J. Pharm. Pharmac., 1978, **30**, 642

COMMUNICATIONS

Limitations of the Heckel relation for predicting powder compaction mechanisms

P. J. RUE*, J. E. REES†, *Department of Pharmacy, University of Aston in Birmingham, Gosta Green, Birmingham, B4 7ET, U.K.*

Recently York (1977) used the Heckel (1961) equation to interpret the consolidation of powders during compaction at low pressures. He states that this equation may be used to elucidate compression behaviour at higher pressures and that such behaviour may be divided into three types, termed A and B (Type 1 and 2 of Hersey & Rees, 1971) and C (York & Pilpel, 1973) depending on the effect of particle size on the Heckel plots.

We now believe that caution is necessary in attempts to classify the compaction behaviour of materials on the basis of changes in the Heckel plots with particle size. As explained below, there is considerable evidence that the type of Heckel plots obtained for many materials will vary depending on the experimental compaction technique used. Furthermore, since different particle size fractions of the same material may exhibit changes in the predominant compaction mechanism ranging from brittle to plastic deformation (Gregory, 1962), we are reluctant to classify a material using data for a range of particle sizes.

Hersey & Rees (1970) using the compaction data of Hersey, Bayraktar & Shotton (1967) found that, in a 12 mm diameter die, the Heckel plots for various size fractions of sodium chloride were non-linear up to an applied compaction pressure of 50 MN m^{-2} and showed a separation of the plots at all pressure levels. York (1977) suggests that this is Type A behaviour, characteristic of a material which consolidates mainly by plastic deformation. In later experiments using a 33 mm die and various size fractions of sodium chloride, Hersey, Cole & Rees (1972) obtained completely linear Heckel plots up to an applied compaction pressure of 60 MN m^{-2} and no separation of the plots was found.

Using the data of Alpar, Hersey & Shotton (1970), Hersey & Rees (1970) investigated the effect of lactose particle size on the Heckel plots. The amount of powder compacted was sufficient to produce a 4 mm thick compact at zero porosity in a 12 mm die and the volume of the compact was determined after ejection. Under these conditions, above an applied pressure of 50 MN m^{-2} the Heckel plots were linear and identical for the four size fractions studied. York (1977) describes this as Type B behaviour, characteristic of a material which consolidates by fragmentation. However, Hersey & others (1972) later studied the compaction behaviour of various size fractions of lactose in a 33 mm die using equal volumes of bulk material. In that study the volume of the compact was measured under load by monitoring the movement of the upper punch into the die. Under these conditions the Heckel relation did not produce identical plots for the different size fractions. Evaluation of the slopes of the linear sections of the plots showed that, as the mean particle size decreased, there was a tendency for the yield strength of the particles to increase. This increase was attributed to the lower probability of cracks being present in smaller particles.

Fell & Newton (1971) found that, with crystalline lactose, different Heckel plots were obtained for different rates of compaction and depended on whether the volume of the tablets was measured under load or after ejection from the die. They concluded that volume measurements recorded with the compact under load include an elastic component which increases the value of ln 1/(1-D) especially at higher compaction forces; this gives a false low value of the yield strength.

We recently obtained Heckel plots for a granular form of microfine cellulose (Elcema G250, supplied

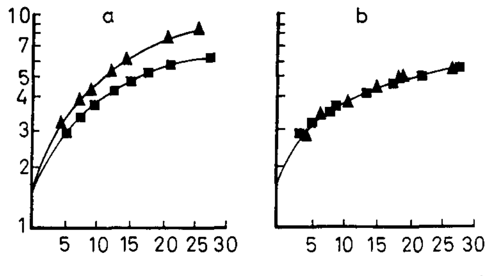

FIG. 1. Heckel plots showing the effect of contact-time on the consolidation behaviour of (a) Elcema and (b) Emcompress. Contact times: ■, 0·17s; ▲, 10 s. Relative density, D is calculated by dividing the density of the tablet by the density of the particulate solid determined using helium pycnometry. Compacts were prepared in a 12·7 mm diameter die; a compaction force of 10 kN on the abscissa is therefore equivalent to 79 MN m^{-2} compaction pressure. Ordinate: 1/(1–D) (log scale). Abscissa: Compaction force (kN).

---

* Present address Beecham Pharmaceuticals, Burgh Heath, Epsom, Surrey.

† Correspondence; Present address: I.D.D.C. Abbott Laboratories, Queenborough, Kent.

by Degussa Ltd) which do not conform to Type A, B or C profiles. As shown in Fig. 1(a), the Heckel plots exhibit a continual decrease in slope. Since there is no linear section, it is not possible to determine a value for the yield pressure of Elcema from these results. Bockstiegel (1972) criticized compaction equations, such as the Heckel relation, suggesting that they are of little practical value since they only fit the experimental data over a limited range owing to the large number of parameters which affect the compaction process but which are not represented in the equations. Certainly our data for Elcema do not appear to conform to the Heckel relation. Furthermore, Bockstiegel (1972) considers that compaction experiments are easy to perform and are more reliable than theoretical predictions.

The two Heckel plots for Elcema shown in Fig. 1(a) were obtained at two different values of contact time, which is defined by Jones (1977) as the time during which the upper punch exerts a force on the die contents. The increased consolidation with increased contact time is related to the deformation behaviour of the material; a large increase, such as that seen for Elcema, indicates a plastically deforming material whereas little or no increase in consolidation, such as seen in Fig. 1(b) for Emcompress, is characteristic of a brittle material. Although, as explained, the Heckel plot for a material may vary depending on the compaction technique employed, it appears possible to quantify the amount of plastic deformation during compaction by measuring the areas under Heckel plots obtained at several contact times. Since this technique is quantitative, it could also provide a basis for comparing the plastic deformation of a range of materials. We suggest that this would be more useful than merely classifying materials into distinct types, especially since the collective evidence presented in this communication indicates that the use of Heckel profiles for the latter purpose would appear to be unreliable.

April 10, 1978

REFERENCES

ALPAR, O., HERSEY, J. A. & SHOTTON, E. (1970). *J. Pharm. Pharmac.*, **22**, *Suppl.*, 1S–7S.

BOCKSTIEGEL, G. (1972). In: Proceedings of the First International Conference on the Compaction and Consolidation of Particulate Matter. Brighton, October 1972. Editor: Goldberg, A. S. London, 1973: Powder Advisory Centre.

FELL, J. T. & NEWTON, J. M. (1971). *J. pharm. Sci.*, **60**, 1866–1869.

GREGORY, H. R. (1962). *Trans Instn. chem. Engrs.*, **40**, 241.

HECKEL, R. W. (1961). *Trans metall. Soc. A.I.M.E.*, **221**, 671–675.

HERSEY, J. A., BAYRAKTAR, G. & SHOTTON, E. (1967). *J. Pharm. Pharmac.*, **19**, 24S–30S.

HERSEY, J. A., COLE, E. T. & REES, J. E. (1972). In: Proceedings of the First International Conference on the Compaction and Consolidation of Particulate Matter. Brighton, October 1972. Editor. Goldberg, A. S. London, 1973: Powder Advisory Centre.

HERSEY, J. A. & REES, J. E. (1970). Second Particle Size Analysis Conference, Society for Analytical Chemistry Bradford 1970. Proceedings S.A.C. 1972.

HERSEY, J. A. & REES, J. E. (1971). *Nature, Phys. Sci.*, **230**, 96.

JONES, T. M. (1977). In: *Formulation and Preparation of Dosage Forms.* p. 29–44. Editor: Polderman, J. North-Holland: Elsevier.

YORK, P. & PILPEL, N. (1973). *J. Pharm. Pharmac.*, **25**, *Suppl.*, 1P–11P.

YORK, P. (1977). *Ibid.*, **30**, 6–10.