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Research Papers

An investigation of die wall friction during the compaction of powders

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Crystalline aspirin, hexamine, sucrose, and sodium chloride, and simple granulations of hexamine and of sucrose, have been compressed and the die reaction determined using a "moving-die" technique. The shear strength of the ejected compacts has been measured under zero load conditions, and when compressed to theoretical density, using a punch penetration test. Calculated values of die reaction based on friction theory have been compared with experimental measurements. Correlation between the two is best for aspirin and hexamine crystals when the compact approaches theoretical density and shear strength values for compacts of zero porosity are used in the calculations; for sodium chloride, correlation is best when shear strength values under zero load conditions are used.

EXPERIMENTAL results on the pressing of powders in cylindrical dies (Duwez & Zwell, 1949; Spencer, Gilmore & Wiley, 1950; Ballhausen, 1951; Sheinhartz, McCullough & Zambrow, 1954; Toor & Eagleton, 1956) conform to the exponential relationship: (see Lewis & Train, 1965)

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

Basic frictional research has shown that the value of μ may be modified by the stress normal to the interface (Pascoe & Tabor, 1956) and by relative interfacial movement (Courtney-Pratt & Eisner, 1957).

In some instances of powder compaction (Ballhausen, 1951; Sheinhartz & others, 1954; Toor & Eagleton, 1956) there was uncontrolled movement because the bottom punch was required to move to actuate the force measuring device; in others (Duwez & Zwell, 1949; Spencer & others, 1950; Train, 1956; Train, 1957) movement at the bottom punch was negligible since the bottom punch was virtually fixed to the die. Spencer and his colleagues (1950) induced relative movement between the die and the material in order to measure the limiting coefficient of die wall friction, and a moving-die technique designed to standardise relative movement has been reported (Train, Carrington & Hersey, 1962).

A punch penetration test has been used to measure the shear strength of solid specimens of talc and graphite (Train & Hersey, 1960a) and other materials (Hersey, 1960) and it was shown (Hersey, 1960), using idealised systems, that the Bowden and Tabor theory of friction (Bowden & Tabor, 1954) could be applied to a compacting system.

Practical values for the shear properties of small crystals are difficult to obtain. The present work describes an investigation of the frictional

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behaviour of some crystalline materials and an attempt to correlate die wall friction with the shear strength of the compact.

MATERIALS AND APPARATUS

The apparatus used has been described elsewhere (Lewis & Train, 1965).

Crystalline samples of sodium chloride, hexamine, aspirin, and sucrose were sieved using British Standard sieves on an Inclyno machine for 15 min. 40–60 mesh sodium chloride and 30–40 mesh fractions of the other materials were used.

Simple granulations of sucrose and of hexamine were made by hand, using crystalline material which had been ball-milled until all passed a 100 mesh sieve, and distilled water as a binding agent. The sucrose granules were dried for 2 hr at 60°; the hexamine granules were dried at room temperature for 1 hr and then at 50–55° and 29 in Hg vacuum for 2 hr. The dried granulations were sieved and 30–40 mesh fractions used.

Experimental

5 g samples of the test materials were compressed in a die with 2·41 cm bore at pressures up to 5,000 kg/cm^2. The rate of application of pressure was constant at 638 kg/sec and the die was moved relative to the compact at 0·22 cm/sec. The compacts were ejected from the die, their weight and dimensions measured, and the shear strength determined immediately using a punch penetration test (Train & Hersey, 1960a); the shear force was applied at a constant rate of 166 kg/sec.

The applied force, the die reaction, F_d, and the ejection force, F_e, were recorded using a U.V. recorder (Lewis & Train, 1965); the change in length of compact with pressure was also measured.

To assess the effect of wall friction on the process of consolidation only, 2% magnesium stearate powder -100 mesh, was added to further quantities of test material. 5 g quantities of the lubricated materials were compressed in the die in an identical manner and the change in density of the compact with load was determined.

Three compacts of each unlubricated crystalline material were made of such a weight that their length at zero porosity was 0·5 cm; the compaction pressure used was 1,000 kg/cm^2. The compacts of hexamine, aspirin, and sodium chloride, were subjected to a compressive load in the shear strength apparatus so that the porosity was nil, and the shear strength was measured in the normal way. The pressure required to reduce the sucrose compacts to zero porosity (4,250 kg/cm^2) was beyond the capabilities of the apparatus, so the shear strength of sucrose compacts under load was measured at the maximum pressure available (3,000 kg/cm^2), at which pressure the porosity of the compacts was 3%.

Results and discussion

SHEAR STRENGTH OF COMPACTS

With the exception of sodium chloride, the shear strength of the compacts attained peak values within the range of compaction pressures used (Fig. 1). For the sake of clarity results for sucrose granulation are given in Table 1.

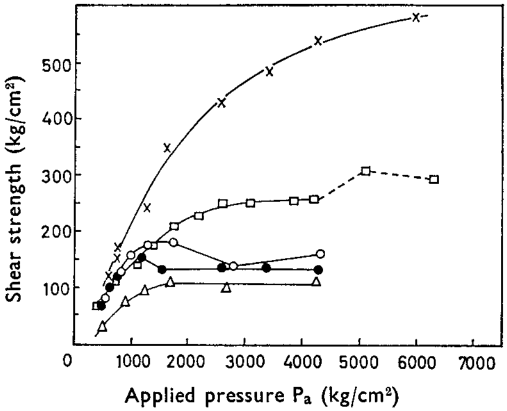

FIG. 1. Effect of applied pressure on shear strength of compact. × Sodium chloride. □ Sucrose. ○ Hexamine. ● Hexamine granulation. △ Aspirin.

TABLE 1. THE EFFECT OF APPLIED PRESSURE ON THE SHEAR STRENGTH OF COMPACTS OF SUCROSE GRANULATION

Applied pressure Pa kg/cm²	Shear strength kg/cm²
547	33
872	68
1,715	211
2,920	124

The decrease in shear strength observed at $P_a = 2{,}920$ kg/cm² was associated with the appearance of lamination lines in the compact. Compaction to greater pressures resulted in compacts which split into horizontal layers when ejected from the die and which expanded radially to such an extent that they could not be inserted into the bore of the shear strength apparatus.

The maximum shear strength of compacts prepared from sucrose, hexamine, and hexamine granulation also corresponds to the appearance of lamination lines, but aspirin compacts reach a maximum strength with no apparent fracture lines in the compact. The compaction pressures at which maximum shear strength is attained are given in Table 2.

The applied pressure for hexamine is that at which the compact approaches zero porosity under load (Fig. 3B) and a maximum density when ejected. Aspirin is fully consolidated under a load of approximately 1,000 kg/cm² and the pressure listed in Table 2 is that at which the porosity of the ejected compact becomes constant (Fig. 2B). Sucrose behaves in a similar manner to aspirin (Fig. 3A).

For these materials it would appear that the maximum shear strength under zero load conditions is associated not with the pressure at which consolidation is complete when compressed, but with the pressure necessary to produce minimum porosity in the ejected compact. Sodium chloride does not fit into this concept, for although the porosity of the ejected compacts becomes constant at applied pressures of 3,000 kg/cm²

TABLE 2. APPLIED PRESSURE WHEN SHEAR STRENGTH BECOMES MAXIMAL

Material	Applied pressure, kg/cm²
Hexamine	1,500
Hexamine granulation ..	1,250
Aspirin	1,700
Sucrose	2,600
Sucrose granulation ..	1,715

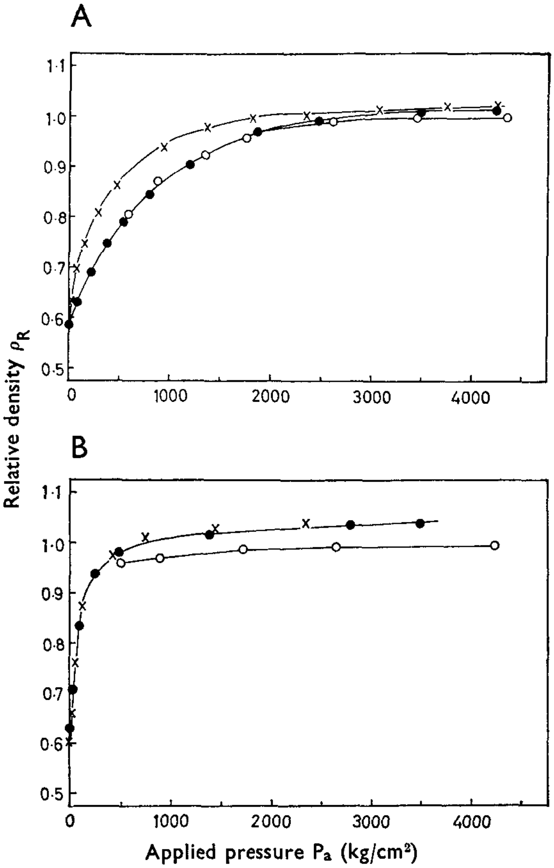

FIG. 2. Effect of applied pressure on density of compact. A. Sodium chloride. ● Unlubricated. × Lubricated. ○ Ejected and unlubricated. B. Aspirin. ● Lubricated. × Unlubricated. ○ Ejected and unlubricated.

and greater (Fig. 2A), the maximum value of shear strength is not achieved.

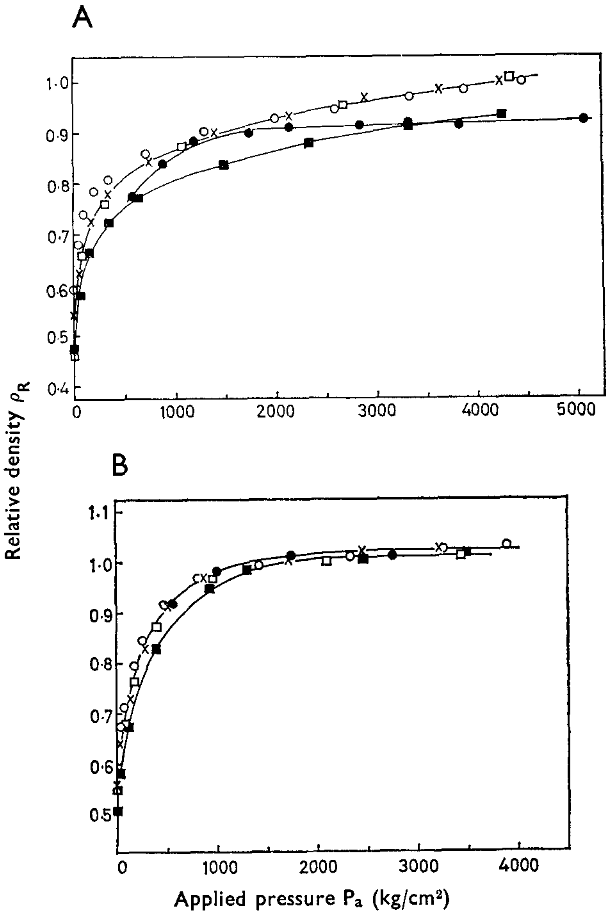

FIG. 3. Effect of applied pressure on density of compact. A. Sucrose. B. Hexamine. × Unlubricated. ○ Lubricated. ● Ejected and unlubricated. □ Granulation lubricated. ■ Granulation unlubricated.

TABLE 3. THE SHEAR STRENGTH OF COMPACTS UNDER AN APPLIED LOAD

Material	Shear strength kg/cm^2	Porosity %
Hexamine ..	471	nil
Aspirin	297	nil
Sodium chloride ..	968	nil
Sucrose	1,112	3·0

Each result represents the mean of three determinations.

The shear strength of compacts under a compressive load is given in Table 3.

Shear strength values for compacts under load are much greater than those for compacts under zero load, as might be expected from the results of Bridgman (1952) and Hersey (1960).

From the present results three values of shear strength of a compact may be chosen:

(1) Shear strength of a compact prepared at a given pressure and measured under zero load conditions, S_p;

(2) Maximum observed shear strength of a compact under zero load conditions (as in Fig. 1), S_m;

(3) Shear strength of a compact measured at zero porosity, S_o.

DIE REACTION

Hersey (1960) indicated that the friction theory of Bowden and Tabor was applicable to his compacting systems and that the die reaction,

$$F_d = S.A \qquad \ldots \quad \ldots \quad \ldots \quad \ldots \qquad (2)$$

where S = shear strength of material being compressed;
A = true area of die wall—compact interface.

If the shear strength of the material remains constant, it would be expected that the die reaction would attain a maximum value when the porosity of a powder compact became zero.

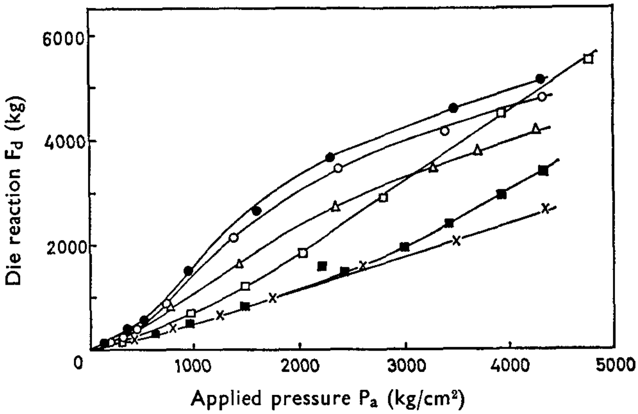

FIG. 4. Effect of applied pressure on die reaction. ○ Hexamine. ● Hexamine granulation. □ Sucrose. ■ Sucrose granulation. × Sodium chloride. △ Aspirin.

It was found that practical values of F_d did not attain a maximum at pressures up to 4,500 kg/cm^2 (Fig. 4), although the increase in F_d with pressure becomes less for hexamine, aspirin, and hexamine granulation, at applied pressures greater than 2,300 kg/cm^2.

The apparent area of compact—die wall contact, A_a, at a given compaction pressure can be calculated from observations of the length of compact. If it is assumed that the porosity is uniform throughout the compact, the true area of contact for compacts less than theoretical density is given by:

$$A = A_a.\rho_R \qquad \ldots \quad \ldots \quad \ldots \quad \ldots \qquad (3)$$

$$\text{where } \rho_R = \text{relative density of compact} = \frac{\text{bulk density of compact}}{\text{density of solid}}$$

Values of F_d were calculated from equation (2) using experimental values of S_p and S_o, and are compared in Table 4 with the measured values for the four crystalline materials, over a range of applied pressures. At applied pressures greater than those necessary to produce maximum values of shear strength, F_d was calculated using S_m in place of S_p.

TABLE 4. COMPARISON OF EXPERIMENTAL RESULTS FOR F_d WITH CALCULATED VALUES

Material	Applied pressure Pa kg/cm²	Relative density under load ρ_R	Exptl. results F_d kg	Calculated F_d values	
				$A.S_p(S_m)$	$A.S_o$
Hexamine . .	4,000	1·03	4,500	1,099	2,878
	3,500	1·03	4,200	1,099	2,878
	3,000	1·02	3,850	1,110	2,905
	2,000	1·02	2,950	1,115	2,920
	1,500	1·01	2,300	1,127	2,950
	1,000	0·98	1,400	986	2,905
	500	0·91	450	432	2,698
	250	0·82	200	155	2,431
Sodium chloride . .	4,000	1·01	2,300	2,002	3,690
	3,000	1·00	1,800	1,772	3,725
	2,000	0·98	1,150	1,369	3,630
	1,500	0·94	800	1,085	3,500
	1,000	0·88	500	694	3,280
	500	0·78	250	270	2,907
	250	0·70	100	54	2,608
Aspirin . .	4,000	1·04	4,000	632	1,702
	3,000	1·04	3,200	634	1,713
	2,000	1·03	2,300	641	1,730
	1,500	1·02	1,700	627	1,739
	1,000	1·01	1,100	473	1,757
	500	0·98	500	176	1,720
	250	0·94	250	84	1,650
Sucrose . .	4,000	1·00	5,250	1,308	5,780
	3,000	0·97	3,850	1,269	5,610
	2,000	0·93	2,450	1,092	5,400
	1,500	0·90	1,750	870	5,230
	1,000	0·87	1,000	610	5,030
	500	0·81	400	325	4,700
	250	0·75	150	196	4,360

Consideration of values of $A.S_p$ (S_m) indicates that the discrepancy between calculated values and experimental measurements is greatest for aspirin, hexamine, and sucrose, at the higher compaction pressures. For sodium chloride and sucrose values of $A.S_o$ are larger than the measured values over the whole range of applied pressures used; for hexamine and aspirin the calculated values are larger than the practical results at low applied pressures, and smaller at high values of P_a. In the instance of aspirin, experimental and calculated values are comparable when $\rho_R = 1{\cdot}02$; the two values for hexamine are comparable when $\rho_R = 1{\cdot}02$ at an applied pressure of 2,000 kg/cm². Of the results listed in Table 4 the best overall relation between experimental measurements of F_d and calculated values is for sodium chloride when shear strength values at zero load (S_p) are used in the calculations.

The material being compacted undergoes maximum shear at the die wall, sufficient in the case of lead shot to cause the production of a compact held together by the outside skin (Train & Hersey, 1960b). It is likely, therefore, that for the softer materials hexamine and aspirin, the true area of contact at the die wall at low pressures will be greater than that predicted from porosity observations, and would account for calculated values of F_d being less than the practical results. The higher practical values for F_d when P_a is large may be due to extrusion of the compacting material past the punch tip—of which there was some evidence—making the actual area of contact greater than the calculated value; or the shear strength of the material may be greater at these high applied pressures than the value measured at zero porosity.

To test this latter statement is beyond the capability of the apparatus available at present, but it is considered that the amount of extrusion of material that took place was insufficient to account for the discrepancy between practical and calculated results.

EJECTION FORCE

Figure 5 depicts the effect of applied pressure on ejection force. Of note is the much larger force needed to eject compacts of crystalline hexamine as compared to the granulation, since the latter would be expected to present a larger surface to the die wall at any one pressure than would the crystals because the granules were friable and the basic particle smaller.

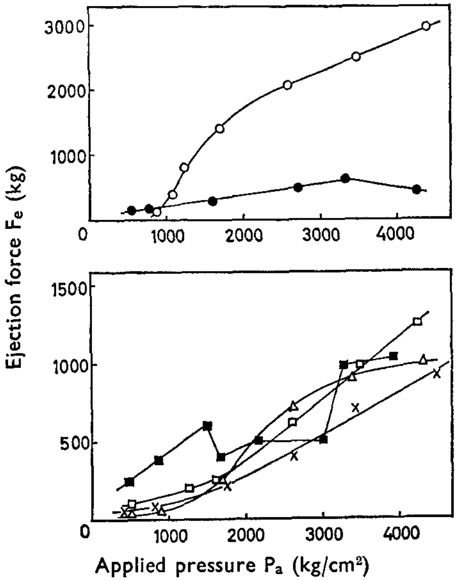

FIG. 5. Effect of applied pressure on ejection force. ○ Hexamine. ● Hexamine granulation. □ Sucrose. ■ Sucrose granulation. △ Aspirin. × Sodium chloride.

Ejection forces for compacted sucrose granulation increase at first with increased compaction pressure but then decrease when $P_a =$

1,770 kg/cm^2 before again increasing. This decreased ejection force occurs at the point where lamination of the ejected compact becomes noticeable and where the compact undergoes appreciable expansion. It is possible that at applied pressures in the range 1,770–3,000 kg/cm^2 the compact is subject to axial recovery after removal of the load, causing the compact to laminate in the horizontal plane thus decreasing the residual radial stresses acting on the die wall. If radial stresses are diminished and all other conditions are the same, then a decreased ejection force would be needed.

Attempts to correlate numerical values of ejection force with the shear strength of the compact were completely unsuccessful, and visual observations of the behaviour of the compacted material during the experimental work indicated that the amount of elastic recovery of the compact after removal of the compacting pressure and before ejection, may well be as important as the shear strength of the friction junctions.

THE RELATIVE DENSITY OF THE COMPACT, ρ_R

Hexamine and aspirin attain zero porosity under load at comparatively low compaction pressures (Figs 2B, 3B), and the presence of lubricant has little influence on the degree of consolidation attained at any one compaction pressure. Sucrose and sodium chloride are more difficult to consolidate when unlubricated, and the addition of a lubricant causes these materials to attain a greater density at the lower pressures (Figs 2A, 3A).

The unlubricated granulations of hexamine and of sucrose are found to be more resistant to consolidation than the respective crystalline material, although the behaviour of lubricated samples is similar to that of the crystals. This behaviour has also been observed in tablets compressed with an instrumented tablet machine (Lewis, 1964). It is considered that this resistance is due to the presence of air trapped in the unlubricated granulation, whilst the lubricant facilitates particle sliding and possibly interferes with particle bonding in the early stages of compaction so allowing the air to escape.

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