DRUG DEVELOPMENT AND INDUSTRIAL PHARMACY, 4(2), 131-156 (1978)

WORK REQUIRED TO CAUSE FAILURE OF TABLETS IN DIAMETRAL COMPRESSION

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ABSTRACT

During diametral compression testing load-displacement curves were obtained for tablets made from some direct compression agents, by continually monitoring the force applied to the tablet and the distance moved by the lower compression platten. The area under these curves, termed work of failure, was calculated by numerically integrating the applied force with respect to platten displacement. The resistance to breakage of the tablets was assessed by a semi-empirical multiple diametral impact test in which a tablet was subjected to repeated impact until failure occurred. Work of failure was related to the number of impacts required to cause failure and appears to be a better quantitative assessment of a tablet's mechanical properties than tensile strength.

INTRODUCTION

The compaction properties of pharmaceutical materials are commonly assessed by the force required to break a tablet in a diametral compression test. As shown originally by Carneiro and Barcellos[1], this type of test can be used to determine tensile strength. Nutter-Smith[2] comments that the Monsanto device was in use as early as 1933 for measuring the resistance of tablets to diametral crushing - a property which is often imprecisely referred to as 'hardness'. Instruments such as the Strong-Cobb and Monsanto (Stokes) have been widely used by the pharmaceutical industry, but during the past decade these and other instruments have been criticised due to variation in measurements between operators and between instruments[3,4]. Motorised instruments such as the Schleuniger (Heberlein) which minimise such variation[5], are now in common use. Although the advantages of further sophistication, including the use of transducers for force measurement, are generally recognised, instruments incorporating such improvements are not used for routine quality control, mainly on the grounds of cost.

Schubert[6] defined tensile strength as:-

> "The unidirectional maximum tensile force per unit of the plane cross sectional area of the bulk material at right angles to the direction of tension when a locally constant, purely tensile

stress prevails in the fracture cross-section of the material regarded as a continuum".

If a disc of elastically isotropic, homogeneous material is compressed diametrally between knife edges, it has been shown theoretically by Timoshenko and Goodier[7] that there is a principal, constant magnitude tensile stress, σ, along the diameter at right angles to the applied load:-

$$\sigma = \frac{2P}{\pi Dt} \qquad \text{Equation 1}$$

where P is the applied load, D is the diameter of the disc and t is its thickness.

In this state of ideal line loading high compressive stresses will prevent tensile failure of the specimen. If parallel plattens are substituted for knife edges, Frocht[8] has shown that, provided the length of the contact surface formed between the tablet and plattens is small compared to the diameter of the disc, the major principal stress is similar to that produced by knife edges except in the regions close to the contact surfaces where the compressive stresses are considerably reduced. Pharmaceutical tablets are not elastically isotropic, homogeneous materials. Nevertheless, failure in tension can be ensured providing the conditions of the test minimise the shear and compressive stresses[9]. In practice the state of ideal line loading is seldom achieved, except for tablets of high elastic modulus because tablets deform in the region of contact with the relatively hard metal platten. This deformation, provided it is not excessive, minimises shear and compressive stresses near the plattens and allows failure to occur in tension.

Schubert, Herrmann and Rumpf[10] working with agglomerates suggested that it is often necessary to study stress-strain behaviour to fully characterise a material. They also stated that if the strain at maximum load (fracture strain) is small an agglomerate will be more sensitive to cracking than if the fracture strain is large, although the tensile strength may be identical. This phenomenon is known in metallurgy as 'toughness' which Dieter[11] described as "the ability to absorb energy in the plastic range". Dieter[11] suggested that one way of assessing toughness is to consider the area under stress-strain curves. For example, in Fig.1 material I has a slightly higher tensile strength than material II, but a considerably lower fracture strain ε_I. The area under the stress-strain curve for material II is therefore larger than for material I. Since this area is an indication of the work that can be done on a material without causing fracture, material II is tougher than material I.

Pharmaceutical tablets must withstand various conditions of stress in coating equipment and during packaging and transport. Even though the force required to break a tablet is used almost universally by the pharmaceutical industry as a quality control

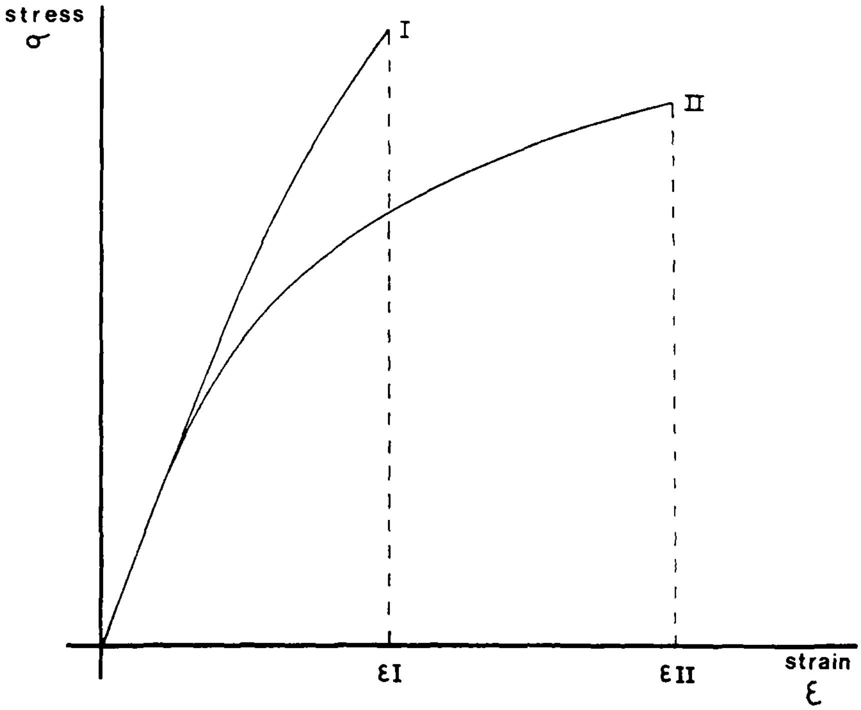

FIGURE 1
Stress-strain curve for two materials in a tensile test.

assessment of this resistance to mechanical failure, it is well known that specifications for different formulations cannot be compared quantitatively. Thus, for a particular type of tablet to withstand mechanical shock and abrasion under specific conditions of processing or transport it may require a considerably higher breaking strength than tablets of a different drug substance or of the same drug substance formulated differently. It would be useful if a test could be devised, compliance with which would ensure resistance to mechanical failure of all types of tablets under given conditions. Toughness of tablets might be a useful basis for quantitative comparison of materials and formulations. We therefore investigated the usefulness of displacement measurements during diametral loading tests with tablets of various excipients recommended for direct compression.

MATERIALS

The following materials were used as received from the suppliers; Elcema G250 - a granular form of microfine cellulose (Degussa, Frankfurt, West Germany), anhydrous lactose (KW-Revai Chemicals Limited, London, U.K.), Emcompress - a direct compression form of dicalcium phosphate dihydrate (K.K. Greef Limited, Croydon, U.K.), Sta-Rx 1500 - a modified Starch U.S.P. (A.E. Staley Manufacturing Co., Illinois, U.S.A.), sodium chloride A.R. (Fisons Scientific Apparatus, Loughborough, U.K.), magnesium stearate (Hopkins and Williams Limited, Essex, U.K.).

METHODS

Preparation of Tablets

Sufficient material to produce a compact, 2.49 mm thick at zero theoretical porosity, was weighed to ± 2 mg, poured into a 12.7 mm diameter die and compressed between plane faced punches, using a Wilkinson STD1 reciprocating tablet machine. The die was previously lubricated by compressing a sample of the same material containing 50% w/w magnesium stearate. Applied compaction forces were monitored using two active foil strain gauges and two temperature compensating gauges type 3/120/EC (Tinsley Telcon Limited, London, U.K.), bonded to the shank of the upper punch. The signal from the strain gauge bridge was conditioned using a bridge balance and amplifier unit types 492BBS and 251GA (Fylde Electronic Laboratories Limited, Preston, U.K.) and recorded using a U.V. oscillograph type 3006 (S.E. Laboratories (Engineering) Limited, Feltham, U.K.) fitted with type 'A' galvanometers (Manarp Electronic Instruments Limited, Hayes, U.K.). All materials and compressed tablets were stored at 50% R.H., 24°C before use.

Testing of Tablets

Tensile strength and work of failure: The thickness of a sample of ten tablets was measured using a micrometer. The tablets were then tested in diametral compression between parallel plane metal plattens installed in a Hounsfield tensiometer modified for use in compression. The force applied to each tablet during the test was monitored continually using a load transducer (Interface 1410-AF, range 0-45 kg, Interface Inc., Arizona, U.S.A.). A displacement transducer (linear range ± 2.5 mm, R.D.P. Electronics Limited, Wolverhampton, U.K.) was used to measure the corresponding distance between the plattens. The rate of lower platten movement was 0.26 mm min^{-1} unless otherwise stated. The arrangement of the transducers is shown diagrammatically in Fig.2.

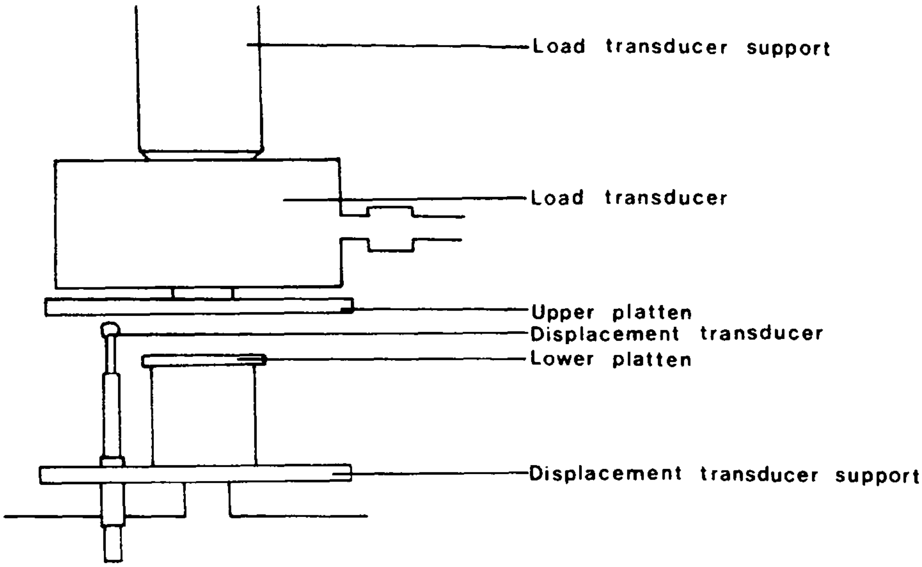

FIGURE 2
The arrangement of transducers on the tablet testing instrument.

The analogue signals from the two transducers were sequentially scanned and digitised by a Data Transfer Unit in conjunction with an A200 Digital Voltmeter (Solartron Electronics Group Limited, Farnborough, U.K.). The digital values were recorded on punch paper tape.

From the values of breaking load, the tensile strengths were calculated using equation 1. The work done on the tablet during the test (work of failure) was determined by numerical integration of applied force with respect to platten displacement, using the trapezoidal rule.

Multiple Diametral Impact Testing: To simulate conditions to which tablets might be subjected in practice, a multiple impact test was devised. Using a modified Engelsmann Jolting Volumeter (J. Engelsmann AG., Ludwigshafen, West Germany) shown diagrammatically in Fig. 3, a mass of 240 g was dropped a distance of 2.4 mm at a frequency of 4 Hz on to the edge of a tablet located diametrally between metal plattens. The number of times the load was applied before breakage occurred was counted. The mass of 240 g was selected empirically so that values of greater than one were obtained for all tablets studied.

Batches of 20 tablets were compressed from each material at a suitable force to produce tablets of similar tensile

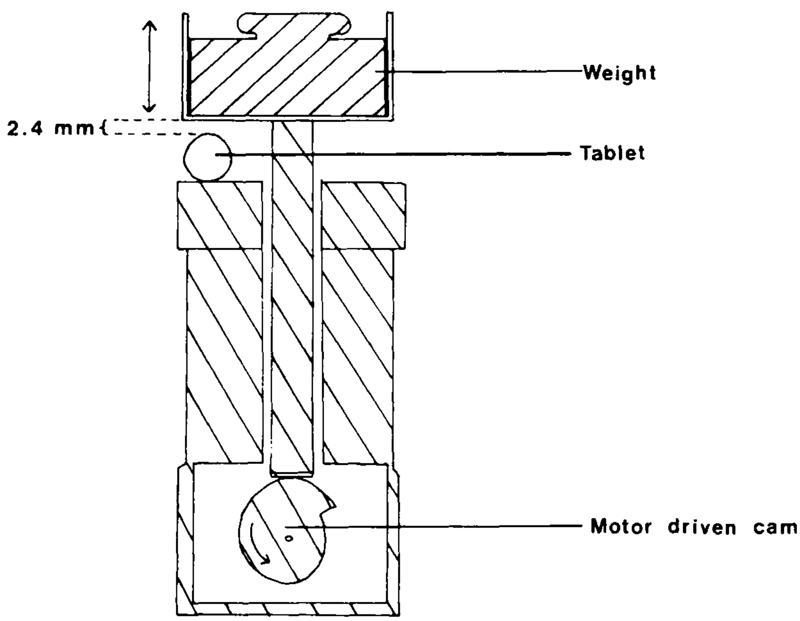

FIGURE 3
Modified jolting volumeter.

strength. Ten tablets were tested for tensile strength and work of failure, and ten tablets were subjected to multiple impact testing.

RESULTS AND DISCUSSION

For each of the materials studied, the relationship between tablet tensile strength and compaction force is non-linear (Fig.4). The characteristic sigmoidal shape of the curves is probably indicative of the different ways in which the work of compaction is utilised at various stages of tablet formation. The first stage as suggested by Seelig and Wulff[12] involves rearrangement and closer packing of particles, work being done to overcome particle-particle and particle-die wall friction. As compaction continues elastic and plastic deformation of the particles takes place, possibly associated with fragmentation, and bonds are formed between the particles. Finally elastic compression of the bulk compact occurs. In Fig. 4 the initial slope of the curve AB suggests that particle rearrangement and slight distortion of surface asperities cause only small changes in tensile strength. As compaction proceeds the slope of curve AB increases indicating an increasingly rapid change in tensile strength as more extensive deformation of asperities and whole particles occurs; this increases the area of interparticulate contact which, in the materials studied, must be associated with bond formation. The low values of tensile strength in region AB cause difficulties in measurement. This fact, coupled

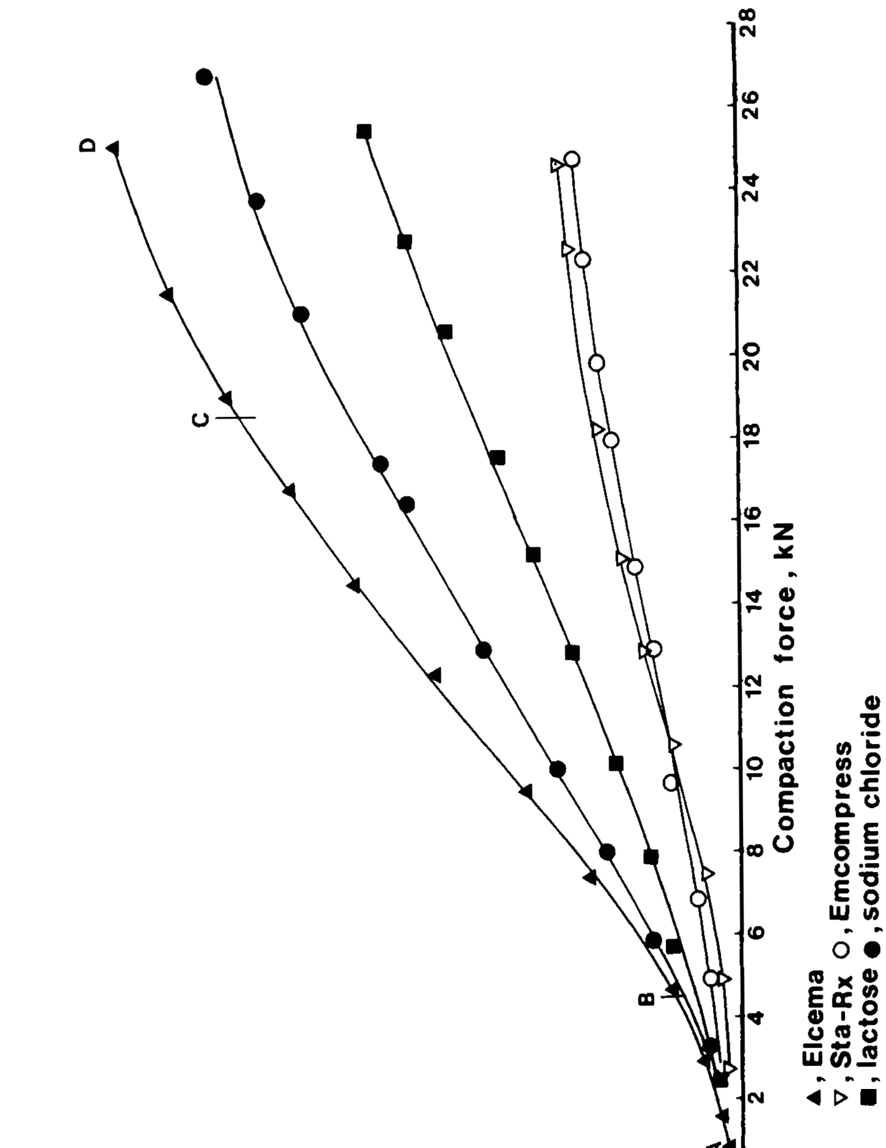

FIGURE 4
The relationship between compaction force and tensile strength.

with the relatively large intertablet variation may explain the conclusion of earlier workers[13,14] that curve BC can be extrapolated to indicate the compaction force below which a coherent compact is not formed. Furthermore, we suspect that if precise measurements of tensile strength could be performed at low values of compaction force, materials which fragment might exhibit a slight increase in tensile strength due to closer packing, followed by a temporary decrease as particle fracture predominates. Evidence for this might be difficult to obtain due to the non-isotropic structure of consolidating material. During stage BC, deformation of the particles at first causes a rapid reduction in void volume associated with particle bonding, but eventually more work must be done to produce an equivalent increase in the area of surface contact. At this stage the slope starts to decrease. Eventually a state of maximum consolidation is approached and an increasing proportion of the work is used for elastic compression of the bulk compact; thus during stage CD the gradient of the curve decreases more rapidly showing a smaller rise in tensile strength with compaction force.

Typical diametral load-displacement curves for tablets of various materials subjected to diametral compression testing are shown in Fig. 5. Each curve shows a linear relationship, followed by a curve of decreasing slope. One might conclude that this indicates plastic deformation preceded by a linear elastic

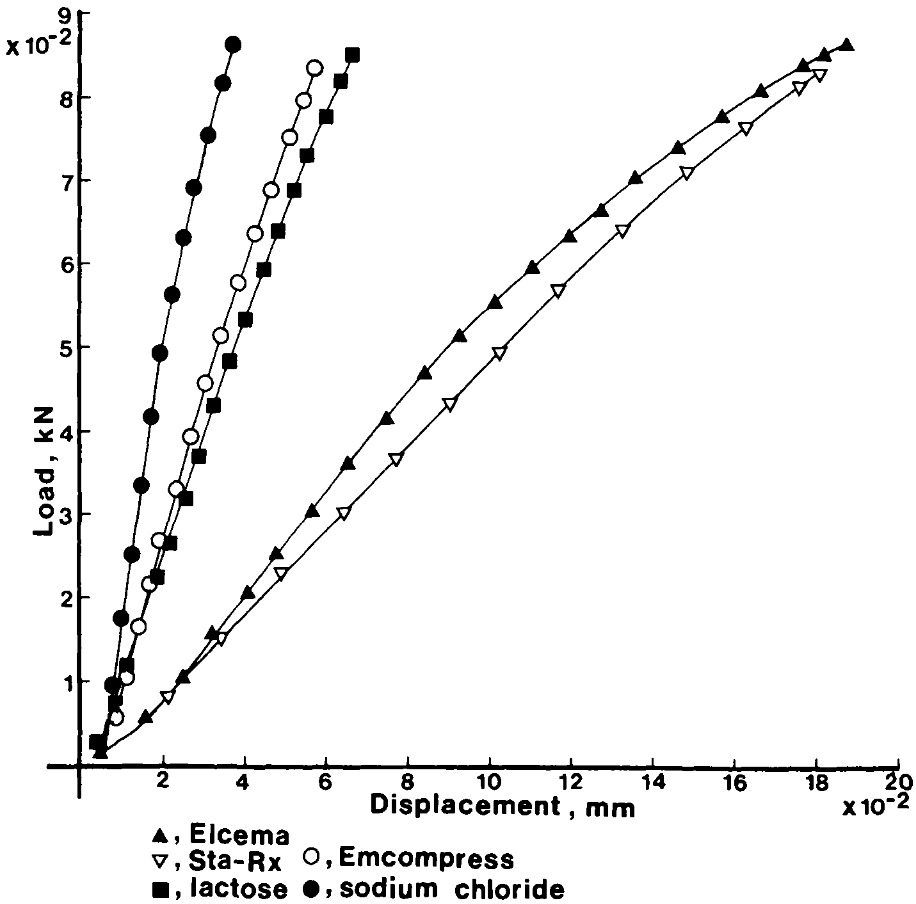

FIGURE 5
Diametral load displacement curves for tablets of similar tensile strength.

region where loading and unloading would follow the same line. However this is not necessarily true. Dieter[11] referring to metals suggested that the true elastic limit may be very low, "if indeed one exists at all". Figs. 6 and 7 show the diametral load-displacement curves for tablets of anhydrous lactose and

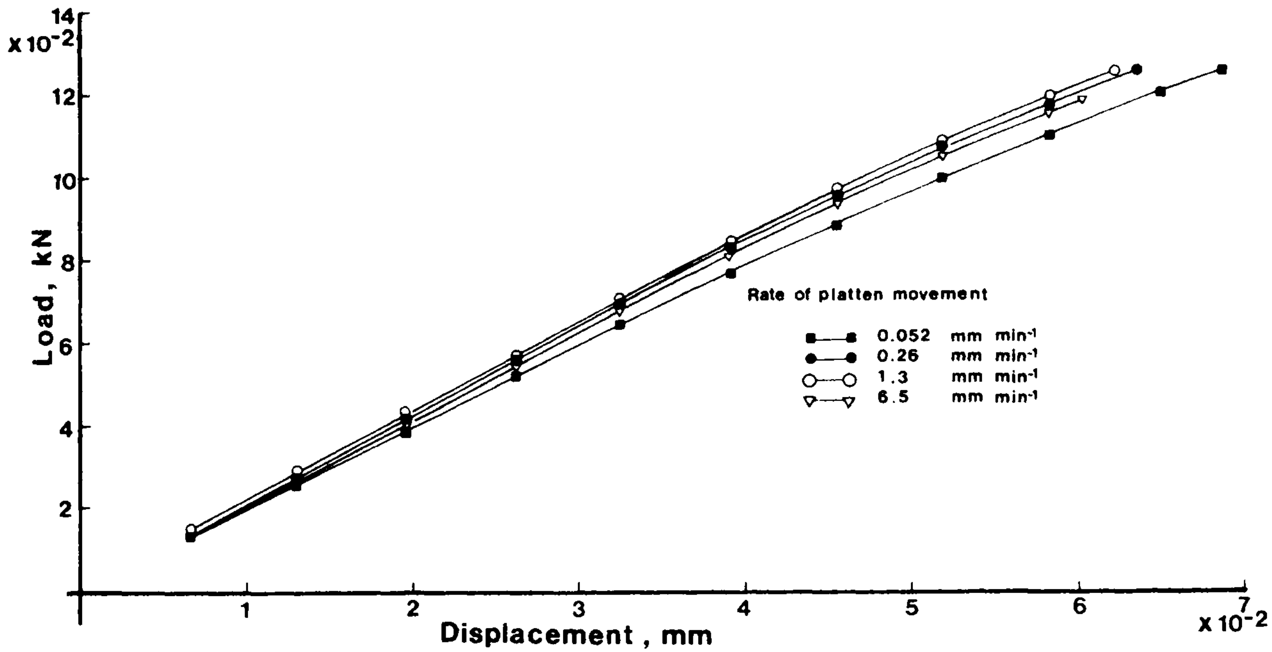

FIGURE 6
Diametral load displacement curves for anhydrous lactose tablets at four rates of loading.

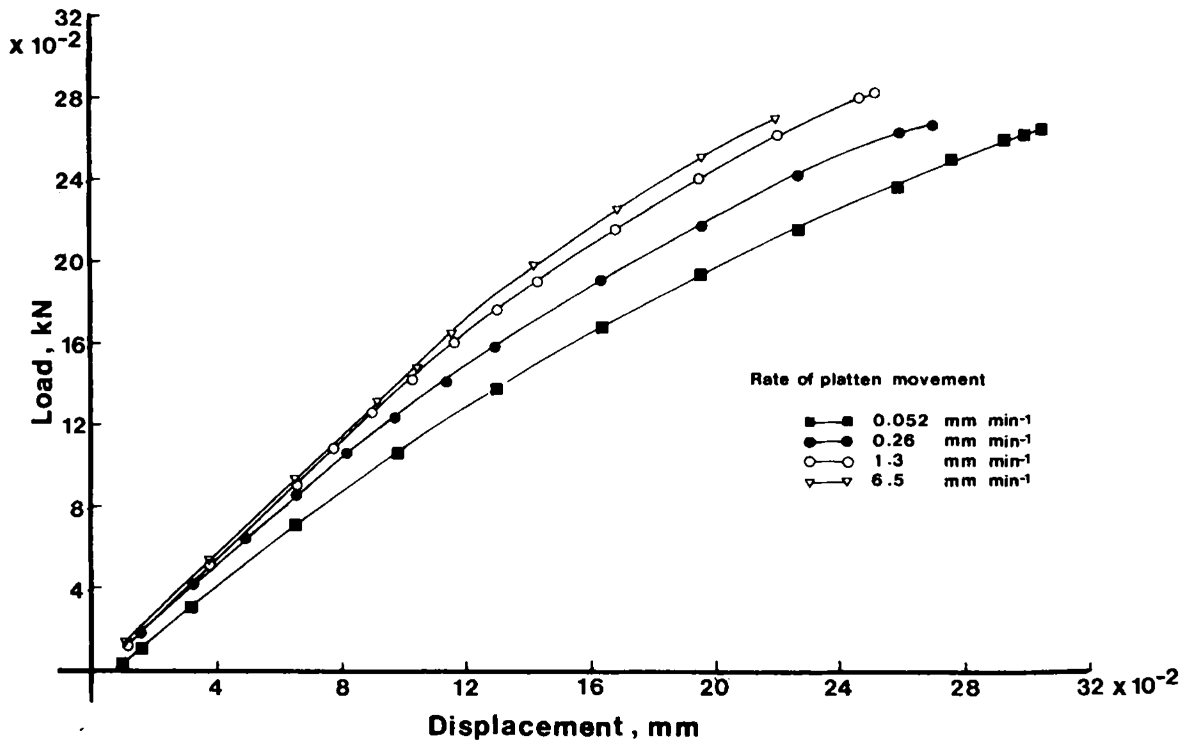

FIGURE 7

Diametral load displacement curves for Elcema G250 tablets at four rates of loading.

Elcema at four rates of loading. The rate has little effect on the more brittle lactose. In contrast, with Elcema as the rate of loading decreases there is a decrease in the 'apparent elastic limit', represented by the limit of linear behaviour, until at a rate of 0.052 mm min^{-1} there is no discernible linear behaviour during the entire loading curve. This time-dependent lowering of the 'apparent elastic limit', suggests that there is no true elastic limit for the more plastic materials. This is not unexpected since it is known that higher rates of loading tend to cause brittle fracture, the stress-strain curve for an ideal brittle material being completely linear to the point of fracture.

The work done by the plattens to induce failure of a tablet is related to the 'toughness' of the tablet as defined by Dieter[11]. However since the measured displacement is not a tensile strain, but a deformation of the compact in the direction of compressive loading, we have used the term "work of failure".

Figs. 8 and 9 show the relationship between work of failure and compaction force for the five direct compression materials studied; the curves show a sigmoidal profile similar to that observed for tensile strength (Fig. 4). Considering the two extreme cases, the tensile strength of Elcema tablets is 3 to 3.5 times that for Emcompress whereas the work of failure for Elcema tablets is 10 to 20 times that for Emcompress,

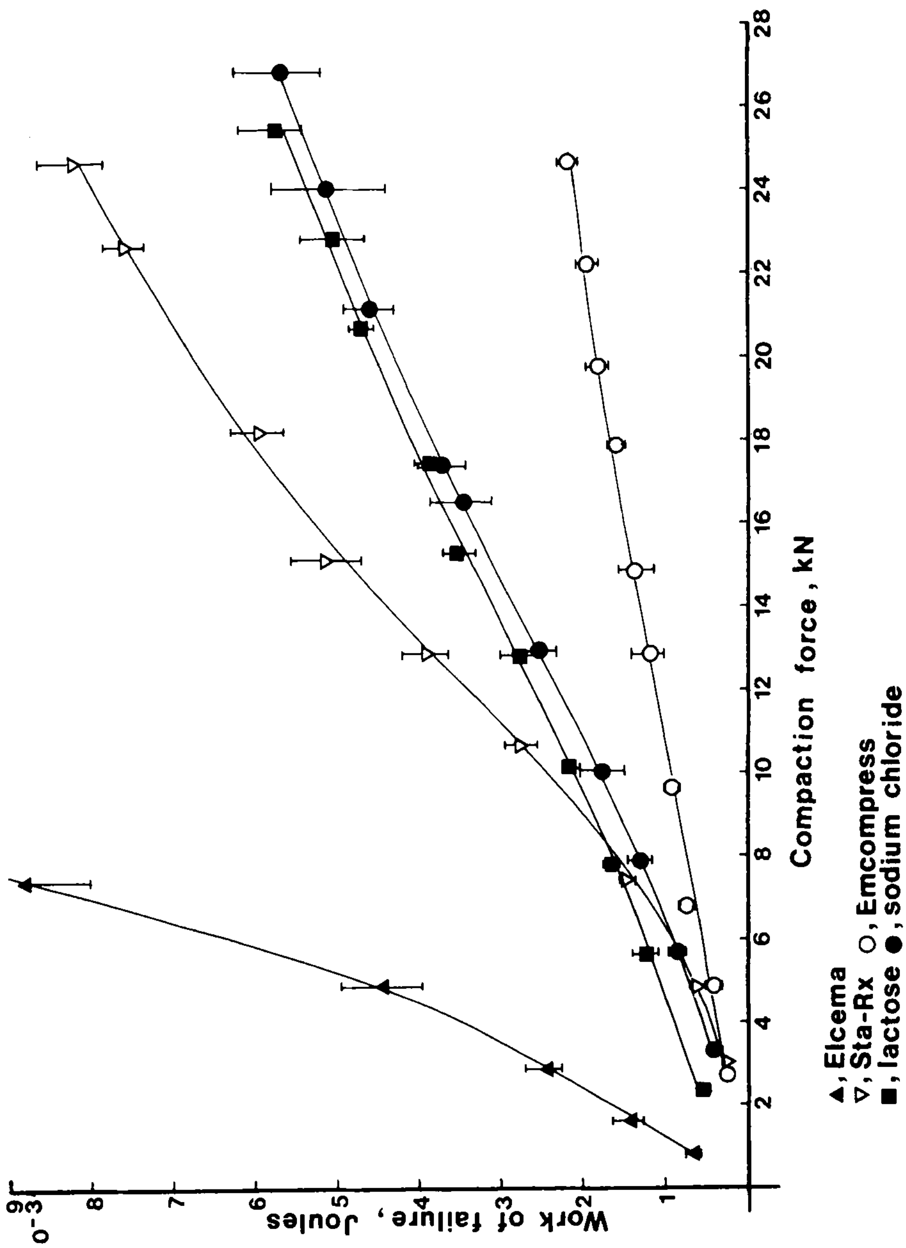

FIGURE 8 The influence of compaction force on work of failure, showing the mean values and standard errors.

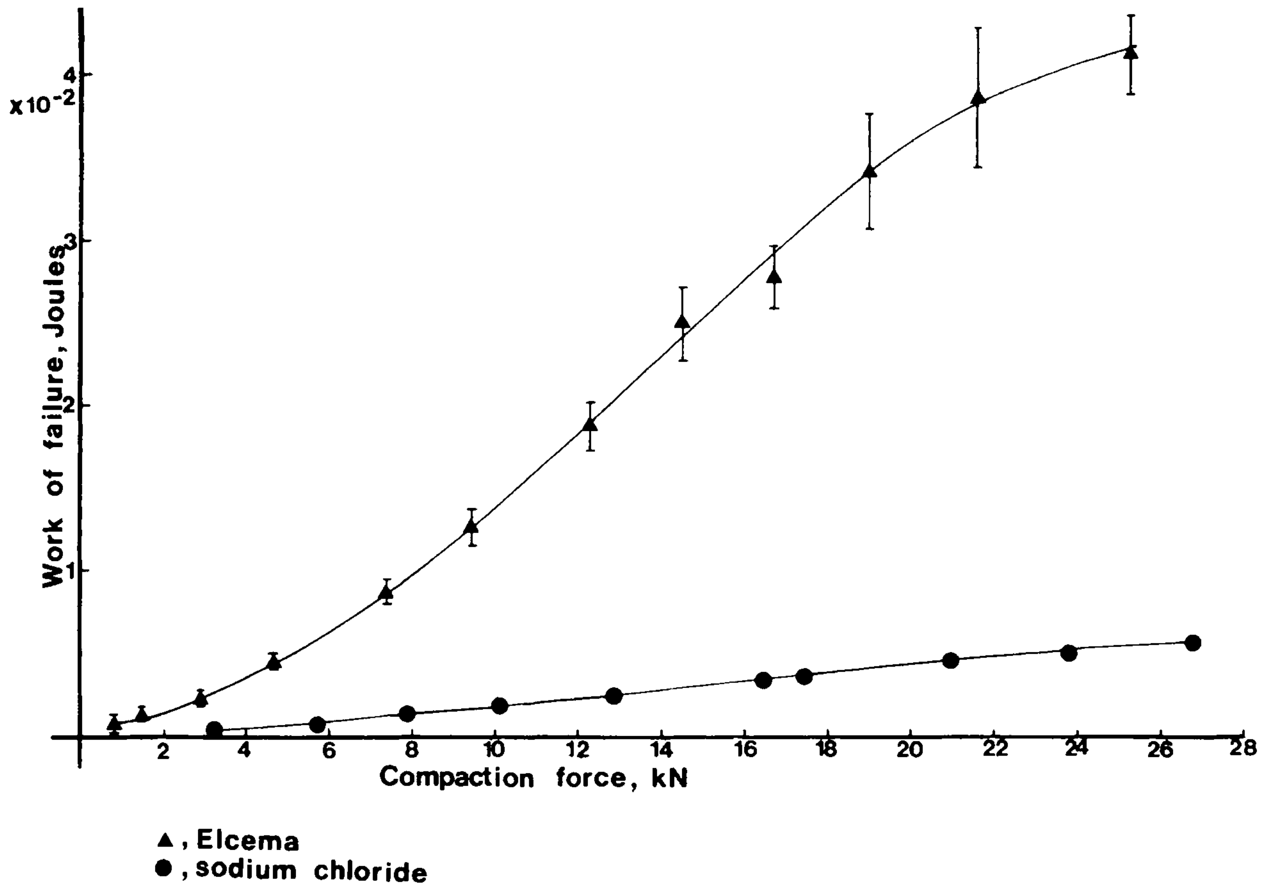

FIGURE 9 The influence of compaction force on the work of failure of Elcema G250 and sodium chloride tablets, showing the mean values and standard errors.

these ratios increasing with an increase in compaction force. For tablets of equal tensile strength, the work of failure for Elcema is therefore considerably higher than that for Emcompress since the Elcema tablets deform more extensively under load.

The distances moved by the platten in diametral compression tests on various tablets are compared in Fig. 10. Materials such as Elcema, which deform plastically as shown by the considerable stress relaxation following tablet compaction[15] and the high values for tensile strength (Fig. 4), require a relatively large platten displacement before tablet failure occurs during diametral compression. The considerable increase in displacement with compaction force is presumably due to the relatively large areas of interparticulate bonding created at higher compaction forces which can accept more plastic strain before diametral failure occurs. In contrast, tablets of a brittle material such as Emcompress or lactose require only a small diametral displacement to cause failure. Compaction force has little effect on the diametral displacement, presumably because for a brittle material the effect of increasing compaction force is primarily to increase the number of brittle interparticulate bonds, rather than to increase the area of existing points of contact as in the case of a plastic material.

Sodium chloride exhibits apparently anomalous behaviour; small diametral displacements were recorded although extensive plastic deformation is known to occur during tablet

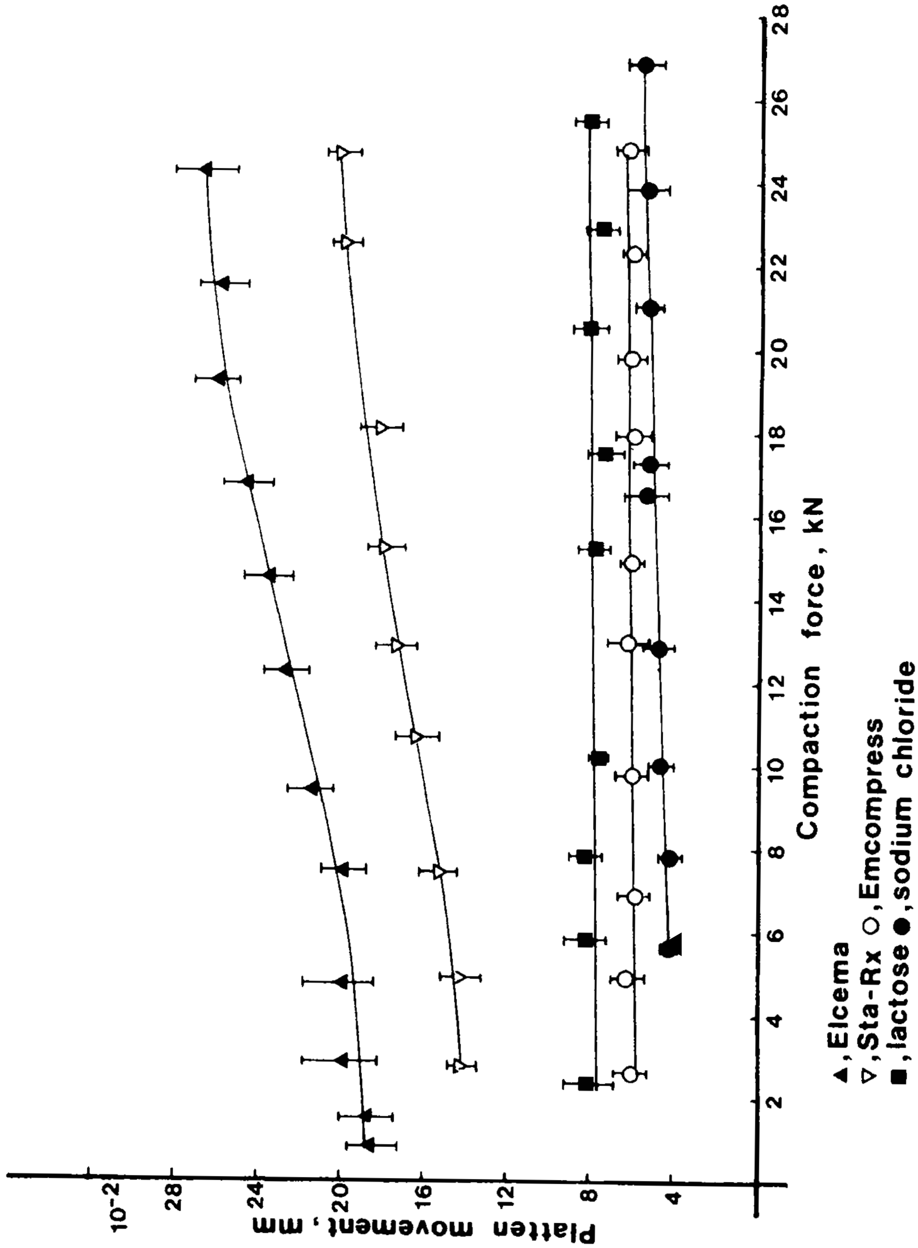

FIGURE 10 The relationship between compaction force and platen displacement during diametral loading of tablets.

formation[16,17]. We suspect that the phenomenon is due to brittle behaviour of interparticulate bonds as a result of work hardening following extensive deformation at the points of contact during compaction.

Despite the small diametral displacements recorded for sodium chloride tablets compared with other materials investigated intermediate values for work of failure are obtained. This is because of the relatively steep tensile strength-compaction force profile for sodium chloride (Fig. 4). Conversely a material such as Sta-Rx which produces tablets of very low tensile strength (Fig. 4) requires a higher work of failure than sodium chloride because the tablets deform by a large amount in a diametral compression test. The work of failure for Elcema tablets is exceptionally high (Figs. 8 and 9) due to the combination of high tensile strength and large diametral displacement.

The results for the semi-empirical multiple impact test, shown in Table 1 and Fig. 11, indicate a relationship between work of failure and the number of impacts required to break a compact. The effective stress imposed on the tablets during multiple impact testing is an unknown combination of compressive, shear and tensile stresses. This probably explains the large coefficient of variation for the number of impacts required to cause failure. For some tablets extensive fragmentation was seen, whereas for others failure occurred along a diameter.

TABLE 1

Multiple Impact Test Data for Tablets of Similar Tensile Strength

Material	Diametral compression test			Number of impacts to cause failure	
	Mean tensile strength ($MN\ m^{-2}$)	Work of failure (Joules)			
		Mean	Coefficient of variation %	Mean	Coefficient of variation %
Elcema G250	1.67	1.51×10^{-2}	5.4	4360	11.8
Sta-Rx 1500	1.36	7.01×10^{-3}	8.8	322	28.3
Anhydrous lactose	1.59	3.45×10^{-3}	8.1	41	17.1
Emcompress	1.63	3.05×10^{-3}	3.6	28	17.9
Sodium chloride	1.85	2.57×10^{-3}	12.8	5	20.0

We suggest that the relationship between work of failure and the number of impacts required to break a tablet indicates that the work of failure is a more useful quantitative assessment of a tablet's mechanical resistance to damage than the tensile strength. Furthermore, we speculate that it may be possible to establish specifications based on work of failure which would ensure that all tablets which met such specifications, regardless of their formulation or method of production, would withstand the mechanical treatment in a particular type of coating equipment or during packaging and transport. Work of failure measurements would be necessary only during the dosage

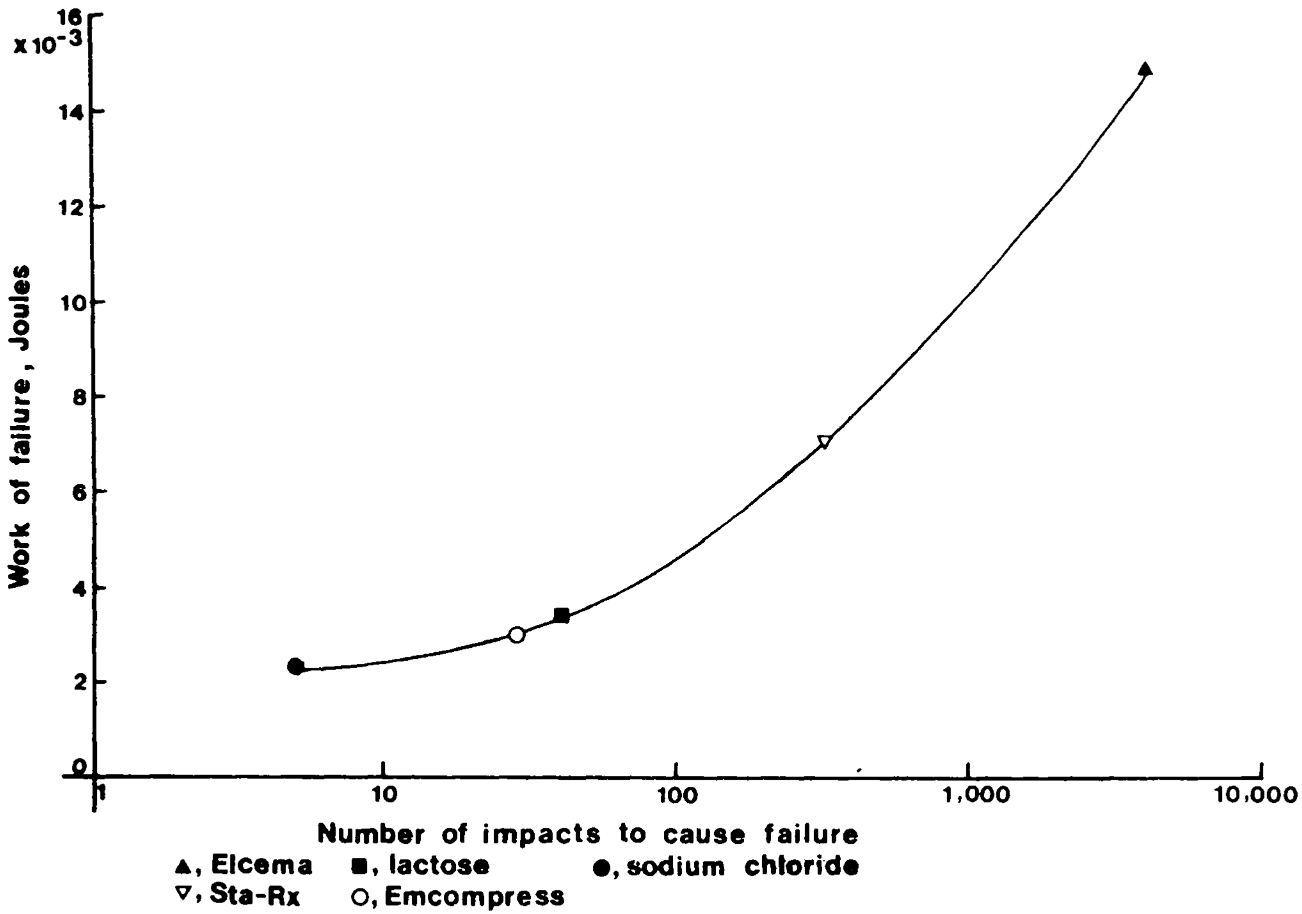

FIGURE 11 The relationship between work of failure and number of impacts to cause failure for tablets of similar tensile strength.

form design stage or if changes in the tablet formulation or production methods were made. Breaking force, or possibly tensile strength, would still be used as a routine quality control parameter since, for relatively small unintentional variations in the manufacturing process for a particular formulation, a direct relationship with work of failure could be assumed.

CONCLUSIONS

Tablets of identical tensile strength prepared from different direct compression excipients vary considerably in their resistance to failure. The work of failure, determined by integrating the force applied with respect to platten movement in a diametral compression test is related to the property "toughness", as defined by Dieter[11], and showed large differences for tablets of similar tensile strength. In general, materials which deform plastically during powder compaction tend to require a high work of failure due to their high tensile strength and because they undergo considerable deformation before failure occurs in a diametral compression test. Conversely, a low work of failure is observed with tablets of brittle materials. Work of failure is related to the number of impacts required before tablets fail in a semi-empirical multiple diametral impact test and may therefore be a suitable quantitative measurement for comparing tablets of different materials in terms of their resistance to mechanical failure.

ACKNOWLEDGMENTS

We wish to thank Mr. G. N. Elson and the technical staff of Aston Services Ltd. and the Physics Department, University of Aston for advice and technical assistance with design and construction of the test instrument and recording systems.

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