THE STRENGTH OF COMPRESSED TABLETS

PART I. THE MEASUREMENT OF TABLET STRENGTH AND ITS RELATION TO COMPRESSION FORCES

BY E. SHOTTON AND D. GANDERTON

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

Received May 19, 1960

A motorised single-punch eccentric tablet machine has been fitted with instruments to measure the forces operating during the compression cycle. Apparatus has also been designed to measure the resistance to crushing of the final compact. Sodium chloride was compressed at varying pressures and the relationship between compaction forces, ejection force, strength and voidage investigated.

FUNDAMENTAL research on the problems of tabletting was begun in 1950 by Higuchi, Arnold, Tucker and Busse[1] and in subsequent investigations many aspects of formulation and production were described. Train[2] has studied the transmission of forces through a powder under compression and other contributions have been made by Munzel and Kagi[3], and by Seth[4].

This investigation is concerned with the factors affecting tablet strength. Sodium chloride was used since it forms a satisfactory tablet without the addition of excipients, and in this way, a simple system could be studied.

The Instrumentation of a Tablet Machine

The instrumentation of a tablet machine has been described by Higuchi, Nelson and Busse[5]. Strain gauges were bonded to the frame of a single-punch eccentric machine to give a measure of the applied force. The disadvantage of this was the non-linear relation between frame distortion and the applied force. We used a Lehman single-punch eccentric tabletting machine, driven at a constant speed through a Kopp Variator. A $\frac{1}{2}$ inch plane-faced, cylindrical punch and die set was chosen and two strain gauges, connected in series (Tinsley, Type 6H, gauge factor 2·16, resistance 50 ohms), were bonded to the upper punch. The compensating resistance consisted of two similar gauges on a piece of punch steel of the same dimensions. The active (upper punch) and compensating resistances formed two arms of a Wheatstone Bridge connected to one channel of a carrier amplifier (New Electronic Products, type 1070). The bridge was activated by a 3,000 c.p.s. source of 4 volts (r.m.s.). The magnitude and phase of the bridge output depended on the resistance of the strain gauges and after amplification and phase sensitive rectification, the signal was applied to a recording galvanometer having a natural frequency of 250 c.p.s. The optical arm was 20 cm. in length and had an ultra-violet light source. The deflection was recorded on a sensitive paper moving at a pre-selected speed (New Electronic Products Ultra-violet recorder, type 1050). For the measurement of the force transmitted to the lower punch, the six upper threads of the lower punch holder were removed. Three strain gauges were bonded to the holder and connected in series (see Fig. 1). A compensating resistance was constructed to complete the second recording channel. The system was calibrated by placing the upper punch in the lower punch holder. The assembly was then mounted on three previously standardised strain columns and stressed by means of a hydraulic press, care being taken to maintain axial loading. A linear relation between galvanometer deflection and applied force was found. The deflection was calibrated by introducing a resistance into the bridge equivalent to a 0·1 per cent out-of-balance. This corresponded to 1,100 kg. for the upper punch and 3,015 kg. for the lower punch, and varied less than 0·3 per cent in three independent trials.

The Estimation of the Resistance to Crushing

The Monsanto Hardness Tester and the Strong-Cobb Hardness Tester are commonly used to estimate the hardness of tablets. Both suffer from

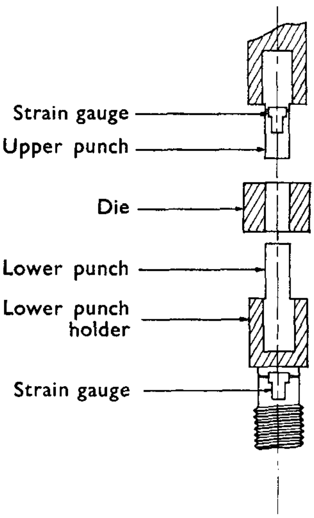


FIG. 1. Instrumented punch assembly.

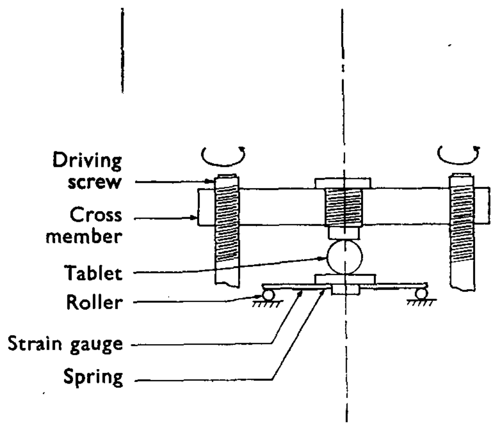


FIG. 2. Apparatus for measuring the resistance of tablets to crushing.

the disadvantage that the rate of loading is not constant. For the present work, it was decided to retain the crushing mechanism of these tests for subsequent comparison but to design apparatus capable of loading the compact under test at a constant rate. This apparatus is diagramatically represented in Figure 2.

The spring was supported at either end by hardened steel rollers. Two strain gauges (Tinsley, Type 6K, gauge factor 2·15, resistance 100 ohms) were bonded to the underside of the spring, one on each side of the central platform, and connected in series. A compensating resistance completed a circuit similar to that already described. By means of a low geared motor, the driving screws were rotated, thus raising or lowering the cross member. The tablet was centrally placed, on edge, on the platform. The cross member was then lowered against the action of the spring until the compact failed. Deflection of the spring caused elongation of the strain gauges and a proportional change in resistance.

The system was calibrated by loading the spring in known increments and recording the galvanometer deflection. The calibration curve was linear, a 0·1 per cent out-of-balance corresponding to 19·10 kg. The trace of deflection versus time was also linear, indicating a constant rate of loading equal to 1·6 kg. sec.$^{-1}$.

Experimental

Sodium chloride was carefully sieved and a 30–40 mesh fraction selected. The density was estimated and the moisture content determined. In the preparation of the tablets a constant weight of material was used, calculated to give a tablet of thickness 0·4 cm. at zero porosity.

TABLE I

SUMMARY OF THE COMPRESSIVE FORCES IN THE PREPARATION OF TABLETS FROM SODIUM CHLORIDE

Upper punch pressure P_a kg./sq. cm.	Lower punch pressure P_b kg./sq. cm.	Mean compaction pressure P_m kg./sq. cm.	$\frac{P_b}{P_a}$	Ejection force F_e kg.
1,854	1,491	1,673	0·804	216
1,504	1,236	1,370	0·822	163
1,224	1,027	1,126	0·840	125
1,016	853	934	0·840	96
793	670	732	0·844	71
606	511	559	0·845	55
417	350	384	0·841	37

Each value given is the mean of twelve results

Tablets were prepared at a constant rate of 68 tablets per minute and at each pressure level feed conditions were allowed to stabilise by rejecting the first ten tablets. The succeeding twelve tablets were then collected in sequence and individually weighed. The thickness and diameter were measured with a micrometer and, finally, the crushing pressure determined.

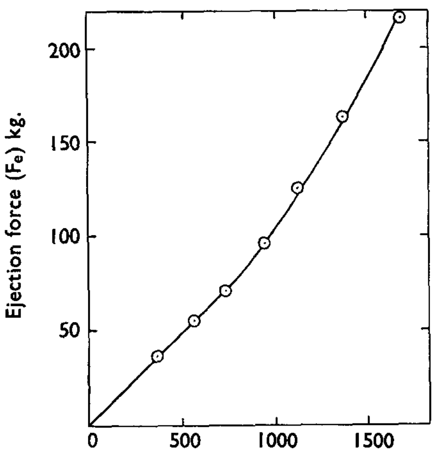
In a typical compression cycle, the deflection of the galvanometer which remained after the upper punch had been withdrawn, represented the force remaining on the bottom punch due to the elastic recovery of the compact against the die wall. Over the range studied, this force was required to eject the tablet from the die.

Results and Discussion

Table I summarises the experiments carried out giving the pressure exerted by the upper punch (P_a), the pressure transmitted to the lower punch (P_b), and the force required to eject the tablet (F_e).

The ratio of the lower punch pressure and the upper punch pressure remains constant over the range 400–1,000 kg./sq. cm. The relation between the mean compaction pressure ($P_m = (P_a + P_b)/_2$) and the ejection force (F_e) is linear over this region. (See Fig. 3.)

Within this range, it is probable that the directional distribution of forces remains the same, with a constant proportion being transmitted to the die wall due to an increase in the area in contact with the die wall proportional to the applied force. Above 1,000 kg./sq. cm., a higher fraction of the applied force was transmitted to the die wall as shown by a fall in the ratio of lower punch pressure and upper punch pressure resulting in an increase in the slope of the ejection force curve. This deviation could be explained either by an increase in the coefficient of friction of the compacting material with pressure, or alternatively, by flow



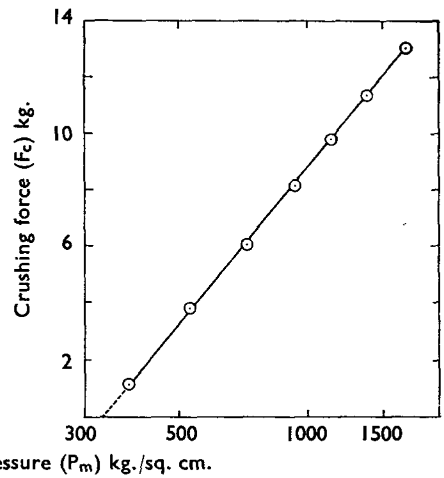


FIG. 3. Relation between the mean compaction pressure (P_m) and the ejection force (F_e).

FIG. 4. Relation between the mean compaction pressure and the crushing strength of the tablet (F_c).

of material past the lower punch. This would give an increase in the area in contact with the die wall. There was, however, no visible evidence of flow as the edges of all tablets were clean and square.

The Strength of the Compacts

The mechanism of compact failure was complex. A wedge of material was formed in contact with the upper plate and the lower platform. The wedges eventually cleaved the tablet across the diameter, giving immediate relief of pressure as shown in Figure 5.

The relation between the mean compaction pressure (P_m) and the resistance to crushing of the compact (F_c) is shown in Figure 4.

The experimental results closely follow the relationship:

$$\log P_m = nF_c + C$$

where n and C are constants. Extrapolation of the line gives a value of C equal to 320 kg./sq. cm. and which represents a minimal pressure for the

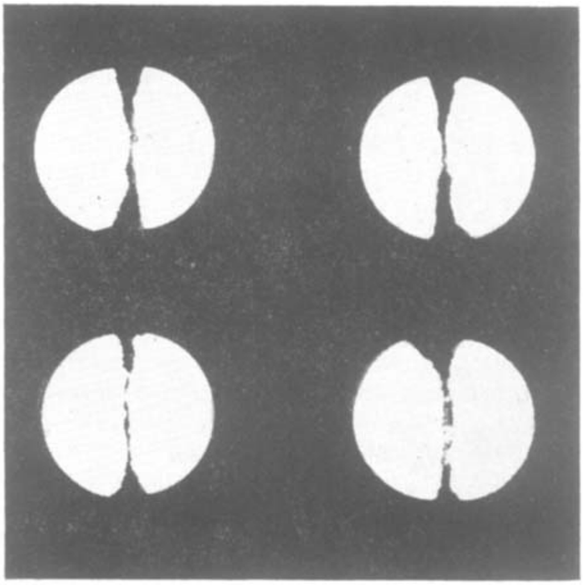


FIG. 5. Examples of the fracture of tablets.
Upper row: Tablets made under high compression.
Lower row: Tablets made under low compression.
A similar wedge formation is shown by both series.

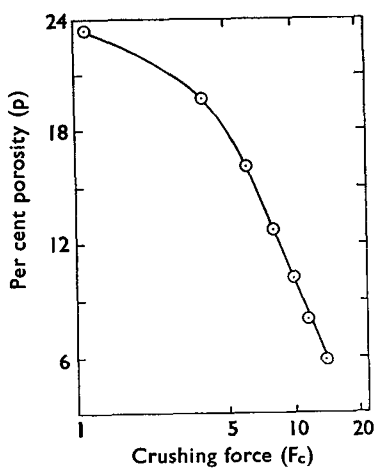


FIG. 6. The effect of porosity on the crushing strength of the tablet (F_c).

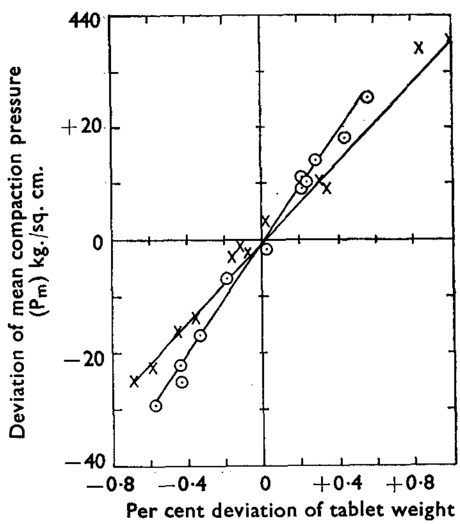


FIG. 7. The effect of variation of tablet weight on the mean compaction force (P_m).
X, P_m = 935 kg./sq. cm.
O, P_m = 1370 kg./sq. cm.

formation of a compact. Below this value, the compact should have no residual strength. Attempts to form such a compact failed.

Bal'shin[6] formulated the following relationship between the strength of a compact and the voidage for powdered metals and ceramics

$$S = S_0 V_r^{-m}$$

where S is the strength of the compact (F_c), V_r is the relative volume, m is a constant and S_0 is the strength when $V_r = 1$. The relative volume, V_r, is the ratio of the observed volume of the compact to the theoretical volume of material.

A further relationship was proposed by Ryshkewitch and Duckworth[7,8]

$$S = S_0 e^{-bp}$$

where p is porosity and b is a constant.

Over the porosity range we studied, these equations are mathematically similar. The results for sodium chloride plotted according to the Ryshkewitch equation are given in Figure 6.

The results show excellent agreement over the range 5–16 per cent porosity. Extrapolation gives a value for the crushing strength of 22 kg. at zero porosity. Above 16 per cent porosity, the results show increasing deviation. The equation was derived from experiments on rigid porous materials and does not seem to apply to the loose compact produced at low pressures. At higher pressures, and certainly over the range of pressures used in the preparation of tablets, the equation would seem to accurately express the relation between strength and voidage.

The effect of variation in weight of fill. As with most tablet machines, the measure of die charge was made by volume. The inherent variation of weight which results from this method was reflected in the forces applied to the tablet. The variation of weight and of applied pressure is shown in Figure 7. Two series are shown (1,370 and 935 kg./ sq. cm.), and the deviation plotted for the twelve individual tablets of each series. Both series indicate that a variation of 0·5 per cent in weight produces a 2 per cent variation in the mean compaction pressure. By reference to Figure 4, this is equivalent to a 1 per cent variation in the resistance to crushing, discounting the effect of a slightly differing thickness.

Acknowledgements. The authors wish to thank Dr. D. Train for the use of the electronic apparatus which was bought with the aid of a grant from the Department of Scientific and Industrial Research. The D.S.I.R. is also to be thanked for a research studentship awarded to Mr. D. Ganderton. We also wish to thank the Distillers Company Ltd. for the basic apparatus for measuring crushing strength which we modified to our own requirements.

References

1. Higuchi, Arnold, Tucker and Busse, *J. Amer. pharm. Ass. Sci. Ed.*, 1952, **41**, 93.
2. Train, *Trans. Instn. Chem. Engrs. Lond.*, 1957, **35**, 258.
3. Munzel and Kagi, *Pharm. Acta Helvet.*, 1954, **29**, 53.
4. Seth, Sc.D. Thesis, Zurich, 1956.
5. Higuchi, Nelson and Busse, *J. Amer. pharm. Ass. Sci. Ed.*, 1954, **43**, 344.
6. Bal'shin, *Dokl. Akad. Nauk S.S.S.R.*, 1949, **67**, 831.
7. Ryshkewitch, *J. Amer. ceram. Soc.*, 1953, **36**, 65.
8. Duckworth, *ibid.*, 1953, **36**, 68.