stress with a rapid deformation under load, as might be expected from an ionic crystal. As soon as the punch force decreases, recovery begins because the substance is capable of following the stress change applied to it.

The softer substances, aspirin, salicylamide and hexamine, show rounded extremities to the cycles. The two latter substances tend to have a horizontal upper contour as the punch force is removed, leaving a higher residual radial stress within the die at zero axial pressure.

The energy put into the tablet by the compression can be estimated, since the force exerted by the punch is known as a function of its instantaneous position. A top punch force versus displacement curve is shown in Fig. 6. The area under the upper part, up to A, is the work done in compressing the powder into a tablet. The area between the line AB and the punch displacement axis is the work done by the tablet

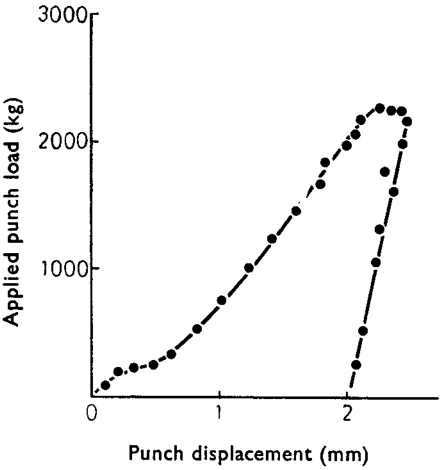

FIG. 6. Load applied by the upper punch as a function of punch displacement. The area inside the curve is the net work done on the tablet in a compaction. Equal work is done by the lower punch.

on the punch as it is withdrawn, and thus represents elastic recovery. The difference between these two quantities is the area of the cycle, and is the net work done on the tablet, $\int F.dx$. It is absorbed as particle fracture energy, plastic deformation, cold welding, wall shear and friction, and appears as heat. Taking upper and lower punch pressures to be equal and opposite, this cycle area is half the total work done on the tablet.

The cycle area in Fig. 6 is 2000 kg-mm, which is 2 kg-m or 18·6 Nm = 18·6 J. The tablet weight was 0·7 g of Asagran, specific heat 1·8 J/g, so that a temperature rise of perhaps 12° would be expected. The net work agrees with the value obtained by Higuchi & others (1955) for a sulphadiazole granulation on a single punch machine, and the temperature rise agrees with the measurements of Travers & Merriman (1970) for Asagran and other materials, also on a single punch machine: values between 10 and 15° on compression were obtained by these authors.

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