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An apparatus for the investigation of die wall friction during compaction

C. J. LEWIS† AND DAVID TRAIN*

A "moving-die" apparatus is described for use in the investigation of die wall friction during the compaction of powders. The powder can be compressed at constant rates varying from 344–860 kg/sec; the die can be moved at a constant rate at values of 0·1–0·25 cm/sec. The apparatus can also be used to apply the force necessary to cause shear in a punch penetration test for the measurement of shear strength of the ejected compacts. Preliminary experiments with 20–30 mesh crystalline sucrose indicate that values of die reaction, F_d, and ejection force, F_e, increase with increased applied pressure. For a given compaction pressure, F_e and F_d decreased as the rate of application of the compacting force increased. For the range of rates available F_d was independent of the rate of movement of the die. The shear strength of ejected compacts prepared at a constant pressure increased with increase in the rate at which the shear force was applied.

THE compression of powders in cylindrical dies has been studied by several workers (Duwez & Zwell, 1949; Spencer, Gilmore & Wiley, 1950; Ballhausen, 1951; Sheinhartz, McCullough & Zambrow, 1954; Toor & Eagleton, 1956), and it is claimed that their results, in general, conform to the exponential relationship:

$$\log_e F_a/F_b = 4\mu\eta L/D \quad .. \quad .. \quad .. \quad (1)$$

where F_a and F_b are the applied and transmitted forces respectively, in a compact of length L and diameter D. The coefficient of friction μ, and the ratio of radial to axial stress, η, have been assumed to be constant.

It has been pointed out already (Train, Carrington & Hersey, 1962) that control of relative interfacial movement between material being compacted and the die wall is important. An apparatus was devised which measured the axial compressive forces and provided a means by which all movements of the test material and top and bottom punch faces could be measured relative to the die.

Reproducible values for die wall reaction could only be obtained in the pressing of solid cylinders of polymers when a minimum relative movement between plug and die wall was induced. Using a similar technique Hersey (1960) was able to show that the Bowden & Tabor (1954) theory of friction could be applied to a compacting system.

In the previous work (Hersey, 1960; Train & others, 1962; Train & Hersey, 1962) the increase in axial force and the movement of the die was incremental, with the possibility of variations in rate of loading and rate of die movement. A modified form of apparatus is now described which permits continuous consolidation of the test material at a fixed speed whilst the die is moved at a constant rate, the compressive force, the die reaction, and the change in length of the compact being recorded during

From the Department of Pharmaceutics, School of Pharmacy, University of London, Brunswick Square, W.C.1.

* Cremer and Warner, 8, Buckingham Palace Gardens, London, S.W.1.

† Present address: Development Department, Boots Pure Drug Co. Ltd., Beeston Works, Nottingham.

the process. Provision of suitable controls enables the rate of compressive loading and the rate of die movement to be varied independently of each other.

APPARATUS

A Tangye four column, 100 ton, hydraulic press was used as the primary ram necessary to compact the powder fill. The secondary hydraulic ram, which moved the die relative to the compact, was mounted on the lower platen of the primary ram and the remaining apparatus was assembled between the top platen of the main press and the secondary ram, in the manner of Train & others (1962), as shown in Fig. 1. The

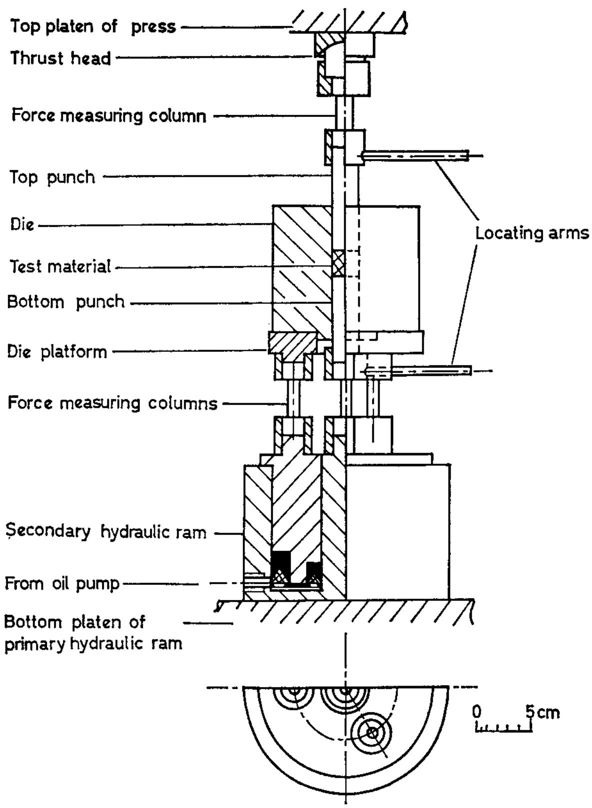

FIG. 1. Die pressing apparatus.

die of hardened A13 steel (Edgar Allen and Co. Ltd.) was 15 cm diameter and 15 cm long, and had a uniform, circular, bore which was chrome plated and ground and polished to a final diameter of 2·41 cm. To minimise flow of test material around the punch faces the clearance at the punch tip was 0·0005 cm. This surface was tapered back for 0·5 cm to give a clearance of 0·005 cm which was maintained for the remaining length of the punches.

Provision was made for raising the lower platen of the press by means of a hand pump, or continuously by means of a pump (Type IH 036, Chamberlain Industries Ltd.) driven by a 1·5 h.p. motor running at 1,450 rpm. The piston of the secondary ram was raised by a similar pump connected to a 0·75 h.p. motor.

The hydraulic circuit for each ram (Fig. 2) was a separate entity enabling individual control. In each circuit the application and release of pressure was controlled by Dowty single bank valves (Type CV2/O3S/BZ) and the rate of movement of each ram was governed by Vickers

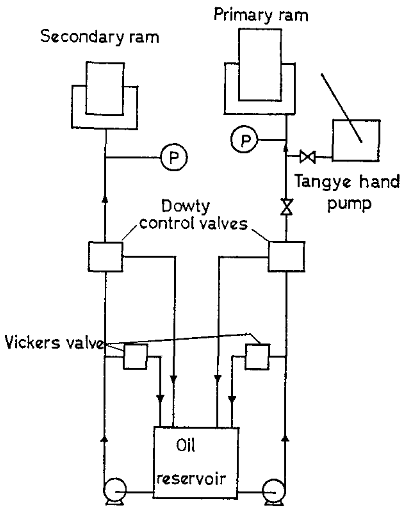

FIG. 2. Hydraulic circuit.

flow control valves (Type FG-02-250-M, Stein Atkinson Vickers Hydraulics Ltd.). By this means it was possible to vary at will the time of application of compressive load on the powder fill, the rate at which the compressive load was applied, and the time and rate of movement of the die relative to the compact.

Force measurements were made by utilising elastically strained pillars on which were bonded resistance strain gauges, enabling the reaction to be measured electrically. The measurements were recorded continuously during a pressing operation using an Ultraviolet Recorder (New Electronic Products, type 1050) with its associated carrier amplifier (New Electronic Products, type 1070).

Relative movements between the punches and die were measured (±0·01 mm) by using suitably placed dial gauges. The initial depth of powder in the die was determined by measuring the distance between location marks on the top and bottom punches. Since the compressive load was applied continuously at constant rates, the pointers on the dial gauges moved rapidly. Readings of punch positions were taken by using a "Robot Junior" camera to photograph the gauges at 1/500 sec at *f*11 on 35 mm FP3 Series II film. As each photograph was taken the event was indicated on the instrument recording the forces, so that the height of the compact could be related to the pressure applied at that instant.

The compaction force can be applied at rates varying from 344–860 kg/sec. The rate at which the compaction force was applied was computed from the paper recording the force measurements. Since the paper issued from the recorder at a known and constant rate, the time taken for the compacting force to increase from zero to its maximum value was obtained by measuring the paper. The rate of loading is then the maximum force divided by the time taken to attain the maximum value.

Free movement of the die could be varied between 0·1–0·25 cm/sec. However, owing to the mechanism of action of the Vickers flow control valve, large die wall forces stopped the movement of the die at low rates of movement. Consequently the practical range of movement was limited to 0·15–0·25 cm/sec.

Experimental

GENERAL METHOD OF OPERATION

The bore of the die was cleaned, polished, and degreased with a mixture of equal parts acetone and carbon tetrachloride before use. The punch tips were lubricated with a film of colloidal graphite, deposited from an acetone "dag" suspension (Acheson Colloids Ltd.).

For each experiment the apparatus and test material were assembled as in Fig. 1. During the compression cycle the applied force, F_a, and the die reaction, F_d, were recorded continuously and photographs of the dial gauges were taken at frequent intervals. When the desired pressure level had been attained the compaction was stopped, the pressure released immediately, the lower punch removed from the die, and the compact ejected by moving the die upwards relative to the stationary upper punch. The ejection force, F_e, was recorded. 5 g samples of unlubricated crystalline sucrose 20–30 mesh, stored for not less than 7 days over silica gel in a desiccator, were used throughout this work.

APPLICATION TO THE MEASUREMENT OF SHEAR STRENGTH

In certain instances the shear strength of the ejected compacts was measured immediately after compaction, using a punch penetration method previously applied to homogenous solid specimens (Train & Hersey, 1960). The shear force was applied at a constant rate by using the motor-driven secondary ram, the force necessary to cause shear being recorded during the test; a typical recording of pressure changes occurring during the measurement of shear strength is shown in Fig. 3.

EFFECT OF APPLIED PRESSURE

5 g samples of sucrose were compressed at pressures up to 9,500 kg/cm^2 with the die moving relative to the compact at a constant rate throughout

the operation. During the compaction, measurements were taken continuously of the force acting on the upper punch, F_a, the die reaction, F_d, and the change in position of the punches. The ejected compacts were weighed, and the length and diameter were measured with a micrometer gauge before determination of the shear strength.

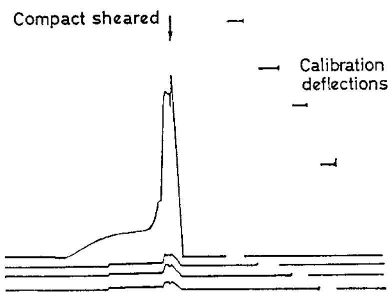

FIG. 3. Pressure changes during the measurement of shear strength.

EFFECT OF RATE OF APPLICATION OF COMPACTING PRESSURE

5 g sucrose was compressed to a predetermined pressure level, applying the compacting force at the slowest rate possible. The compact was ejected from the die and the shear strength determined. Further samples were compressed to the same pressure level but the force was applied at different rates. For each rate of loading F_a, F_d, F_e, and change in density of compact, were measured.

EFFECT OF DIE MOVEMENT ON VALUES OF F_d

5 g samples of sucrose were compressed to a pressure level of approximately 4,300 kg/cm^2. For each sample the die was moved at a different rate and values of F_d were measured during the compaction cycle.

INFLUENCE OF RATE OF APPLICATION OF SHEAR FORCE ON THE OBSERVED SHEAR STRENGTH

Compacts were prepared at a fixed pressure of $1{,}547 \pm 10$ kg/cm^2 from 5 g samples of sucrose, the rate of application of compacting force and all other conditions being maintained the same for each compact. The shear strength of the compacts was then measured applying the shear force at different rates.

Results and discussion

The results are presented in Tables 1–3, and Figs 4–6.

It was observed that the ejected compact bore lamination marks in the horizontal plane when compressed at pressures of 2,852 kg/cm^2 and higher. Sometimes the compact was whole with horizontal cracks visible, at other times the various layers were quite loose and capable of easy separation. There appeared to be no definite correlation with the applied pressure but the layers were particularly loose at pressures of 2,852–6,350 kg/cm².

The length of the ejected compact was not reduced below that at 2,605 kg/cm² compaction pressure until P_a = 7,450 kg/cm² (Table 1), at which pressure the number of laminations in the compact were fewer and the upper laminations were particularly highly compressed and becoming translucent in appearance, indicating a consolidated mass with fewer internal reflecting surfaces.

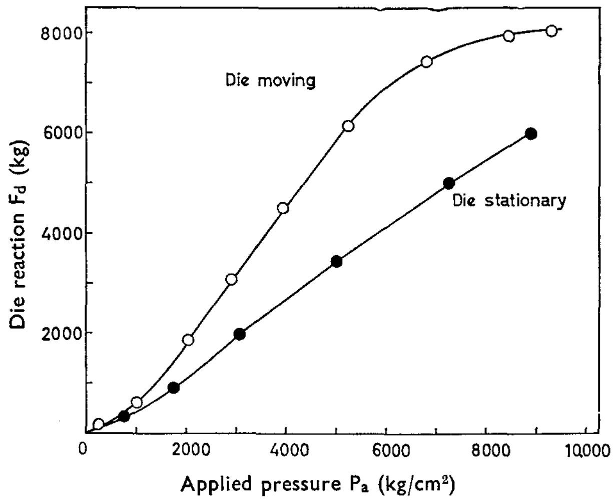

FIG. 4. Effect of applied pressure on die reaction.

TABLE 1. THE EFFECT OF APPLIED LOAD ON THE COMPACTION OF SUCROSE 20–30 MESH

Length of compact cm	Diam. of compact cm	Max. P_a kg/cm²	Max. F_d kg	Ejection force, F_e kg	Relative* density ρ_R	Shear strength kg/cm²
0·821	2·42	884	763	—	0·856	91·5
0·780	2·42	1,198	773	134	0·900	135
0·765	2·42	1,752	901	262	0·919	211
0·745	2·43	2,151	1,873	360	0·943	235
0·742	2·43	2,605	1,851	495	0·948	253
0·754	2·43	2,852	2,670	760	0·933	224
0·752	2·42	3,338	2,914	701	0·934	252
0·753	2·42	4,193	4,160	879	0·934	259
0·746	2·42	5,097	5,199	1,751	0·943	310
0·745	2·42	6,350	6,407	1,128	0·943	296
0·741	2·42	7,450	7,484	2,465	0·949	345
0·749	2·42	8,050	7,983	2,893	0·938	353
0·740	2·42	9,895	8,371	—	0·950	281

* Relative density is calculated from dimensions of extruded compact.

Values of F_d only begin to approach a maximum (Fig. 4) at the highest pressure used. Since the force lost to the die wall is unlikely to reach a maximum until the area of the compact—die wall interface is a maximum, i.e., at zero porosity ($\rho_R = 1$), the explanation lies in Fig. 5 where it is seen that this degree of consolidation is only attained at pressures greater than 7,250 kg/cm².

The relative density, ρ_R, is defined as the ratio of compact density to density of the solid material. Curve A of Fig. 5 shows the density of the compact under load and Curve B indicates the densities of the ejected compacts. The maximum relative density attained in the ejected compacts was 0·95 and was achieved first at an applied pressure of 2,600 kg/cm²; compaction to greater pressure levels did not decrease the porosity further. It is apparent also that the compacts compressed to pressures

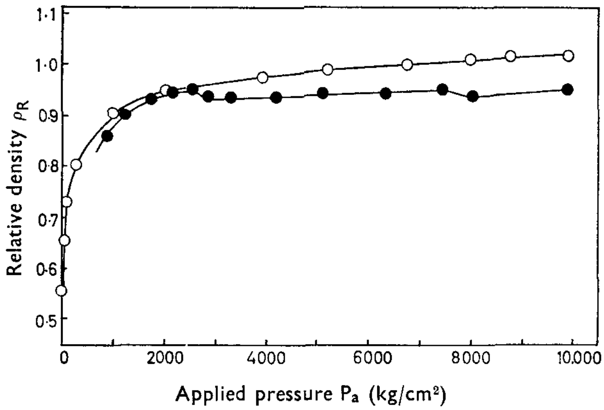

FIG. 5. Effect of applied pressure on density of compact. ○ Compact under load (Curve A). ● Ejected compacts (Curve B).

greater than 2,600 kg/cm² undergo appreciable axial expansion when ejected from the die.

The pressure at which lamination of the compact was apparent is that at which the ejected compact approached its maximum density. Pressing beyond this point is equivalent to the pressing of a solid body, serving only to increase the elastic strains residing in the compact when the applied pressure is removed. It is not known if these laminations are present after removal of the applied load and before ejection of the tablet.

The ejection force, F_e, increased with increased compaction pressure (Table 1). However, at a given pressure F_e and F_d decreased as the rate of application of the compacting pressure was increased (Table 2). At

TABLE 2. THE EFFECT OF RATE OF APPLICATION OF COMPACTING PRESSURE

Rate of applying load, kg/sec	Max P_a kg/cm²	Max F_d kg	F_e kg	Shear strength kg/cm²	a ρ_R	b ρ_R
344	1,779	1,319	586	201	0·95	0·91
534	1,772	972	436	224	0·97	0·91
677	1,755	708	249	230	0·98	0·91
826	1,760	651	233	234	0·99	0·91
860	1,797	698	258	221	1·02	0·91

a. Value when under load of Max P_a.
b. Value calculated from dimensions of ejected compact.

the same time values of ρ_R increased with increased rate of application of pressure, although the density of the ejected compacts was unaffected.

The compaction force was followed by observation of a Bourdon gauge which registered the oil pressure in the primary hydraulic ram. Owing to the rate at which the applied pressure increased it was difficult to stop the compaction process at exactly the same pressure level in each compression, and this accounts for the observed variations in the maximum value of P_a recorded in Table 2. It is considered that the small variations in the magnitude of P_a will have a negligible effect on the experimental results, compared with the effect of changing the rate of application of pressure.

Values of F_d were greater when the die was moved at constant rate than when maintained stationary (Fig. 4). For the rates of movement available, the die reaction was independent of the rate of die movement. Relative movement between the powder and the stationary die as the material consolidates under an applied pressure is not uniform along the length of the compact; the amount of movement near to the lower punch is less in a single-ended pressing than near the face of the upper punch. Movement of the die increases the number of powder-die contacts which are sheared, and this is reflected in an increased value of F_d.

OBSERVATIONS ON THE SHEAR STRENGTH OF SUCROSE COMPACTS

The shear strength of the ejected compacts increased with increased applied compaction pressure (Fig. 6), approaching a plateau as the compact assumed minimum attainable porosity. This plateau represents the maximum shear strength of a coherent compact; thereafter the values represent the strength of the most densely compacted upper laminations.

For a given compaction pressure, increasing the rate at which the compaction force is applied increased the shear strength of the compacts (Table 2). In addition the observed shear strength of compacts prepared at a constant pressure of 1,547 $\pm$ 10 kg/cm^2 increased as the rate of application of the shearing force increased (Table 3). The latter results are in agreement with other determinations of "strength", e.g., tensile strength, compressive strength, crushing resistance of tablets, in that they are influenced by the rate at which the load necessary to cause failure is applied.

TABLE 3. THE EFFECT OF RATE OF SHEAR ON SHEAR STRENGTH

Mean compaction pressure: 1,547 kg/cm^2

Rate of shear kg/sec	Observed shear strength kg/cm^2
23	182
29	189
166	201
308	223
432	205

CONCLUSIONS

On the basis of these preliminary results it is concluded that a standard operating procedure should be adopted when comparing the behaviour of different materials. For convenience of operation the following conditions were chosen for later work:

1. Rate of application of compacting pressure to be constant at 638 kg/cm² per sec.

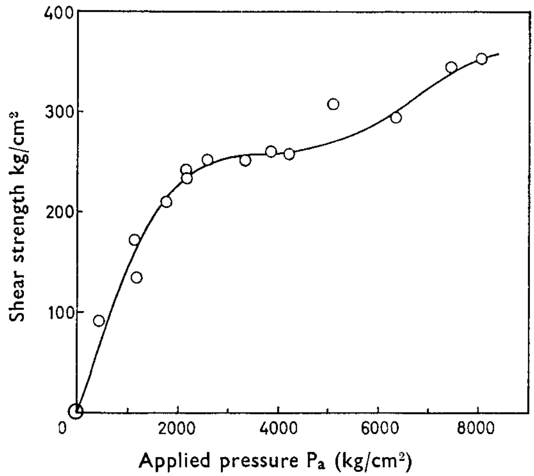

FIG. 6. Effect of applied pressure on shear strength of compact.

2. The rate of die movement to be constant at 0·22 cm/sec.

These operating conditions influence each other. If the die moves too quickly it will reach the top of the upper punch before maximum pressure is attained. The conditions decided on allow die movement throughout the entire compaction stage even when pressing to high pressure levels.

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