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THE USE OF AN ELASTIC RECOVERY INDEX AS A CRITERION OF COMPACTIONAL BEHAVIOUR OF SOME DIRECT COMPRESSION BASES

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ABSTRACT

The effects of compression force and holding time on computer logged strain movements (at constant stress) and elastic-viscoelastic expansions (on load release) of compacts made from eight pharmaceutical powders are reported. The feasibility of using an elastic recovery index (the ratio of elastic recovery to viscoelastic strain movements) to predict the quality of a material is also discussed.

All compacts continued to consolidate by viscoelastic and plastic flow when held under constant stress during the holding period. The "compressible" bases Avicel PH-101 and Sta-Rx 1500 exhibited more time dependent movements than powders such as Paracetamol DC, Paracetamol, Emcompress and magnesium stearate. Poorly compressible materials such as magnesium carbonate and paracetamol showed the greatest elastic expansions on load release.

It has been found that the elastic recovery indices of the compressible materials were lower than those of poorly compressible powders.

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INTRODUCTION

Several authors (1-4) have investigated the plastic and viscoelastic flow that occurs when a tablet is held under constant compression force. Some other techniques have also been developed to measure stress relaxation under constant strain conditions (5-8).

It has been pointed out (2-4) that on the release of compression force, the compact will expand within the die and may continue to do so after ejection. Armstrong and Haines-Nutt(9), Krycer and others(10) and several more authors (11-13) have used the percentage elastic recovery (E) defined by

$$E = 100 * (H - Hc)/Hc \qquad (1)$$

to measure the disruptive effects of elastic deformation on the compacts and to predict the capping tendencies of the materials. In the above equation Hc and H are the heights of the compacts under pressure and after ejection respectively. However Rue and others(14) using an acoustic emission technique showed that capping and lamination can occur during the decompression within the die. Travers and others(3) and Celik and others(4) employed a computer aided logging technique to prove that elastic expansion of their compacts took place in about 0.1s and this occured within the die immediately the load was released. After this short period compacts continued to expand viscoelastically.

The purpose of this study was to investigate the feasibility of using an Elastic Recovery Index (ERI) defined by

$$ERI = ER / SM \qquad (2)$$

where ER is the elastic expansion of the compact within the die on load release and SM is the viscoelastic strain movements under defined constant stress conditions. The effects of compression force and holding time (duration of maximum compression force) on the parameters ERI, ER and SM were also investigated.

MATERIALS AND METHODS

In this study, five direct compression bases Avicel PH-101 (a micro-crystalline cellulose), Sta-Rx 1500 (a modified starch),

Paracetamol DC (direct compression of paracetamol), Emdex (a maltose/dextrose mixture) and Emcompress (an excipient based on dicalcium phospate) and three poorly compressible materials (paracetamol, magnesium stearate and magnesium carbonate) have been used as received from the suppliers after being stored at 40-60 °C for 12-18 hours.

The Mayes Universal Hydraulic Testing Press employed for compaction studies and a 2.54 cm diameter punch-die set used in conjunction with the press have been fully described elsewhere (3,4).

Compression forces and punch movements were monitored by a load cell built into the press and an external short range displacement transducer (Sangamo WC, AC type) respectively. Analog signal voltages from the displacement transducer (amplified by a Sangamo Type C56 transducer meter) and load cell were simultaneously passed to a dual analog to digital logging convertor (ADC) connected to the user port of a CBM Model 4032 microcomputer (3,4).

The Compaction and Data Logging Process

Enough material (6.5-10.5g) to give a final predetermined compact thickness (12mm) was poured into the die set and the upper punch was inserted. All compacts were prepared from unlubricated materials throughout the study, but in the case of Emdex, Emcompress and magnesium carbonate the die was first lubricated by compressing a mixture of the material with 50% (w/w) magnesium stearate to prevent the material sticking to the die wall.

The die set and its holder were placed between the platens of the Mayes machine. It was necessary to set a small preload (about 1kN) prior to applying the compression force at a set loading rate. The final axial force was set at 45kN, 30kN or 15kN at a loading rate of 83kN/s on the control panel of the Mayes press.

An external transducer attached to a magnetic stand on the crosshead of the Mayes was set to register the punch movements when the compacts had been completely formed and were under constant stress.

The first and second channels of the dual ADC were selected to monitor compression force and punch displacement respectively. The machine code operating program (which was capable of logging up to 4048 items/s with three programmable variable speeds within the same run) was loaded into RAM (Random Access Memory) of the computer.

When all was ready, pressing the space bar of the computer simultaneously with the operating button on the Mayes Console set the compaction and logging process in motion.

When the maximum compression force was attained, the formed compact was maintained at constant stress for a predetermined period of up to 60s (usually 10s) by means of a servomechanism built into the machine which compensated for any viscoelastic movement of the compact. Constant stress was maintained during this period by a slow movement of the ram and punch which was monitored by the displacement transducer.

After a set holding period at constant stress, the load was released and the compact was ejected after a further 45s. During this period the rapid elastic and much slower viscoelastic expansions of the compact were also monitored by the transducer.

The data were transferred to a floppy disk under descriptive file names after being captured and stored in the RAM.

Several programs were written to recall these files, read the data into memory and print them in graphical form using a Commodore Tractor Printer (Type 3022). Further details have been given elsewhere (3,4,15).

Throughout the study four replicate compacts were prepared under identical conditions of compression force, loading rate, holding time and final compact thickness.

RESULTS AND DISCUSSION

In order to determine the point of maximum compression force, the concurrent values of compression force and punch movement were simultaneously recorded. It had been previously shown (4) that

the Mayes machine took longer to attain a set compression force when bases such as Avicel and Sta-Rx were in the die then was the case when less compressible materials such as Paracetamol DC and Emcompress were present.

The movements undergone by a formed compact under constant stress and on load release are shown schematically in Fig.1 which also indicates the changes in logging rates.

Strain Movements at Constant Stress

During the analysis of data, the compression force input reached a maximum and constant byte value and this point was taken as the starting point of the constant stress period (Point A in Fig.1). Strain movements vs time profiles of the compacts were then obtained using a plotting program from the data stored on the disk.

The strain movements of the compacts examined are shown in Fig.2and the effect of varying compression force and holding time on these movements are summarised in Table 1. The plastic and viscoelastic flow of Sta-Rx and Avicel which are known to be time dependent plastic bases (6) were greater than those of none time dependent bases such as Emdex, Paracetamol DC and Emcompress and poorly compressible materials (paracetamol, magnesium carbonate and magnesium stearate). The last three materials produced either very brittle or laminated compacts.

The rank order of the degree of further consolidation of the compacts at constant stress (under our experimental conditions as stated in Table 1) is Sta-Rx > Avicel > Emdex > Paracetamol DC > magnesium stearate > magnesium carbonate > paracetamol > Emcompress. This is further evidence that the magnitude of these movements can be used to predict the compressional behaviour of the materials (4).

Elastic Recovery on Load Release

We had previously shown that the Mayes machine was not able to release its load instantaneously but it was evident that the duration of decompression was about 100ms and the rate of load release was independent of the bases present in the die (3,4).

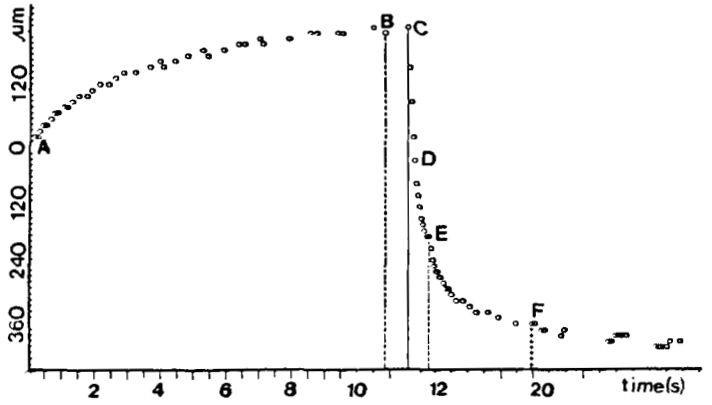

FIGURE 1
Strain Movements at Constant Stress(A-B), Elastic Recovery(C-D) and Viscoelastic Recovery(Point D onwards) on Load Release (Schematic). B,E and F are the points where the logging rate and/or time scale have been altered. Logging Speeds (items/s): 99(A-B) ; 990(B-E) ; 49(E onwards). (Vide reference 4).

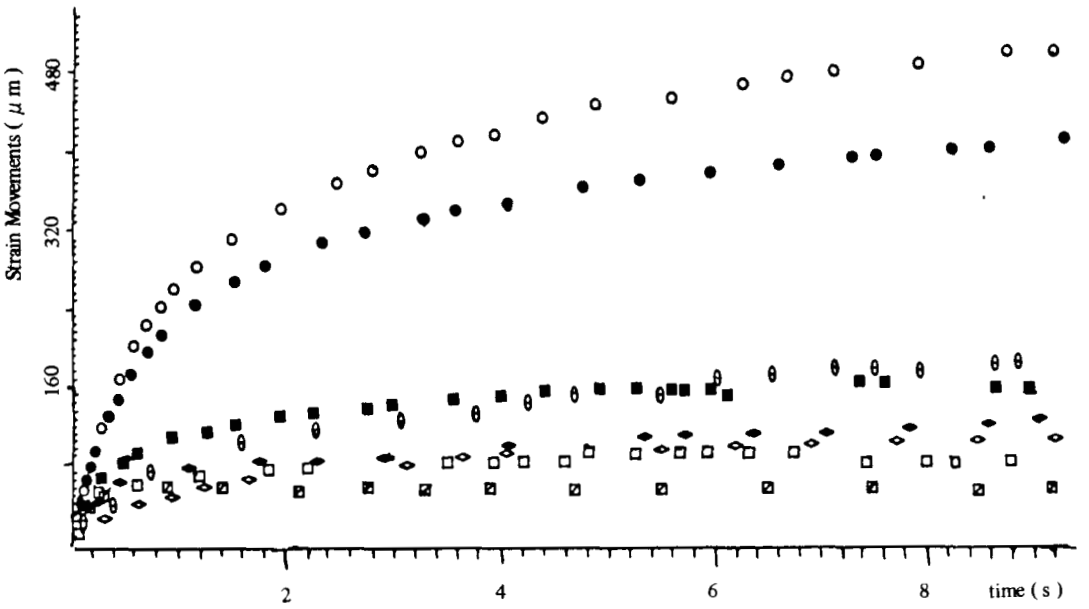

FIGURE 2
Strain Movements at Constant Stress. CF=30kN ; LR=83kN ; HT=10s
○,Sta-Rx 1500 ●,Avicel PH101 ☑,Emcompress
■,Paracetamol DC □,paracetamol θ,Emdex
◆,magnesium stearate ◇,magnesium carbonate

TABLE 1

Effect of Varying Compression Force and Holding Time on Strain Movements of The Compacts Under Constant Stress. CF=Compression Force(kN) R=Loading Rate(kN/s); HT=Holding Time(s); SM=Strain Movement(μm)

	LR=83kN/s ; HT= 10s			CF= 30kN ; LR=83kN/s					
	COMPRESSION FORCE (kN)			HOLDING TIME (s)					
	15	30	45	5	10	15	30	45	60
Sta-Rx 1500 (8g)	574	503	277	449	503	558	58	605	621
Avicel PH-101 (8g)	660	417	222	386	417	464	472	496	519
Emdex (8g)	199	185	355		185		222		
Emcompress (10.5g)	66	66	74		66		74		
Paracetamol DC (6.5g)	175	168	160		168		168		
Paracetamol (6.5g)		82			82				
Mg. stearate (8g)		121			121				
Mg. carbonate (7g)		105			105				

When the compression load was released after a predetermined holding time, all the compacts examined exhibited a rapid elastic expansion followed by a much slower viscoelastic recovery. Figures 3-7 show the 'expanded' elastic recovery plots of the punch alone on load release contrasted with the plots obtained when compacts are formed in the die. As can be seen from these figures, for a period of 15ms on load release, all the expansions could be attributed to machine characteristics and punch recovery and not to any property of the compacts themselves. After this period, though the machine continued to release its load, the compacts exhibited their expansions which could be calculated by subtracting the recovery of the punch from the total expansion within 100ms. These corrected values are summarised in Table 2.

The compacts of non time dependent bases and poorly compressible materials rapidly completed their plastic and viscoelastic movements within 3-4s and a prolonged holding time did not produce any increase or decrease in strain movements. In contrast the compacts prepared from Sta-Rx and Avicel exhibited further slower deformation over a long period under load. Rees and Rue(6) have reported that Sta-Rx is capable of extensive plastic deformation if sufficient time is allowed for this to occur.

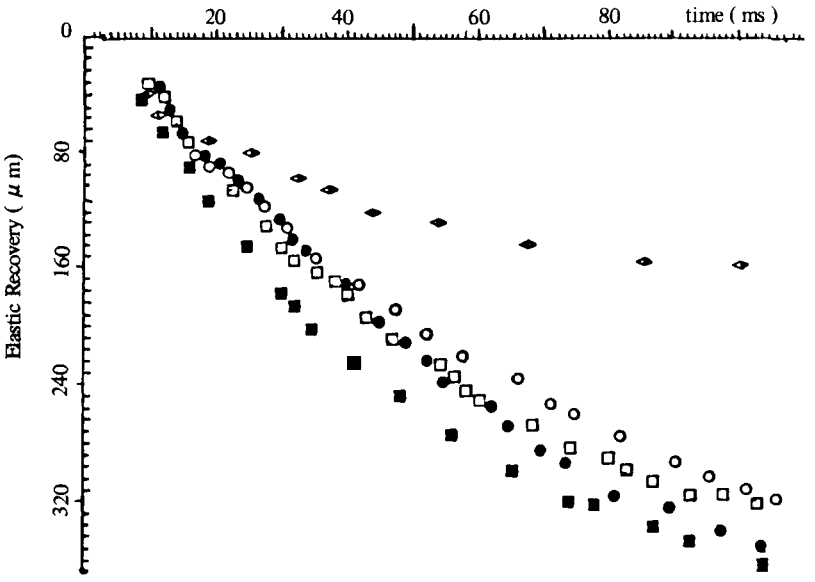

FIGURE 3-a

Elastic Recovery of The Compacts on Load Release. CF=30kN ; HT=10s ; LR=83kN/s. ◇,Blank Run (No powder in the die) ○,Sta-Rx 1500 ●,Avicel PH101 ■,Paracetamol DC □,paracetamol

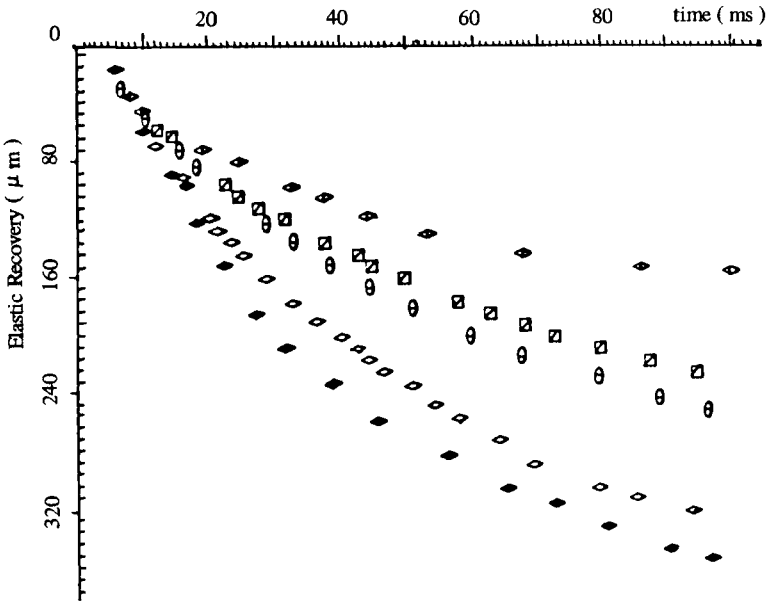

FIGURE 3-b

Elastic Recovery of The Compacts on Load Release. CF=30kN ; HT=10s ; LR=83kN/s ◇,Blank Run ▨,Emcompress θ ,Emdex ◆,magnesium stearate ◇,magnesium carbonate

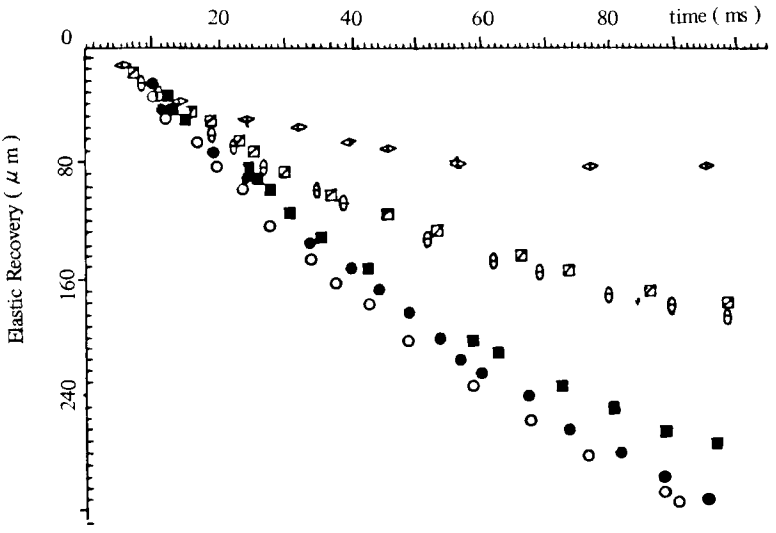

FIGURE 4
Elastic Recovery of The Compacts on Load Release. CF=15kN ;
HT=10s ; LR=83kN/s. ◈,Blank Run ○,Sta-Rx 1500
●,Avicel PH101 ■,Paracetamol DC θ,Emdex ⊠,Emcompress

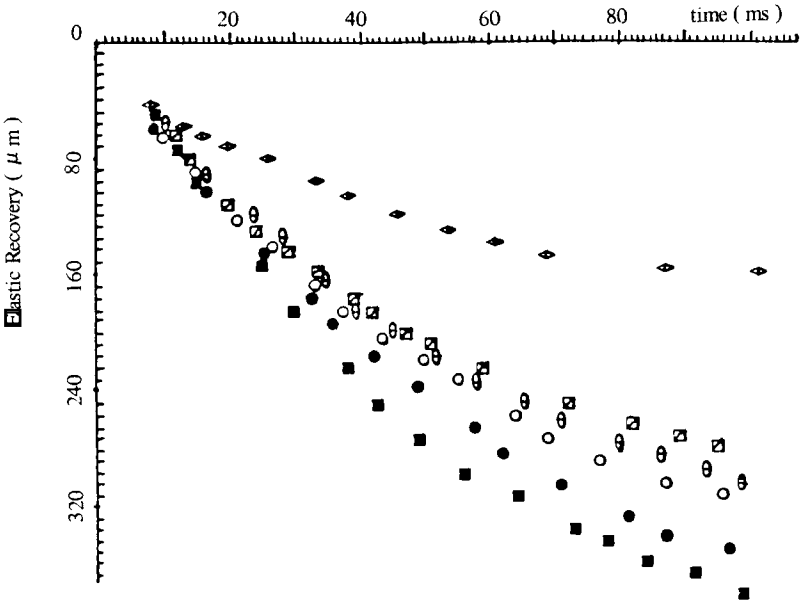

FIGURE 5
Elastic Recovery of The Compacts on Load Release. CF=45kN ;
HT=10s ; LR=83kN/s. ◈,Blank Run ○,Sta-Rx 1500
●,Avicel PH101 ■,Paracetamol DC θ,Emdex ⊠,Emcompress

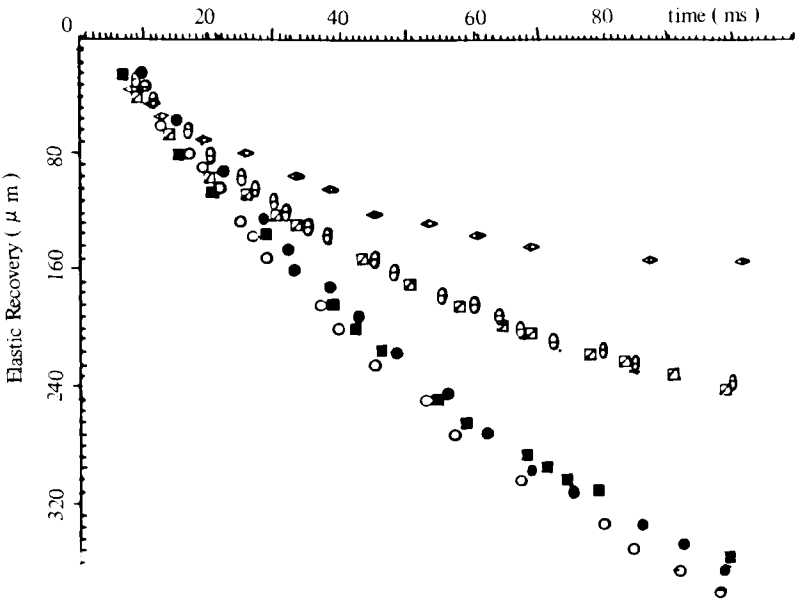

FIGURE 6
Elastic Recovery of The Compacts on Load Release. CF=30kN ; HT=0s ; LR=83kN/s. ◇,Blank Run ○,Sta-Rx 1500 ●,Avicel PH101 ■,Paracetamol DC θ,Emdex ⊠,Emcompress

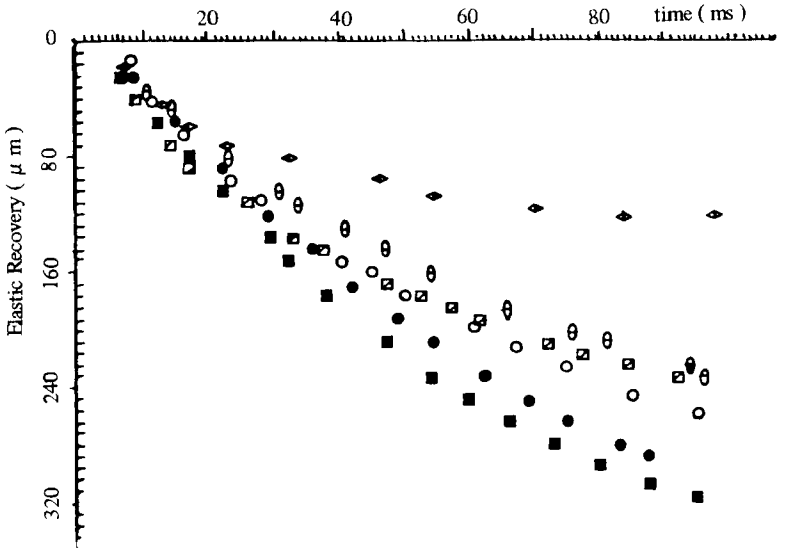

FIGURE 7
Elastic Recovery of The Compacts on Load Release. CF=30kN ; HT=30s ; LR=83kN/s. ◇,Blank Run ○,Sta-Rx 1500 ●,Avicel PH101 ■,Paracetamol DC θ,Emdex ⊠,Emcompress

TABLE 2
Effects of Varying Compression Force and Holding Time on Elastic Recovery of The Compacts on Load Release. ER=Corrected Elastic Recovery (μm)

	LR=83kN/s ; HT= 10s			CF= 30kN ; LR=83kN/s						
	COMPRESSION FORCE (kN)			HOLDING TIME (s)						
	15	30	45	0	5	10	15	30	45	60
	Corrected ER Values=Total Recovery-Punch Recovery(*)									
Sta-Rx 1500 (8g)	242	186	152	256	205	186	155	139	131	123
Avicel PH-101 (8g)	234	217	200	241	217	217	194	170	162	162
Emdex (8g)	100	130	140	108		108		92		
Emcompress (10.5g)	93	93	125	109		93		93		
Paracetamol DC (6.5g)	195	226	226	235		226		186		
Paracetamol (6.5g)		186				186				
Mg. stearate (8g)		225				225				
Mg. cabonate (7g)		194				194				
* Blank Run (No powder)	82	130	160	130	130	130	130	130	130	130

Varying the final compression force did not have any appreciable effect on the deformation of the compacts prepared from Emdex, Emcompress, Paracetamol DC. However if the compression force on Sta-Rx and Avicel was increased then their compacts exhibited considerably less plastic and viscoelastic flow during the holding time than was the case if a lower compression force was employed. The decrease in strain movements attributed to the effect of a higher compression force was greater for Avicel compacts than for Sta-Rx. Sixsmith(16) has reported that Avicel tablets become more plastic at high compaction pressures and our observations are in agreement with this view.

The large increase in strain movement when Emdex compacts are strongly compressed (45kN) is rather unexpected. It is possible that the material flows plastically into the voids at high pressures.

As shown in Figures 3a, 3b, 4 and 5, when the compression force was increased, the time dependent plastic bases Avicel and Sta-Rx exhibited less elastic expansion while Emdex, Paracetamol

DC and Emcompress showed a slightly increased recovery. A possible explanation is as follows. As the compression force increased, Avicel and Sta-Rx compacts become more plastic and produce stronger interparticulate bonding which may result in failure under recovery stresses. However, as mentioned earlier, the degree of plastic and viscoelastic flow of the compacts prepared from Emcompress and Paracetamol DC is not influenced by a higher compression force. The relatively greater elastic expansions of these compacts at high compression forces may be attributed to a lower degree of plastic flow and weaker interparticulate bonding.

The effect of prolonged holding time on the elastic recovery of the compacts can be deduced from the figures 3a, 3b, 6 and 7 and Table 2. If the holding time was set as low as possible (<1s) then the elastic expansions of the compacts was always greater than was the case if this period was prolonged up to 60s. The decrease in elastic expansion with increased holding time may be attributed to further consolidation of the compacts under constant stress which allows some conversion of elastic energy into plastic and viscoelastic movement and which usually will result in better tablets.

Milosovich(17) pointed out that capping was due to the expansion of elastically deformed particles and subsequent rupture of interparticulate bonds. More recently Rue and others(14) showed that capping and lamination can only occur during the decompression within the die. Ritter and Sucker have also formed the opinion that rate of decompression is an important factor (18). Our results have shown that elastic recovery of the compacts occured in about 100ms which was also the duration of decompression. After this period, the compacts continued to expand viscoelasticaly but these slow movements are less likely to cause lamination than those due to elastic recovery (Table 3).

Elastic Recovery Indices of The Compacts

Numerous techniques have been employed to quantify the expansion of formed compacts and some of these were recently

TABLE 3

Effects of Varying Compression Force and Holding Time on Viscoelastic Recovery of The Compacts on Load Release (for 45s).

	LR=83kN/s ; HT= 10s			CF= 30kN ; LR=83kN/s						
	COMPRESSION FORCE (kN)			HOLDING TIME (s)						
	15	30	45	0	5	10	15	30	45	60
	Viscoelastic Recovery (μm)									
Sta-Rx 1500 (8g)	606	511	462	605	543	511	496	433	394	378
Avicel PH-101 (8g)	453	324	290	339	332	324	316	308	308	300
Emdex (8g)	101	101	121	78		101		105		
Emcompress (10.5g)	46	43	58	43		43		50		
Paracetamol DC(6.5g)	121	113	121	105		113		113		
Paracetamol (6.5g)		74				74				
Mg. stearate (8g)		152				152				
Mg. carbonate (7g)		101				101				

evaluated by Crycer and others(19). The most widely used criterion is the percentage elastic recovery (E) as defined by equation (1) previously quoted. The expansion of compacts has been measured by different authors at varying times after ejection and so may include slower viscoelastic recovery as well as the rapid expansion. Therefore the equation (1) can not be a direct measurement of the elastic expansion of the tablets which can only occur within the die and during decompression.

In this study we propose an alternative technique which may be used to predict the compressional behaviour of the materials and to measure the disruptive effects of elastic expansion. This parameter is the Elastic Recovery Index (ERI) defined by

$$ERI = ER/SM \qquad (2)$$

where ER is elastic recovery of the compact on load release (corrected for punch recovery) and SM is the strain movement under a constant load. ERI values of the compacts examined are given in Table 4.

TABLE 4
Elastic Recovery Indices (ERI) of The Compacts Examined.

	LR=83kN/s ; HT= 10s			CF= 30kN ; LR=83kN/s					
	COMPRESSION FORCE (kN)			HOLDING TIME (s)					
	15	30	45	5	10	15	30	45	60
Sta-Rx 1500	0.422	0.369	0.548	0.456	0.369	0.278	0.239	0.217	0.198
Avicel PH101	0.355	0.520	0.630	0.562	0.520	0.418	0.360	0.326	0.312
Emdex (8g)	0.502	0.702	0.394		0.702		0.414		
Emcompress	2.657	1.410	1.950		1.410		1.257		
P.cetamol DC	2.190	1.345	1.413		1.345		1.110		
Paracetamol		2.260			2.260				
Mg. stearate		1.850			1.850				
Mg. carbonate		1.847			1.847				

The rank order for the ERI of the compacts examined (CF=39kN ; HT=10s ; LR=83kN/s) is Sta-Rx 1500 (0.369) < Avicel PH-101 (0.520) < Emdex (0.720) < Paracetamol DC (1.345) < Emcompress (1.410) < magnesium carbonate (1.847) <= magnesium stearate (1.850) < paracetamol (2.260).

We can therefore suggest that if a material has a low ERI value then it may produce a good tablet. Sta-Rx and Avicel which are known to be compressible bases exhibited the lowest ERI values. Magnesium carbonate, magnesium stearate and paracetamol, as mentioned earlier, produced either very brittle or capped tablets. We would predict this from their high ERI values which were greater than 1.500 suggesting that if a material has an ERI value more than this then that will result in a weak, possibly capped tablet under our experimental conditions as stated above.

An interesting feature of the ERI values was their dependency on holding time. The ERI value of the compacts was always smaller at a prolonged holding time than was the case if this period was as low as possible (<1s). A high 'dwell time' in manufacturing practice is well known to favour the successful tabletting of difficult materials.

One of the factors causing some tabletting problems such as capping is the use of too high a compression force (18). We would predict this from our ERI results which were higher for a given material compressed at 45KN than those obtained at lower final compression forces.

CONCLUSION

The conclusions that may be drawn from the work described are as follows.

a) An Elastic Recovery Index (ERI) defined by

$$ERI = ER/SM$$

where ER is elastic recovery of the compact on load release (corrected for punch recovery) and SM is the strain movement under a constant load, can be employed to predict the compressional behaviour of a material.

b) If a material has a low ERI value then it forms satisfactory tablets.

c) If the ERI value of a material is higher than 1.5 (under the experimental conditions cited) then it forms a poor and brittle compact prone to lamination.

d) The ERI value of a material is decreased by a prolonged holding time and increased by a high final compression force.

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