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HECKEL PLOTS AS INDICATORS OF ELASTIC PROPERTIES OF PHARMACEUTICALS

Petteri Paronen
Department of Pharmaceutical Technology
University of Kuopio, POB 6, SF-70211 Kuopio, Finland

ABSTRACT

In this study the usefulness of different kind of Heckel treatments to describe the elastic properties of the compressed materials was evaluated. Two methods were used to find the values for Heckel function, namely, tablet-in-die method and ejected tablets method. The slope values were calculated by means of the first derivative of the Heckel function. The reliable conclusions from the total elastic recovery were possible to obtain using the reciprocal of the difference of the Heckel upward plot slopes obtained from the two methods used in this study. The downward part of the Heckel plot was noticed to be useful for describing the fast elasticity during the tableting process.

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INTRODUCTION

The Heckel equation is a widely used method for obtaining information from the compression properties of the pharmaceuticals. The mathematical form of this equation is (1,2)

$$\ln \left(\frac{1}{1-D}\right) = kP + A \qquad (1)$$

where D is the packing fraction of the tablet, or the apparent density of the tablet divided by the effective particle density. P is the applied pressure. The constants k and A are determined from the slope and intercept, respectively, of the extrapolated linear portion of the plot of ln (1/(1-D)) vs. P.

Two different methods have been used for obtaining data into the Heckel treatment. Firstly, tablet-in-die-method, in which the applied pressure and the packing fraction of the powder column are determined at several points during one compression process. Most often only the upward part of the Heckel plots has been used for conclusions. Thus the values obtained after the maximum compression phase are not used for calculations. Duberg and Nyström (3) have, however, supposed that downward part of the Heckel plot could be useful for describing the elastic recovery of the materials during the compression process. In the second method, ejected tablets method, the maximum upper punch pressures and the packing fractions determined by measuring the dimensions of tablets after the ejection of the tablet from the die, are used for conclusions. Different kind of data is obtained using different methods. Paronen and Juslin (4) have pointed out that useful conclusions from the deformation properties of the compared materials are possible to make using both of these methods at the same study.

The aim of this study was to evaluate the usefulness of different kind of Heckel treatments to describe especially the elastic properties of the compressed materials.

MATERIALS AND METHODS

Materials

The materials studied were; microcrystalline cellulose, Avicel PH 101 (FMC Corp. Philadelphia, USA), dicalcium phosphate dihydrate, Parmcompress (Firma Parmantier, West-Germany), modified starch, Starch 1500 (Colorcon Ltd., UK), sodium chloride (Merck, West-Germany). These materials were chosen because of the well-known differences in their deformation properties. All the materials, besides dicalcium phosphate, are plastically deforming. Dicalcium phosphate is fragmenting during the compression process. Microcrystalline cellulose and especially modified starch are rather much elastically deforming materials otherwise than dicalcium phosphate and sodium chloride, which deforms elastically only a little.

Tableting

The tablet weight used was the amount of powder that would create a tablet with zero porosity, height of 2.00 mm and diameter of 12 mm. Tablets were compressed using an instrumented Hanseaten Exakta single punch machine. The upper and lower punches of the press are equipped with strain gauge load washers and the upper punch with the inductive displacement transducer mechanically connected also to the lower punch. The samples were compressed at a speed of 35 tablets/min.

Tableting parameter

Two methods were used to find the tableting parameters. Firstly, the applied pressure and the packing fraction of a powder column were determined at about 220 points during the compression phase. The data from ten tablets compressed at the maximum pressure of 170 MPa were used for conclusions by this tablet-in-die-method. Secondly packing fractions were determined by measuring the dimensions of the tablets with a micrometer screw about 24 h after the ejection of the tablet. The data from tablets compressed using 22 different maximum upper punch pressures were used for conclusions by this ejected tablet method.

The lowest first derivative values of the Heckel function determined separately from the upward and downward parts of the plot were used as slope values to decribe the densification and recovery stages for compression, respectively. This kind of calculation method was chosen because the Heckel plots obtained were more or less unlinear. Thus the least square method usually utilized was noticed to be unsatisfactory method to obtain accurate slope values.

RESULTS AND DISCUSSION

The stage of densification obtained during the compression process by the compressional pressure of about 170 MPa decreaces at the order; modified starch, microcrystalline cellulose, sodium chloride and dicalcium phosphate dihydrate (see Fig. 1). The tendency of the materials to the total deformation containing both the plastic and elastic deformation is shown by the reciprocal of the Heckel plot slope obtained using the tablet-in-die method (K_d values in Table 1). The order of the materials according to this parameter is the same as the above mentioned densification order. The tendenxy of the materials to the plastic deformation is shown by the reciprocal of the Heckel plot slope obtained using the ejected tablet method (K_p values in Table 1).

The order of the materials according to this permanent densification tendency is the following: sodium chloride, microcrystalline cellulose, modified starch and dicalcium phosphate dihydrate. The difference between the orders of the materials obtained by these two methods indicates great differences in the tendencies to elastic recovery.

Paronen and Juslin (4) have shown that it is possible to use the reciprocal of the difference of the Heckel upward plot slopes obtained from the above mentioned two methods as a parameter that describes the tendency of a material to deform elastically (K_{et} values in Table 1). These values describe the total elastic deformation of the materials thus containing both the fast recovery during the compression process and slow recovery after the

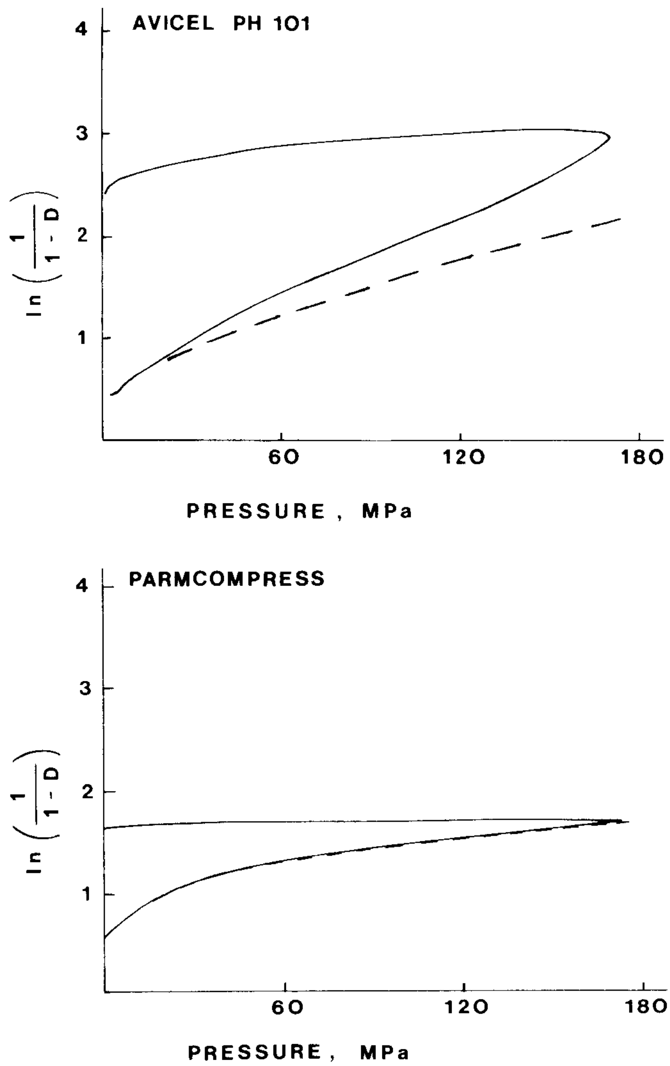

FIGURE 1

The Heckel plots obtained using tablet-in-die method (solid lines) and ejected tablets method (broken lines).

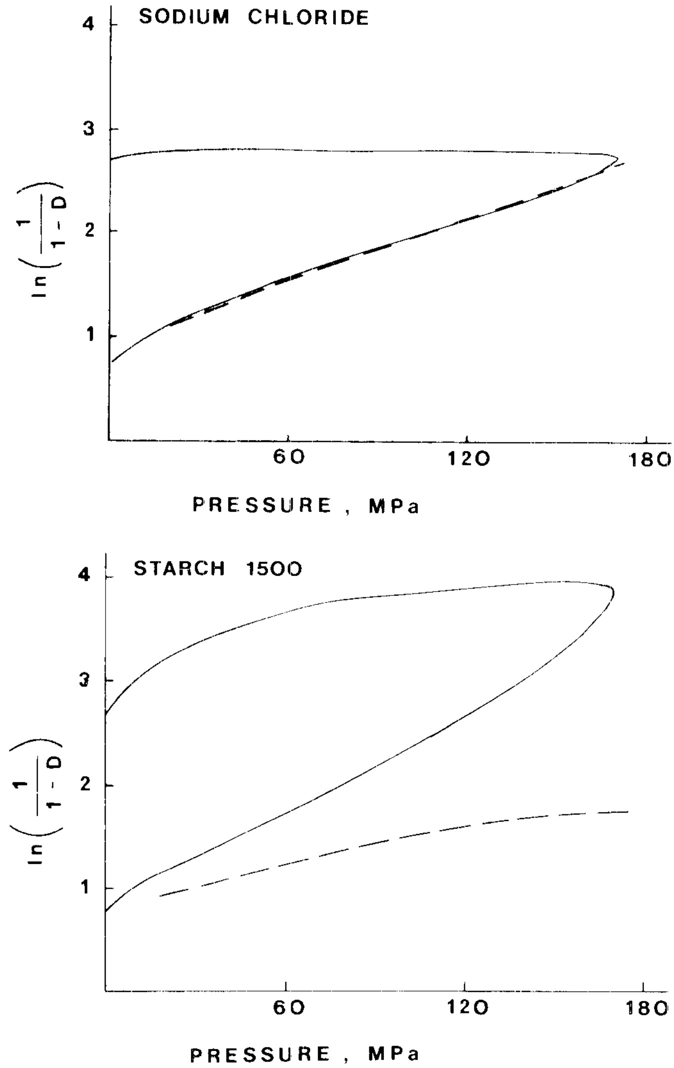

FIGURE 1 Continued.

TABLE 1

The reciprocal of the slope values obtained from the Heckel plots using different methods

Material	Tablet-in-die method		Ejected tablets method	Combination method
	K_d	K_{ef}	K_p	K_{et}
Avicel PH 101	104(3.1 %)	799(5.3 %)	163	287
Parmcompress	252(2.1 %)	6930(6.2 %)	272	3377
Sodium chloride	125(4.4 %)	16197(16.2%)	129	4031
Starch 1500	78(3.9 %)	237 (8.2 %)	216	122

K_d = yield pressure of total deformation
K_{ef} = yield pressure of fast elastic deformation
K_p = yield pressure of plastic deformation
K_{et} = yield pressure of total elastic deformation

compression process in the die and also after the ejection of the tablet from the die. According to this parameter dicalcium phosphate dihydrate and especially sodium chloride are clearly less prone to this total elastic recovery than microcrystalline cellulose and modified starch are.

Duberg and Nyström (3) have supposed that the downward part of the Heckel plots would be possible to use for describing the fast elasticity of the materials during the compression process. In this study the reciprocal of the slopes obtained from the downward part of the Heckel plots were calculated (K_{ef} values in Table 1). The deviation of these values between different measurements is some larger than the deviation of other parameter values used in this study. The numerical values of this parameter differentiate from each other so clearly that it is still possible to make reliable conclusions using this parameter. Modified starch seems to be especially prone to rapid elastic recovery during the compression process. The elasticity of sodium chloride seems to be only trivial at this compression phase.

CONCLUSIONS

According to the results of this study it is well possible to make reliable conclusions from the elastic properties of the pharmaceutical materials using the Heckel treatment. More information is gained using different methods at the same study for obtaining data to the Heckel treatment. The numerical parameter value describing the total elastic recovery is possible to be calculated using the data obtained from the ejected tablets method and from the tablet-in-die method. The downward part of the Heckel plot is especially useful for describing the fast elasticity during the tableting process.

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