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"STUDIES OF THE EFFECT OF COMPACTION FORCE ON DISPLACEMENT OF LARGE COMPACTS FORMED FROM DIRECT COMPRESSION MATRICES"

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ABSTRACT

Large compacts (3.93 cm x 4 cm) formed from commercial direct compression bases have been prepared by hydraulic compression and then loaded in axial compression when removed from the die.

Avicel and Sta-Rx formed compacts resistant to shear, and failure in free axial compression was due to induced tensile stresses both radial and circumferential. Paracetamol DC, a direct compression form of paracetamol, behaved as a brittle solid and split axially along its length. Emdex and Encompress formed compacts weak in shear and failed along double shear cones at low axial loading.

Emdex gave force v displacement plots for both axial and radial displacement which suggests that it flows plastically at low loads insufficient to cause failure. The other compacts behaved as elastic solids at low axial loadings.

A method was devised to compare radial pressure at various points along the axial length of the compacts when they were formed or recompressed in the die. Although easily ejeeted, compacts of Avicel and Sta-Rx were the best transmitters of radial pressure. Emdex and Encompress compacts were both difficult to eject. It is suggested that this is due to shear failure and

and rebonding along the shear cones demonstrated in free axial compression.

During the past decade the pharmaceutical industry has been greatly attracted by the simplicity and economic advantages of the commercial direct compression bases and these have been the subject of many published studies. Most, however, such as that of Esnard and others[1], have dealt mainly with formulation problems and the properties of the compressed tablets. Data on the physics of their compression, though increasing, is relatively scarce.[2, 3.]

This paper describes some force v displacement measurements during the compression of large compacts, (3.93 cm diameter and 4 cm high), formed from direct compression bases and also some tests performed on the free compacts. Though larger and geometrically dissimilar to normal tablets, this disadvantage was accepted since their size enabled point measurements to be made which would have been impossible with smaller compacts, and axial crushing tests were also feasible.

The work arose in part from an attempt to correlate the compressional behaviour of these bases with some results obtained by Aulton and others through long term indentation tests on tablets made from them, [4, 5.] Phenomena such as plastic and viscoelastic flow under load, which can be studied via indentation testing, are thought to influence whether or not the material is easily compressed.

MATERIALS AND METHODS

The bases used were Avicel PH 101, Emdex, Encompress, a direct compression paracetamol (Paracetamol DC) and Sta-Rx 1500. The preparation and properties of certain of these have been fully discussed by Mendell[6.] Paracetamol DC (Graesser Salicylates) has been stated by Obiorah and Shotton[7] to contain about 4% of gelatin hydrolysate as an aid to compression and Sta-Rx 1500, a directly compressible starch, has been the subject of a report by Manudhane and others[8].

All the bases were kept in well closed containers prior to use, but otherwise they were as received from the manufacturers. No internal lubricants were added.

For brevity, the unqualified use of the base name will also be used to refer to its compacts where the context demands it.

FORMATION OF THE COMPACTS

The punch and die assembly and the Denison hydraulic press have been fully described elsewhere[9], and the die assembly is sketched as part of figure 1a.

The die wall and punch were lightly lubricated with silicone grease which proved preferable to the solutions of fatty acids commonly used.[7].

Enough material was poured into the die to give a final compact height of 4 cm. In some cases, e.g. Avicel, it was necessary to compress lightly and refill the die before final compression in order to obtain this length. The compaction force was up to 203 kN equivalent to an axial pressure of 160 MN m^{-2}.

MEASUREMENT OF RADIAL MOVEMENT AT OUTER DIE WALL

The die wall was marked with a "stick on" cm scale and a displacement transducer (Sangamo NER/1.0 mm) mounted horizontally on a 'Tupnol' plastic frame (fig. 1a) so that an axial traverse could be made from the compact base to beyond its top.

To measure radial movement, the transducer was set at the required position and roughly zeroed with the adjusting screw s. A bias voltage obtained from a mercury cell and potentiometer gave an exact zero. The transducer output was fed to the X axis of an XY recorder (Bryans 24 000 A4).

The Y axis (force axis) was operated by an output from a displacement transducer built into the movement of the Bourdon pressure gauge of the press. For most measurements this was adjusted so that 1 cm movement on the recorder was equivalent

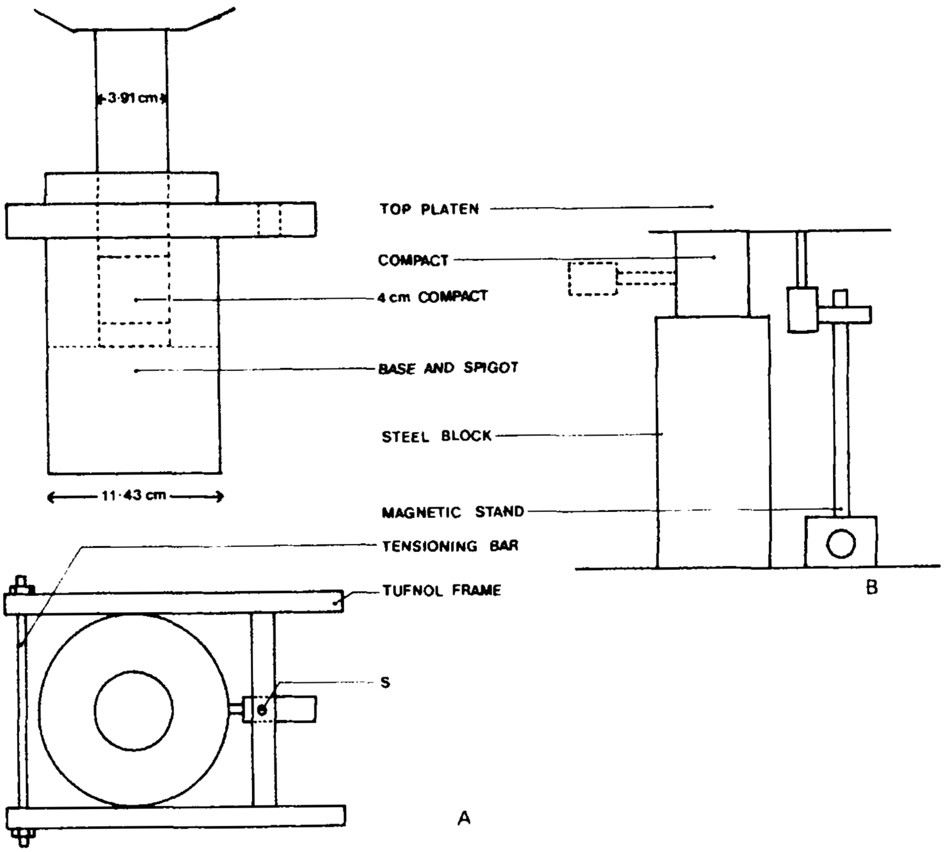

FIG. 1A The punch and die assembly FIG. 1B Loading of free compacts

to one ton (10 kN), but the sensitivity could be increased when required.

As the radial die movement was very small the transducer was previously calibrated by mounting in a jig incorporating a micrometer. The reading in mm equivalent to the whole available length of the X axis of the recorder was thus obtained under the conditions of use.

By mounting the transducer at various positions along the axial length of the die wall tracings such as those of figure 5a were produced. The traces for compact formation were measured at a point 1.25 cm above the compact base. Thereafter measurements were made by recompression of the formed compact after moving the transducer to the required positions.

Axial compression of the free compacts.

After ejection and measurement of the ejection force, a thin smear of silicone grease was applied to the compact ends and they were compressed axially between a static hardened steel block and the top platen of the press (fig. 1b). XY plots were obtained firstly as axial compression (X) v compression force (Y) and then as radial expansion v compression force with the displacement transducer (RDP Electronics Type D5/500A) relocated in the dotted position (fig 1b) and bearing on the mid point of the side. The maximum compression force was kept between 10-20 kN which in most cases was within the elastic limits of the compact. The transducer was then reset axially to obtain the XY plot when compression force was increased until fracture occurred. The X and Y scales were adjusted to give traces utilizing most of the graph paper. In all cases the strain rate was kept constant at 1.25 cm min^{-1} as shown by an indicator on the press.

Punch displacement v compression force plots.

These were obtained for preliminary compression, and also for a series of recompressions, of compacts formed from a smaller weight of fill. (Usually 20-30g of material.) This smaller weight was necessary since punch travel on preliminary compression was much greater than it was for recompression and would otherwise have been outside the linear range of the displacement transducer (Sangamo Type DL/125).

For these measurements the transducer bore on a plate fixed to the top (moving) platen of the press. The recompressions were performed using the XY plotter on a 10x more sensitive setting of the displacement (X) axis.

RESULTS AND DISCUSSION

Behaviour of free compacts on axial loading to fracture.

It is convenient to deal with this aspect initially since the results may be used to aid the interpretation of the other data.

The stress existing within a body may be completely defined at any point within it in terms of up to three principal stresses acting on three principal planes mutually at right angles to each other. By definition shear stresses are absent across these planes but may exist on other planes inclined to the principal planes [10,11]. Thus in the well known diametral crushing test on tablets failure may occur through the induced tensile stress, at right angles to the compressive force or by shear failure in a plane roughly at 45° to the compressive force as discussed by Fell and Newton[12]. In axial loading of compacts provided friction is absent, and the ends are completely covered by the platens, there is only one principal (compressive) stress. If the crushing strength is adequate, failure should eventually occur along shear planes inclined to the axis. Hiestand and Peot[13] have discussed the more complex forces involved in the transverse compression of square compacts with narrow platens.

The compacts we tested had a height/diameter ratio slightly greater than one and are similar in size and shape to those employed in the compressive testing of rock samples. Such compacts often fail along shear surfaces which generate two cones of failure[14] and this was the case for Emdex and Encompress, which failed at moderate loadings around 20 kN.

Avicel and Sta-Rx failed by spalling at higher loadings. Pillars of material broke off around the circumference but stopped short of the ends (fig. 2), and radial cracking could be demonstrated on cutting the compacts transversely. This type of failure was probably due to end restraint. Although the ends were lubricated, radial expansion would be impeded at higher loadings and 'barrelling' would occur around the mid point. This would create radial and circumferential stresses which together form the other two principal stresses responsible for radial cracking and spalling. It follows that these compacts were both resistant to shear stress.

Paracetamol DC failed by a brittle fracture which split the compacts axially indicating that the material has a low resistance to induced circumferential stress.

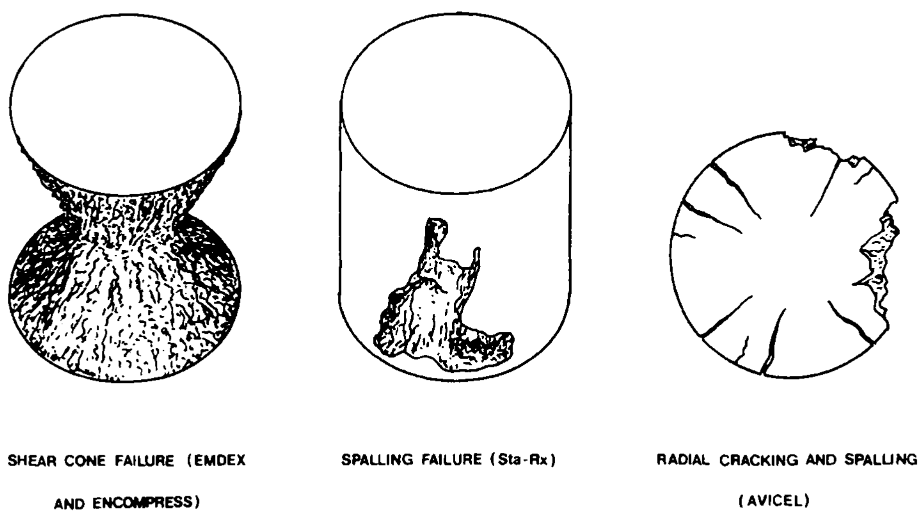

FIG. 2 Modes of failure of compacts

Force v Displacement Plots on Testing to Failure

Some typical plots are reproduced in fig. 3. None show Hookean behaviour but some were approximately linear until yielding began. The load then increased slowly and the slope of the plots became shallower.

If cone failure occurred then this last period was short but spalling was accompanied by a period of apparent plastic flow perhaps partly due to stress relief by internal cracking. Failure was progressive with an intact central portion still able to support the load when spalling was well advanced. This may be due to the fact that as radial and circumferential stresses decrease with decreased radius, the central region could withstand greater axial loading before failure. Paracetamol DC exhibited sudden failure with no evidence of prior yielding. This behaviour is characteristic of a brittle material.

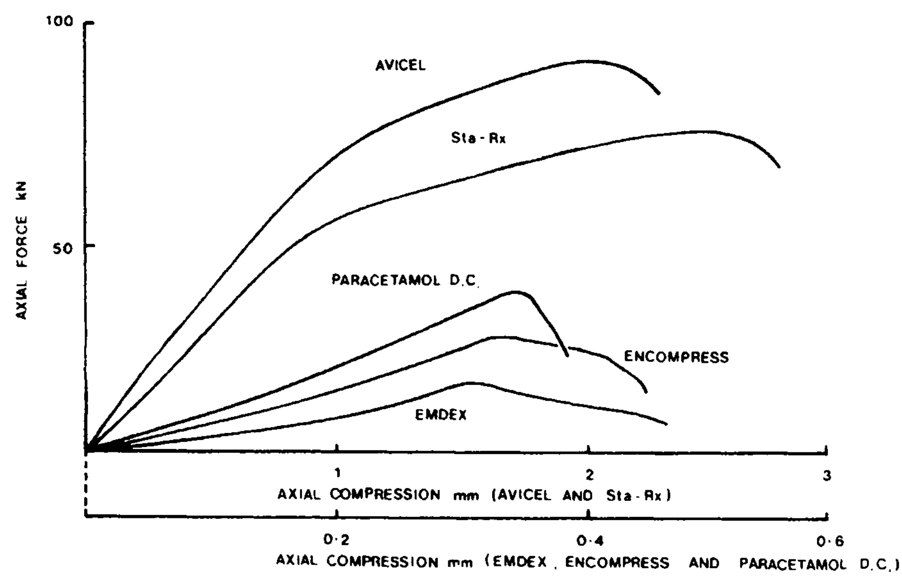

FIG. 3 Axial force vs. punch displacement plots on testing to failure in free compression

Axial and Radial displacement v Compression Force below failure stress.

These plots are sketched in fig. 4. All compacts except Emdex were elastic in that they gave closed hysteresis loops for both radial and axial strains but all were non linear. They are similar in form to those obtained by Travers, White and Lewis[9] for axial compression of compacts within the die and to those obtained by free axial compression of certain rock specimens[14]. Although the plots are non linear, an approximate value of Poisson's ratio can be calculated and values are listed in Table

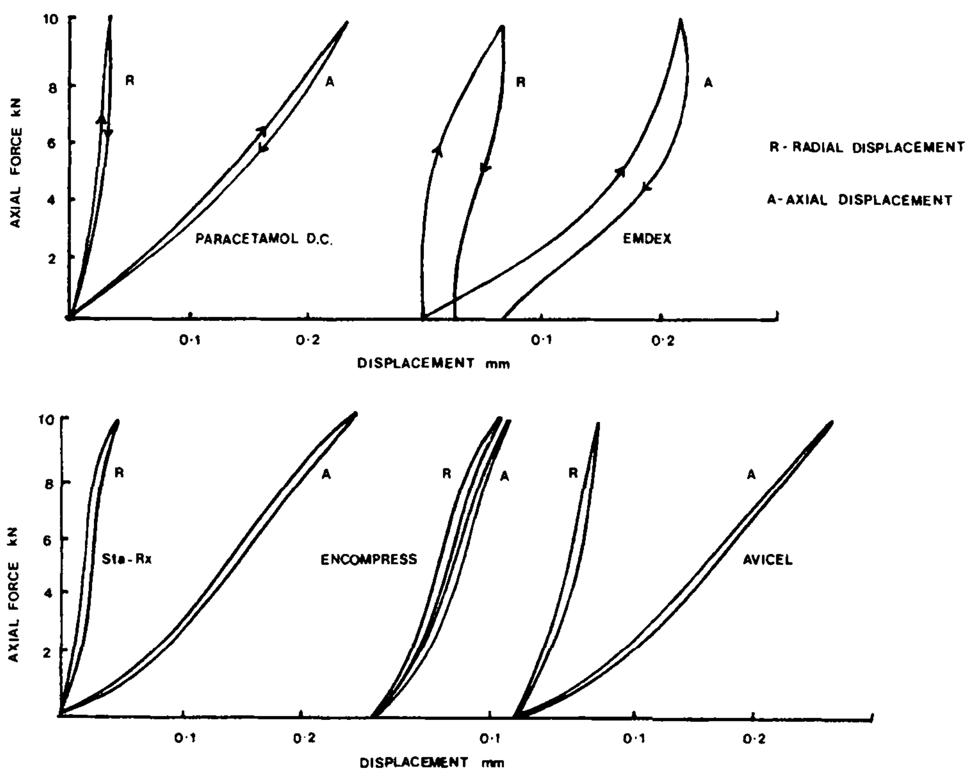

FIG. 4 Force vs. displacement plots (free compacts) below failure stress

Table 1.

Some Properties of the Compacts*

	Ejection Force required kN†	Axial Crushing Strength kN†	Apparent Poisson's Ratio
Encompress	75	22	1.42
Emdex	47	15	Not applicable
Avicel	3	90	0.36
Paracetamol DC	30	34	0.18
Sta-Rx	7	65	0.40

* Made at 200 kN axial force.

† Mean of three determinations.

1. A low value resulted for Paracetamol DC which is in accord with its brittle nature and values below the theoretical maximum of 0.5 were calculated for others. For Encompress, however, the apparent value was nearly three times the maximum, and there appears to be no obvious explanation for this result.

Emdex was interesting in that it flowed plastically at these low loads so that its radial and axial loops did not close even after several recompressions. The two loops were different in form with no apparent radial expansion until a minimum axial force was exceeded (fig. 4). This could be due to yielding along the plane of the shear cone until sufficient radial reaction was exerted to expand the side walls. This yielding appeared to have been partly plastic so that on relaxation some radial and axial strain remained. No meaningful value for Poisson's ratio could be deduced from these plots.

Radial Movement of Outer Die Wall on Compression and Recompression of the Compacts.

Several authors have published data on die wall pressure during tablet formation. Methods assume a linear relation ship between outer circumferential or radial strain as measured by strain gauges or piezoelectric load cells, and pressure at the inner die wall. Compressed rubber behaves hydrostatically so that gauges can be calibrated on the assumption of equal axial and radial stresses using this material in the die. Data on tablet materials can then be plotted as radial stress v axial stress where the radial stress is always less than the axial stress.

Fig. 5a shows similar plots obtained by our axial traverse method where compression force is plotted against radial movement of the outer die wall. The loops for preliminary compression were obtained at a point 1.25 cm above the base of the compact. All other loops were the result of recompressions after moving the transducer to the required position. Carless and Leigh,[15] Obiorah and Shotton[7], and other earlier authors[16,17,18], have commented on the residual stress which may remain after relaxation of axial stress. This may account for the apparent strains shown in fig. 5a but as some strain remains in the case of rubber where it should be absent, the strains may be artefacts due to zero drift. However, the residual strain for compacts on formation was always greater than for subsequent recompressions and was large for compacts such as Encompress which required a large force for ejection. The loops for rubber, Avicel and Sta-Rx on recompression, were nearly linear at all station. For Encompress there was an abrupt change of slope at the points indicated, with increased radial transmission thereafter. Other authors [7, 19], have reported similar effects on first compression and have interpreted them as due to the materials yielding in shear above a certain stress. Since these breaks also occur on recompression it suggests that Encompress yields along the shear cone with possible rebonding, and then retraces this movement when the axial force is relaxed. The changes on slope were more pronounced at the lower stations than they were for stations nearer the top of the compact. In the case of Emdex there was increased transmission at station 1 (compact base) but at higher stations the opposite occurred and a shallower slope signified reduced transmission. The changes in slope for Emdex took place at an axial force of 120 kN suggesting that the two effects were related.

On compression, a hydrostatic material such as rubber, cannot sustain shear stresses and therefore the plots are linear. All other materials are non hydrostatic and in these cases a difference stress (axial stress - radial stress) will produce shear stresses which will be less than those produced by a pure axial stress [10]. Conditions in a tablet die are therefore similar to those attained in tri-axial tests on soils [11] where the samples, though strengthened by a hydraulic radial thrust, can still fail in shear under sufficient axial loading.

Our plots for the recompression of Emdex and Encompress could be due to partial failure along the shear cone demonstrated in free compression. This would result in increased die wall reaction varying in intensity with the axial position. The upper and lower shear cones would be expected to produce greatest reaction at the top and bottom of the compact. Train [20], in his work on pressure distribution in compacts of similar size to ours, demonstrated that these were indeed regions of high pressure. He explained them by an argument based on Boussinesq's 'bulbs of pressure' theory [21] but slippage along shear planes would give a similar pattern.

Fig 5 b gives profiles obtained when maximum radial movement on recompression is plotted against axial height above the compact base. These profiles will not necessarily reflect the true stress distribution since the unstressed die above the compact restricts movement in this area to a greater extent than prevails in the shorter section below the compact. Nevertheless, movements at corresponding points were probably a good measure of the transmission by different compacts provided the axial lengths are kept constant.

Sta-Rx and Avicel, despite their low ejection force (Table 1), transmit about 60% of the axial pressure. This agrees with the figure given by Sixsmith [3], in a detailed study of the compression of various grades of Avicel. These compacts have profiles similar in shape to that of rubber. The other compacts have profiles with maxima occurring above the base of the compact indicating that their mode of transmission is dissimilar to the others. We suggest that Avicel and Sta-Rx exert their pressure mainly by the Poisson ratio effect with no yielding in shear whereas the others owe at least part of their transmission to such yielding. The 'self lubricating' properties of Avicel and Sta-Rx may be due to their elastic properties. With no shear yielding or failure, they regain their original dimensions when the axial force is removed and thus little or no 'locked in' stress remains. In simple tests involving measurement of the force required to move the compacts across the surface of the die, all, including Avicel and Sta-Rx, had a coefficient of sliding friction about 0.3. It is also interesting to note that whereas self-lubricating materials normally flow easily, the poor flowability of Avicel is one disadvantage to its use [6].

It is likely that the usefulness of Avicel in tabletting lies in its ability to confer shear resistance. That weakness in shear is undesirable was illustrated by the work of Jungerson [22] who found that the best meprobamate formulations were those yielding tablets failing in tension when subjected to diametral compression. A similar view has bean indirectly expressed by Obiorah and Shotton[7] who suggest that preferred formulations are those whose tablets produce the least residual die wall stress.

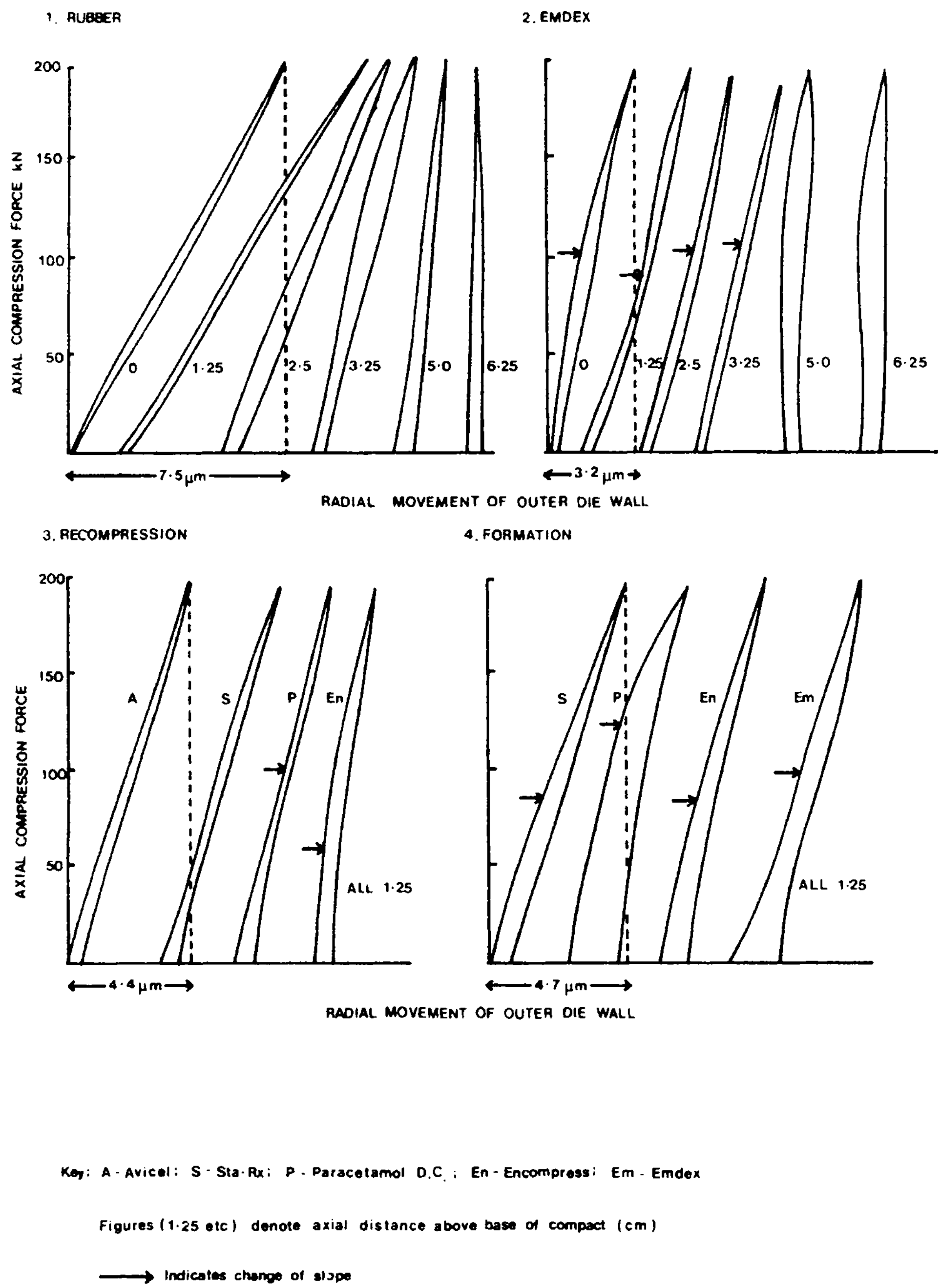

FIG. 5A Axial force vs. radial movement of outer die wall

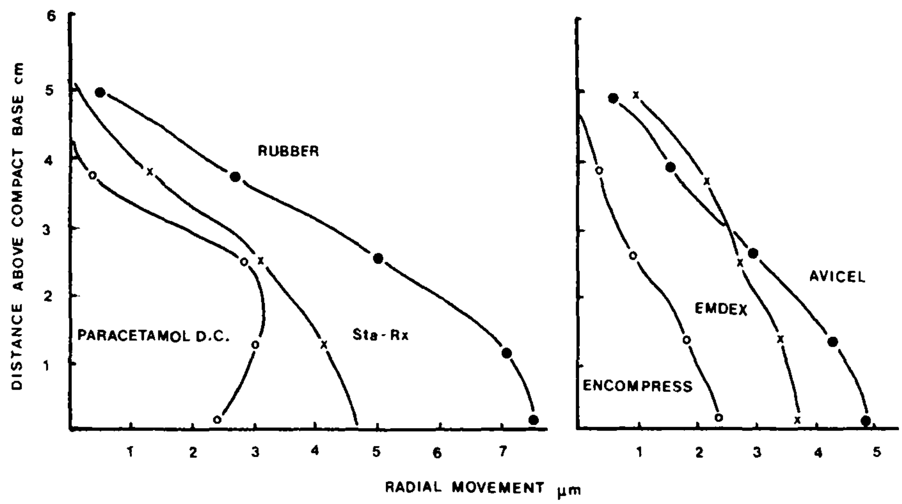

FIG. 5B Die wall radial movement at various axial positions

<u>Punch displacement v compression force plots.</u>

Some of these are reproduced in fig. 6. For first compression they are similar to those obtained by Gillard and others for bases which included Avicel and Encompress [2]. As these authors have discussed the interpretation of these loops in detail we shall pass on to consider the plots for recompression. These decrease in area with the number of recompressions and they represent energy dissipated as heat by the movement of dislocations within the compacts which do not entirely regain their original positions when the stress is removed.[9] Zener[23] states that materials showing such mechanical hysterisis must also possess a degree of viscoelasticity and creep. These properties were

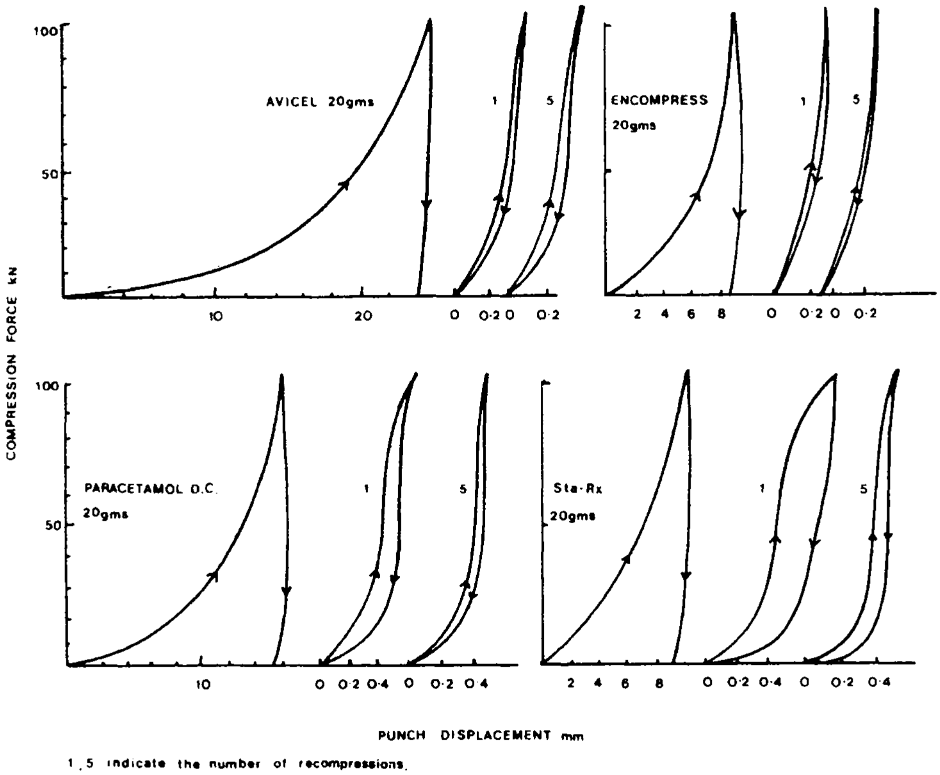

FIG. 6 Punch displacement vs. compression force plots on formation and recompression of the compact

studied by Aulton and Tebby[5] by long term indentation testing of tablets made from direct compression bases.

For all tablets, on application of a loaded indenter, there was a rapid elastic indentation followed by a much slower penetration due to viscoelastic/plastic flow. Emdex exhibited the greatest movement which is not surprising in view of its plasticity as demonstrated by the present work. Paracetamol did not show time-dependent indentation and again this is consistent with its brittle nature.

The hysteresis loops decrease in area with recompression. This is evidence of work hardening[24] which should modify the indentation behaviour of the compacts by decreasing the visco-elastic/plastic component. Work is currently in progress to investigate this point.

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