Working with Heckel Plots: Method for Checking the Plausibility of Force Displacement Data

K. Duchatsch[a], J. Falconet[b], G. Tardy[b], R.F. Lammens[a], B. Fretter[a]

[a] Department of Pharmaceutical Technology, University of Bonn, Gerhard-Domagk-Str. 3, 53121 Bonn, Germany
[b] Medelpharm, 43, Porte du grand Lyon, 01700 Neyron, France

Introduction:

For characterising powder compression behaviour and deformation mechanisms of materials often the Heckel Plot is used. It describes the volume reduction of a powder bed during compression by plotting ln(1/) versus pressure. Hereby, the sections of the Heckel Plot describing the loading phase represent the deformation behaviour of the powder. The value of the slope is very sensitive to small errors in the in-die density determination which during tableting, are mainly due to lack of accuracy of the displacement transducers, the reference positions and punch tilting etc.[1] To minimise these errors, an accurate external height-measurement system was established at the compaction simulator Stylcam 200R. For characterising powder under conditions close to production, the Stylcam 200R was chosen because of its ability to simulate tableting speeds like the ones prevailing on various production tablet presses. Furthermore, it was also equipped with a pair of concave punches. This leads to the advantage that capping and laminating might be detected early in development. Because Heckel Plots strongly depending on the measurement accuracy, a plausibility check of the acquired data was developed.

Materials & Methods:

Materials

- Compaction simulator Stylcam 200R (Medelpharm, France) simulating different rotary presses at various speeds, instrumented with load cells and internal inductive displacement transducers (Fig. 1+2), data acquisition and analysing software: Analis (Medelpharm, France)
- punches: 9 mm concave R15, 10 mm flat, 11.28 mm flat, all EuB
- external displacement measurement system (Fig. 3): 4 inductive displacement transducers, amplifier, (all Schreiber, Germany), overall accuracy 10 µm[2], data acquisition software: DAQ4 (Hucke Software, Germany), the external system can be clamped to any B-type punch (Eu or TSM)
- reference tablets: dome-shaped metal tablet for flat punches; highly densified Avicel tablet with equal cup radius as the concave punches to be used

Methods

Determining reference positions:
Often, the top edge of the die is taken as the reference point although the absolute position of upper and lower punch is irrelevant for the in-die tablet height measurement. Therefore, only the distance between the punches is determined by applying small forces (ca. 100 N) on a reference tablet, three times before and after a set of experiments.

Fig.1: The compaction simulator Stylcam 200R

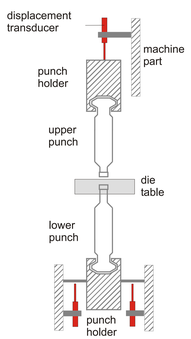

Fig.2: measurement setup of the internal displacement transducers

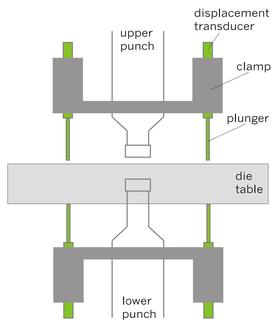

Fig.3: measurement setup of the external displacement transducers

Measuring reference tablet height:
Before and after establishing the reference positions, the height of the reference tablet (metal as well as Avicel) is measured with a linear gage having an accuracy of 3 µm.

Considering the elastic deformation:
To calculate the exact tablet height under pressure the elastic deformation of punches and machine parts must be taken in account. For the external displacement measurement system, the deformation between punch tip and mounting clamp of the transducers is calculated from Hooke's law and punch geometry.[3] For the internal displacement measurement system, a calculation is not possible and therefore, the deformation has to be determined experimentally.

Results:

Reproducibility of reference data

Measuring the Avicel reference tablet height turn out to be within an accuracy of 5 µm which is little larger than the accuracy of 3 µm of the linear gage. The reasons therefore are the difficulty of positioning a concave tablet exactly vertically under the contact tip of the linear gage without tilting as well as the roughness of the tablet surface.
The reproducibility of repeatedly determining the reference positions for the in-die powder bed height measurement without removing or changing the position of the reference tablet turn out to be within 5 µm. Establishing reference positions before and after a series of experiments is reproducible within 10 µm.

Method for Checking the Plausibility

When working with Heckel Plots, small errors can have a large influence on the results. An easy and fast method for checking the plausibility of displacement data is examining the curve progression of Heckel Plots at different maximum compaction pressures. Those sections of the Heckel Plot describing the loading phase should coincide because at the same powder density the pressure should prevail as long as the influence of tableting speed is moderate. Only near the reversal point of the punch movement, the curves start to deviate due to relaxation because of low densification speeds. Fig. 4 shows an example of non coinciding curves due to a change of the mounting positions of the external displacement transducer clamps which results in different reference positions before and after the experiment. This error would not be recognised when examining just one Heckel Plot.
In Fig. 5 a consistent set of Heckel Plots is shown with congruent sections describing the loading phase. These data are obtained with the concave punches under conditions close to production concerning the velocity of the simulated tablet press (effective dwell time of 7.5 ms).
But consistent sets of Heckel Plots are only indicating correct measurement data. Also the shape of the Heckel curves must be considered.

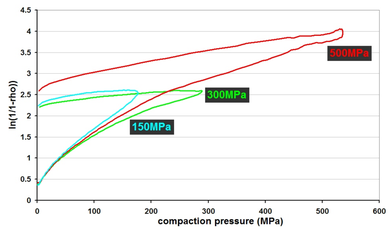

Fig. 4: inconsistent Heckel Plots of Avicel Ph102; 10mm flat punches

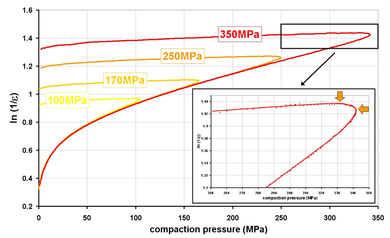

Fig. 5: plausible Heckel Plots of Ca-Phosphate; 9mm concave punches

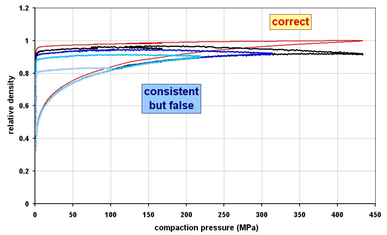

Fig. 6: consistent but false measurement data of Technische Tablettiermischung, 11,28mm flat punches

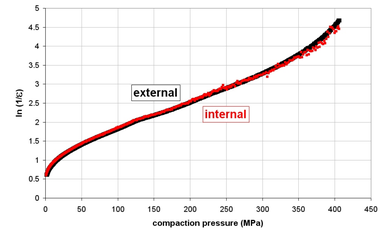

Fig. 7: comparing Heckel Plots of internal and external displacement measurement system, Technische Tablettiermischung, 11,28mm flat punches

Fig. 6 gives an example of consistent, but false plots of relative density versus compaction pressure. The blue curves would lead to the conclusion that density gets lower upon an increase of force, which is nonsense, and that the density maximum is obtained at approx. 30 per cent of the pressure maximum. Usually, this density maximum is located within the first 5 per cent after the pressure maximum[4] (cf. Fig. 5). The reason for receiving the false values is calculating with the wrong deformation. Determining the deformation for the external displacement measurement system is easier than for the internal one. In Fig. 7 the relevant part of two Heckel Plots acquired simultaneously with the internal and the external displacement measurement system shows good accordance. This is a good starting point for using the internal system, which is outside the actual tabletting zone and therefore easy to operate. A closer investigation on the differences between the two displacement measurement systems and a validation of the internal one particularly with regard to the accuracy will be made in the near future.

Conclusions:

It is possible to obtain exact Heckel Plots with concave punches under conditions close to production (effective dwell time of 7.5 ms).
Determining the reference positions for the in-die height measurement with a highly densified tablet seems to be a good method although the precision can be optimised slightly for example by using reference tablets with a smooth surface.
To check the plausibility of Heckel data, a set of experiments made at different maximum compaction pressures up to 400 to 600 MPa is strongly recommended. Moreover, the shape of each Heckel curve must be scrutinised. As obvious from the data, Heckel Plots are an error-prone method describing deformation behaviour of powders, due to the difficulties in measuring the in-die tablet height sufficiently accurately. Therefore, it might be difficult to evaluate literature data of Heckel Plots properly.
First results show, that internal displacement transducers of the Stylcam mounted outside the tabletting zone can measure the in-die tablet height in good accordance to the external ones. Closer investigations on the comparability of the two systems are on-going. The external measurement configuration described in this paper has the advantage of being able to calculate the deformation from the punch geometry. It can be adapted for most single-punch tablet presses. The internal measurement setup of the Stylcam has the advantage of location and is therfore easy to use.

References:

[1] Sonnergaard, J.M.; International Journal of Pharmaceutics 193(1), 63-71 (1999)
[2] Fretter, B. et al; Tablet Tech, Brussels (2007)
[3] Fretter, B. et al; 6th World Meeting on Pharmaceutics, Biopharmaceutics and Pharmaceutical Technology, Barcelona (2008)
[4] Schierstedt D., Müller F.; Pharm.Ind. 44 Nr.9 (1982), S.932-937

FIP Pharmaceutical Sciences 2010 World Congress in Association with the AAPS Annual Meeting and Exposition; November 2010, New Orleans, USA