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An experimental evaluation of an effective medium based compaction equation

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ABSTRACT

Tablet production involves compression of free flowing powder in an enclosed cavity of defined geometry. The complexity of the powder bed system necessitates that a way be found to better understand what occurs during compression. One such approach is by means of compaction equations, of which, the Heckel and Kawakita equations are the best known. This work attempts to experimentally evaluate the applicability of the effective medium (EM) equation introduced by Frenning et al, to powder systems. Two powder types (sodium chloride and lactose monohydrate), each consisting of three size fractions ($<40\ \mu\text{m}$, $125\text{--}212\ \mu\text{m}$ and $212\text{--}300\ \mu\text{m}$) were characterised and compressed to a pressure of 500 MPa. These powders were chosen because of their differing mechanical properties. An invariance which is inherent in the EM equation is exposed by varying the starting points of compression, and can yield insights into compression mechanisms. Such invariant regions were observed once plastic particle deformation started to dominate the compression behaviour, and enabled the determination of the point where particle rearrangement stops.

1. Introduction

Tablet production involves compression of free flowing powder in an enclosed cavity of defined geometry, situated within a die. Compression can be applied from various directions by pistons (punches), which reduce the volume of the powder bed, through expulsion of air from between the component powder particles. In pharmaceutical tablet production, compression is applied in a uniaxial direction by punches, which are analogous to pistons. The elimination of inter-particulate pores, creates intimate contact between adjacent particles, and begins the process of bonding. The ability of a powder bed, to reduce in volume when under compressive stress, is referred to as compressibility. The ability of a powder bed, to form a structure, when under compressive stress, which possesses a tensile strength upon ejection, is referred to as compactibility.

The production of a tablet comprises three predominant stages, which can be understood as, but not always exclusively, occurring in sequence (Johansson and Alderborn, 1996; Sun and Grant, 2001). When powder is first poured into the die, it forms, relative to its compressed state, a loose and unstable structure that is composed of pockets of trapped air surrounded by powder particles. Initial contact with the punch causes this structure to collapse, as the particles shift and occupy any pores that they can fit into; consequently this stage is referred to as rearrangement. The extent to which rearrangement occurs is dependent on particle size, which governs the probability of pore occupation. Soon thereafter, the system reaches a state where its capacity to rearrange is exhausted as the particles are constrained or locked into position by contact with their neighbours. This junction is often referred to as the jamming transition (Keys et al., 2007; Majmudar et al., 2007), and any further volume reduction is as a result of particle deformation (plastic or elastic) and/or fragmentation, which are material and size dependent. It is during this transitional phase that bonding occurs between the contacting surfaces of the powder particles, either as in the case of deformation, by an increased area of contact between particles, or by an increase in the number of bonding sites as in the case of fragmentation (Nyström et al., 1993). Eventually, assuming enough pressure is applied, the powder will no longer be a system of distinct particles, but rather a solid unit of zero porosity. Since particle fragmentation and plastic deformation cannot occur beyond zero porosity, the final stage of the compression process is invariably controlled by the elastic deformation of this solid unit. However, it must be noted that as mentioned above, the stages of compression are not always distinct. Fragmentation and deformation can span across the first two stages. Plastic and elastic deformation can occur simultaneously to different degrees. This intermeshing of phases adds a layer of complexity to the compression process. In any case, if the definition of a tablet is to be satisfied, the degree of bond formation between powder particles must be high enough, to yield the strength necessary, to maintain the structure of the newly formed compact upon ejection and subsequent handling.

Previous workers have made attempts to mathematically describe the compression behaviour of a powder bed, most notably Heckel and Kawakita, whose respective equations bare their names (Heckel, 1961; Kawakita and Lüdde, 1971). Other workers have attempted to extract single particle fracture force data from bulk powder compression (Adams et al., 1994; Adams and McKeown, 1996). Attempts have been made to modify and elaborate upon these equations, as well as to assign physically tangible meanings to their parameters (Sonnergaard, 1999; Denny, 2002; Hassanpour and Ghadiri, 2004; Nordström et al., 2008; Klevan et al., 2009; Nordström et al., 2009). The current article is an extension of work previously conducted by our group (Frenning et al., 2009), where a compaction equation was derived using effective medium (EM) theory, with the aim of modelling the compression response of granular materials consisting of approximately spherical agglomerates.

The EM equation in its derivation, assumed an endpoint compact of zero porosity, and that no elastic, but rather only plastic particle deformation took place during compact formation. Contrary to the largely empirical equations of Heckel and Kawakita, the EM equation was derived from single particle mechanics. As long as the assumptions underlying the derivation are met, the physical significance of its parameters is therefore known from the outset, at least in principle, as discussed by Frenning et al. (2009). The aim of this study was to investigate the applicability of the EM compaction equation to powders, to see whether its parameters truly reflect the underlying mechanical response of single particles, and to determine to what extent the equation can provide insights into the rate controlling compression mechanisms.

Sodium chloride (from hereon referred to as NaCl) and lactose monohydrate (from hereon referred to as lactose) powders were chosen as test materials because of their contrasting mechanical properties. NaCl is known to deform plastically under compression (Hardman and Lilley, 1970; Hersey and Rees, 1971), whereas lactose is brittle and fragments upon compression (Hersey and Rees, 1971). Down (1983) found that cracking occurred simultaneously with plastic flow during compression for NaCl particles within the 30-60 mesh size range. However, these cracks were sealed by plastic flow at higher pressures and did not propagate through the entire particle, indicating that brittle fracture does not occur (Down, 1983). The authors view on this matter is that the cracks seen in the NaCl particles are a result of a shear tearing mechanism rather than brittle fracture. Thus, it can be concluded that the choice of materials for this investigation remains rational in terms of contrasting compression mechanisms.

2. Experimental

2.1 Materials

Two different powder materials were used in this study; NaCl (Fluka Analytical, Switzerland) and lactose monohydrate (Pharmatose 50M, DMV international, Veghel, Netherlands).

2.2 Powder preparation and characterization

Apparent density: Both apparent densities of NaCl (2.147 g/cm^3) and lactose (1.538 g/cm^3) were determined with a helium pycnometer (AccuPyc 1330, Micromeritics, Norcross, GA). The reported values are the mean of three (NaCl) ($n=3$) and four (lactose) ($n=4$) independent measurements each composed of 10 runs.

Powder preparation: Three size fractions of lactose and NaCl were investigated. Two of these (125–212 and 212–300 μm) were obtained through dry sieving (Retsch, Type RV, Haan, Germany and Endecotts Ltd, London, England). The third was obtained by milling in a pin disk mill (Alpine 63C contraplex Labormuhle, Alpine AG, Augsburg, Germany). The volume specific surface areas (S_V) of the milled powders were determined by a transient air permeability apparatus (Blaine). Three experiments each with three determinations of flow time were performed for both powder types. S_V was calculated via the slip flow corrected Kozeny–Carman equation (Alderborn et al., 1985). The ratio between S_V and a surface to volume shape factor (Heywood shape factor) (Alderborn and Nyström, 1982) of 10 yielded an estimate of the milled powder particle diameters; NaCl: $(28.4 \pm 0.3) \mu\text{m}$, lactose: $(22.4 \pm 0.3) \mu\text{m}$ (mean $\pm$ standard deviation).

Bulk and tapped density: Bulk density (BD) was determined by pouring each size fraction of powder (4.1–12.7 g) into a graduated 25 ml measuring cylinder with a diameter of 14.25 mm (measured with a vernier caliper). The height occupied by the poured powder was determined with a height gauge (Mitutoyo Corporation, Japan) ($n=4$). The same cylinder was then tapped 1000 times (PharmaTest, PT-TD, Hainburg, Germany). The resultant height was determined with the height gauge mentioned above and used to calculate the tapped density (TD, $n=4$). The Hausner ratio (HR) was calculated from the BD and TD in the standard manner.

All the powders studied in this investigation were stored in approximately 40 % RH.

2.3 Powder compression

Powder compression was carried out with a Zwick Z100 materials tester (Zwick/Roell, Zwick GmbH & Co. KG, Germany) equipped with a mobile upper punch attached to a 100 kN load cell, and a stationary lower punch with a diameter of 11.3 mm. Prior to all compressions the die wall was lubricated with 1% magnesium stearate suspension in ethanol, and subsequently manually filled with powder. To ensure column height consistency, the weights required for both powders were calculated (NaCl; 0.50 g, lactose; 0.36 g) based on the density of a zero porosity compact. Five compressions were performed for each size fraction of lactose and NaCl. All powders were compressed to a maximum pressure of 500 MPa at a rate of 25 mm/min. Punch and machine deformation were compensated for in all compressions. The relative humidity of the room was regularly monitored, and ranged between 36.6–41.8%. It is of interest to note that, Ahlneck and Alderborn (1989) have shown that adsorbed surface water has a negligible effect on powder volume reduction properties. This finding demonstrates the robust nature of the powder compression process.

2.4. Analysis of compression data

Heckel equation: The Heckel equation is commonly written as (Heckel, 1961)

$$\ln\left(\frac{1}{1-D}\right) = KP + \text{Intercept} \quad \text{Eq. (1)}$$

where D is the relative density of the powder, thus equating $1 - D$ to porosity, P is pressure and K is a constant and slope of the linear region of the plot, whose reciprocal is equivalent to the yield pressure of the material being compressed.

Kawakita equation: The Kawakita equation takes the form (Kawakita and Lüdde, 1971)

$$C = \frac{abP}{1+bP} \quad \text{Eq. (2)}$$

where C is the degree of compression and a and b are constants. The degree of compression is in turn calculated as

$$C = \frac{V_0 - V}{V_0} \quad \text{Eq. (3)}$$

where V_0 is the initial powder bed volume and V is the powder volume upon compression. The Kawakita constant a describes the maximum degree of compression, and $1/b$ is the pressure applied to get $\frac{1}{2}$ the maximum degree of compression or $a/2$.

The Kawakita Eq. (2) may be rewritten in the linear form,

$$\frac{P}{C} = \frac{P}{a} + \frac{1}{ab} \quad \text{Eq. (4)}$$

enabling the constants a and b to be determined by linear regression. The starting heights of the Kawakita analysis in this investigation were determined from the bulk density. The maximal engineering strain, C_{max} , was determined as the degree of compression at 500 MPa.

EM equation: The underlying premise and derivation of the EM equation are presented by Frenning et al. (2009), however, a short description will be presented here. The equation considers that only plastic particle deformation takes place upon compression of bulk powder, and in its original form, that the end result of this compression is a compact of zero porosity. The EM equation describes how the compression pressure P increases with decreasing bed height H , from a starting height H_1 corresponding to a starting pressure P_1 , and is here written as

$$P - P_1 = A \left[\alpha \ln \left(\frac{H}{H_1} \right) - \ln \left(\frac{H - H_{\min}}{H_1 - H_{\min}} \right) \right] \quad \text{Eq. (5)}$$

where A , α and $H_{\min}$ are parameters. The A parameter determines the pressure scale and is on theoretical grounds expected to be proportional to the plastic stiffness/yield stress of the particles. The α parameter controls the initial slope in a P vs. H plot and is related to the structure of the force network (coordination number and Watson integral (Thorpe et al., 1989)). Finally, $H_{\min}$ is the minimal height attainable by compression. The EM Eq. (5) has a slightly more general form than the one given by Frenning et al. (2009) and reduces to the latter if, firstly, the starting height H_1 is selected such that the starting pressure P_1 is zero and, secondly, the minimal height $H_{\min}$ is selected such that it corresponds to a compact of zero porosity.

When written in this form, the EM Eq. (5) has an interesting and useful invariance under changes of starting height. To see this, assume that a starting height H_1 has been found, such that Eq. (5) is valid for all H smaller than H_1 (but not smaller than $H_{\min}$). At an arbitrary H_2 (between H_1 and $H_{\min}$), the pressure P_2 then satisfies

$$P_2 - P_1 = A \left[\alpha \ln \left(\frac{H_2}{H_1} \right) - \ln \left(\frac{H_2 - H_{\min}}{H_1 - H_{\min}} \right) \right] \quad \text{Eq. (6)}$$

Subtracting Eq. (6) from Eq. (5), and performing a straightforward calculation using the properties of logarithms, it is found that

$$P - P_2 = A \left[\alpha \ln \left(\frac{H}{H_2} \right) - \ln \left(\frac{H - H_{\min}}{H_2 - H_{\min}} \right) \right] \quad \text{Eq. (7)}$$

an equation that clearly is identical in form to Eq. (5).

The usefulness of this invariance is twofold. Firstly, it minimises the influence of the often somewhat arbitrarily selected starting height. Secondly, it allows a well-defined initial height H_0 to be determined (see below). The thus determined initial height H_0 may be used as an indicator of the point at which rearrangement stops (the jamming transition (Keys et al., 2007; Majmudar et al., 2007)).

In practical terms, two different procedures can be used to extract values of H_0 . For the essentially non-fragmenting NaCl, H_0 could be determined by straightforward extrapolation. The initial step was to determine the pressure range at which A , α and $H_{\min}$ remained constant. This was performed through selecting (by approximation) incrementally increasing starting pressures, and carrying out least squares minimisation with each of these chosen values. Once the constant A , α

and H_{min} values were obtained, at a certain starting height H_1 and pressure P_1 , H_0 was determined as the solution to Eq. (5) when P was put equal to zero. However, this procedure could not be used for the fragmenting lactose, because the derivative

$$\frac{dP}{dH} = A \left(\frac{\alpha}{H} - \frac{1}{H - H_{min}} \right) \quad \text{Eq. (8)}$$

was equal to zero at a height corresponding to a positive pressure. For this reason, H_0 was instead determined as the solution to Eq. (8) when the derivative dP/dH was put equal to zero. Finally, the experimental pressure that corresponded to the experimental powder bed height most closely matching H_0 , was selected as being presented below.

As noted above, the EM equation in its derivation disregards elastic particle deformation. For this reason, it was considered advantageous to correct in-die compression data for elastic compact deformation prior to the application of the equation. During the unloading branch of the compression cycle, the compact height is expected to increase linearly with the decrease in pressure,

$$\frac{\Delta H}{H_{relaxed}} = \frac{H_{relaxed} - H}{H_{relaxed}} = \frac{P}{E} \quad \text{Eq. (9)}$$

where $H_{relaxed}$ is the compact height after unloading and E is an appropriate elastic modulus. Values of E were extracted by fitting Eq. (9) to unloading data in a pressure range of about 490–120 MPa, with an R^2 of at least 0.99. Compact height data under pressure were subsequently corrected for elastic deformation by adding the value of ΔH calculated by Eq. (9). Although the following discussion will largely be based on this corrected data, results obtained for uncorrected data will also be presented for comparative purposes.

3. Results and Discussion

3.1 Powder characteristics

From looking at the values presented in Table 1, it is apparent that the milled and unmilled powders form two distinct groups in terms of HR, BD and TD. The milled powders have lower BD and higher HR than their unmilled counterparts, as is to be expected due to their greater cohesiveness. Although possessing similar HR, the BD and TD of the NaCl powders are higher than that of their lactose counterparts, as would be expected from the difference in apparent density between the powders (Sec. 2.2). To summarise, the unmilled powders within each powder

type can be grouped together due to their similar BD, TD and HR, which are generally significantly different from their milled counterparts. The only exception to this is the TD of the milled lactose with respect to the unmilled lactose. Rather than having a lower TD than its unmilled counterparts, it has a TD which is higher while remaining numerically close to the unmilled, unlike the milled NaCl.

3.2 Compaction analysis

Compression profiles: Fig. 1 displays the averaged compression profiles of all powders based on starting heights (Table 2) determined from bulk density (the data presented in this and the following figures are the averages of five independent runs). The striking feature of this plot is the similarity between the unmilled samples within each powder type, and the large difference with their milled counterparts. The plot highlights the significant difference in compressibility between the milled and unmilled samples, as is evidenced by their C_{max} values (see Table 2).

Heckel analysis: The averaged Heckel profiles of all powders are displayed in Fig. 2. The Heckel parameter was determined by calculating the inverse of the slope of the linear region of the Heckel plot. The linear region was determined for each powder type by visual inspection of the plots. The pressure range for NaCl was approximately 30–120 MPa and that for lactose was approximately 90–240 MPa. The R^2 values of all the linear regions were > 0.9999 . It is apparent from the Heckel plots that the NaCl powders densify with greater ease than the lactose powders. It is also apparent that the two unmilled samples within each powder type behave nearly identically to each other, as a consequence of which, their yield pressures can be said to be the same (see Table 2). Conversely, the milled samples require higher pressures to achieve equivalent porosities, and have higher yield pressures (see Table 2) than their unmilled counterparts.

Kawakita analysis: The averaged Kawakita plots of all the powders are displayed in Fig. 3. The linear region of the Kawakita plot was determined visually, and for all the plots was taken between 260–500 MPa, with the R^2 values being ≥ 0.99999 . It is apparent from Fig. 1 and 3 that the unmilled samples within each powder type display near identical behaviours. The milled powders however are markedly different and have significantly higher a values and lower $1/b$ values (see Table 2), indicating that they are much more compressible than the unmilled samples.

EM analysis: Fig. 4 displays representative EM fits to the elastically corrected experimental compression curves of all the powders at a starting pressure of 30 MPa (taken from the third compression run for all powders). Elastic moduli for the compacts, extracted from the unloading data using Eq. (9), are provided in Table 2. It is apparent that the EM fits describe the experimental

data very well in all cases, demonstrating that the EM equation is valid for systems of μm sized particles. The corresponding parameter values extracted via the EM equation using different starting heights and pressures are displayed in Fig. 5. The powders employed in this study can be thought of as representing general types of systems, and what follows below is an interpretation showing how the EM equation can be used to better understand what is happening in the powder bed, in terms of both compression mechanisms and their sequence.

Unmilled NaCl represents a plastically deforming system which undergoes limited rearrangement. For this type of system it is apparent from Fig. 5 that the A and α parameters are largely independent of starting pressure. Milled NaCl on the other hand represents a plastically deforming system that undergoes marked rearrangement. In this case, an initial decrease in A and α preceding a flat line is observed. A highly rearranging system close to its brittle ductile transition (see below) is represented by milled lactose, where there is an initial increase in A and α preceding a flat line. Brittle systems undergoing limited rearrangement are represented by the unmilled lactose samples, where the values of A and α increase across a larger pressure range prior to leveling off. For all the powders, the minimum height H_{min} was consistently between about 2.27 and 2.37 mm (data not shown).

The H_0 values determined from the EM equation (see section 2.4) provide an indication of the point at which the system reaches a constrained/jammed state, i.e. the point at which rearrangement stops (see Table 2). Once the constrained height is known, rearrangement can with greater certainty be eliminated as a factor when trying to understand the mechanisms of compression for a particular system. This finding is of great value, as it clarifies what is often an unknown in compaction analysis. Based on the assumptions underlying the derivation of the EM equation, it can be assumed that the flattening of the curve is indicative of a predominantly plastic particle deformation. Again, in the same way as mentioned above, the identification of a predominating process in the sequence of compression, allows for a more focused, detailed and comprehensive analysis of the compression process. Additionally, the presence or absence of, and the curvature preceding or following the flat regions in Fig. 5, can also aid in better understanding the compression process in terms of the mechanical properties of the material.

The invariance of the A and α curves in Fig. 5 for all powders, indicates that the materials are behaving in such a way that they fulfill the criteria of the EM equation i.e. that plastic deformation is occurring. Milled lactose however reaches a constrained state (at approximately 5 MPa) prior to its invariant region. The particle size of the milled lactose (22.4 μm) is below its critical particle size (45 μm (Rowe and Roberts, 1996)) where the “brittle–ductile transition” occurs, implying that fragmentation should be limited for this powder (Roberts and Rowe, 1987). Since lactose is well known for its fragmenting properties, it is possible that there was a small degree of brittle fracture

after the constrained state was reached, resulting in some rearrangement immediately prior to the invariant region, from where plastic deformation took over as the predominant mechanism. The milled NaCl on the other hand begins to level out very close to the vicinity where it was constrained, indicating a near immediate transition to plastic deformation. Due to their larger particle sizes, the unmilled lactose and NaCl powders are expected to cease rearranging at low pressures. From Fig. 5 it is apparent that the unmilled NaCl powders nearly immediately transition to a constrained state followed by plastic deformation. The invariant region for the unmilled lactose powders as in the milled lactose occurs after their jamming transition. This is most likely due to a consecutive cycling process of fragmentation and rearrangement that occurs over a wider pressure range than their milled counterpart.

For comparative purposes, Fig. 6 shows the results of an EM analysis applied to the uncorrected compression data. The results obtained for the NaCl powders are seen to be largely unaffected by the correction for elastic deformation, both in terms of the A and α parameters. In contrast, for the lactose powders, the extracted A values are significantly higher for the uncorrected compared to the corrected compression data, whereas the α parameter is less affected. In addition, the invariant region is conspicuously absent from the curves in Fig. 6 of the unmilled lactose powders. These results may be understood in terms of differences in the E/A ratio, which for NaCl is on average of about 380 and for lactose approximately four times smaller when the A values are based on elastically corrected data. As mentioned in Sec. 2.4, our A parameter is expected to reflect the yield stress of the particles, but need not be numerically similar. Hence a critical value of the E/A ratio should exist (Hassanpour and Ghadiri, 2004), below which the A parameter extracted from the uncorrected data no longer reflects the plasticity of the particles, exactly as observed in our data for lactose. Conversely, if the powder system is in such a state that it undergoes mainly plastic deformation without much elastic deformation or fragmentation, the EM equation is capable of accurately describing its compression process with or without correction for elastic compact deformation, as for NaCl.

3.3 General discussion

Milled powders in the investigated size range are known to be very compressible. This is evidenced in Fig. 1, where the maximal degree of compression is significantly higher for the milled powders. In addition, these powders exhibit a large initial compressibility, with a degree of compression of around 0.5 at 1 MPa. When the initial height is based on bulk density, the initial degree of compression is a powerful determinant for the parameters of the Kawakita equation. This in turn means that the $1/b$ parameters reflect the resistance to initial compression by particle

rearrangement rather than particle deformation. Additionally, Fig. 2 and 3 share the same trends with regard to the similarities between the unmilled powders, and their differences with the milled powders. This difference can be seen as a result of the greater compressibility of the milled powders.

Both the Heckel $1/K$ and the EM A parameter are expected to reflect the plasticity of the particles. Although the EM A varies somewhat with starting height for the fragmenting materials, it is evident that the Heckel $1/K$ and the EM A follow similar trends, with larger values obtained for lactose than for NaCl, and larger values for the milled than the unmilled powders (Table 2). This observation strengthens the role of the EM A parameter as an indicator of plasticity, comparable to the Heckel yield stress.

In addition to the A parameter, the EM analysis also provides values for the α parameter, which were consistently about twice as large for the lactose as for the NaCl powders (Fig. 5). The α parameter controls the initial slope in a P vs. H plot, as shown by Eq. (8). We interpret α as an indicator of initial compressibility. Based on the theoretical analysis presented in (Frenning et al., 2009), it can be concluded that α depends on the force network at the jamming transition, in terms of the coordination number (relative to the maximum value obtained during compression), relative density and Watson integral (Thorpe et al., 1989). Higher values of α are promoted by a decreased relative coordination number, an increased relative density and an increased Watson integral. For the lactose powders, this picture is complicated by particle fracture. Although the causative agents are known in principle, further studies are needed in order to pinpoint their relative contributions.

The characteristic that sets the EM equation apart from the Heckel and Kawakita, is what seems to be its ability to accurately describe the compression response of plastically deforming particles, for which an invariance under changes of compression starting point is observed. This in turn enables the determination of the point at which rearrangement stops, allowing the underlying compression mechanisms to be deduced.

4. Conclusion

An invariance which is inherent in the EM equation is exposed by varying the starting points of compression, and can yield insights into the mechanisms that control the rate of the compression process. This invariance was experimentally observed once plastic particle deformation started to dominate the compression behaviour, and enabled the determination of the point where the powder bed entered a jammed state. The descriptive ability of the EM equation for μm sized plastically deforming NaCl has been demonstrated, and corroborates previous work performed on systems composed of plastically deforming mm sized particles. The potential applicability of the

EM equation in terms of material type and particle size has been demonstrated. The A parameter of the EM equation is analogous to the Heckel $1/K$ and as such to the yield pressure of the material. Further work with the EM equation may involve validating the existing results by expanding the range of materials that are tested, and further probing the ability of the EM equation in determining the constrained height of powder beds.

List of symbols

A	EM parameter that determines the pressure scale	[Pa]
a	Kawakita constant	[-]
b	Kawakita constant	[Pa ⁻¹]
C	Degree of compression	[-]
$C_{\max}$	Maximal engineering strain	[-]
D	Relative density of the powder	[-]
E	Elastic modulus	[Pa]
H	Powder bed height	[m]
H_1	Powder bed starting height	[m]
$H_{\min}$	Minimal height attainable by compression	[m]
H_{relaxed}	Height after unloading	[m]
K	Constant and slope of the linear region of the Heckel plot	[Pa ⁻¹]
P	Pressure	[Pa]
P_1	Starting pressure	[Pa]
V	Powder bed volume upon compression	[m ³]
V_0	Initial powder bed volume	[m ³]
α	EM parameter that controls the initial slope in a P vs. H plot	[-]

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Figures

Fig. 1. Averaged experimental compression profiles for NaCl and Lactose. Starting height based on bulk density. To maintain clarity error bars are not shown as standard deviations are smaller than the symbols.

Fig. 2. Averaged Heckel plots for NaCl and lactose. Error bars (some obscured by the markers) are standard deviations.

Fig. 3. Averaged Kawakita plots of NaCl and lactose. To maintain clarity error bars are not shown as standard deviations are smaller than the symbols.

Fig. 4. Representative EM fits (lines) to experimental compression curves (symbols) for all powder types with starting pressure at 30 MPa (data taken from the third compression run for all powders; EXP = experimental data, EM = effective medium fit).

Fig. 5. Average EM A and α parameters against varying starting pressures, extracted from data corrected for elastic compact deformation. Error bars (some obscured by the markers) are standard deviations.

Fig. 6. Average EM A and α parameters against varying starting pressures, extracted from uncorrected data. Error bars (some obscured by the markers) are standard deviations.

Tables

Table 1. Bulk and tapped densities with corresponding Hausner ratios for all size fractions of NaCl and lactose.

	NaCl			Lactose		
	Milled	125-212 μ m	212-300 μ m	Milled	125-212 μ m	212-300 μ m
Bulk density (g/cm ³)	0.54 (0.013)	1.08 (0.022)	1.11 (0.003)	0.46 (0.011)	0.65 (0.011)	0.66 (0.022)
Tapped density (g/cm ³)	0.83 (0.024)	1.18 (0.012)	1.19 (0.015)	0.77 (0.016)	0.72 (0.009)	0.71 (0.010)
Hausner ratio (-)	1.56 (0.070)	1.09 (0.027)	1.07 (0.015)	1.68 (0.070)	1.11 (0.011)	1.08 (0.039)

Table 2. Average Heckel, Kawakita and EM parameters (A , α and H_{min} were taken at a starting pressure of 30 MPa).

	NaCl			Lactose		
	Milled	125-212 μ m	212-300 μ m	Milled	125-212 μ m	212-300 μ m
Heckel						
$1/K$ (MPa)	91 (0.3)	73 (0.2)	72 (0.2)	155 (1.1)	138 (1.3)	137 (0.8)
Kawakita						
C_{max} (-)	0.757 (0.0008)	0.511 (0.0009)	0.499 (0.0005)	0.703 (0.0002)	0.583 (0.0003)	0.579 (0.0003)
a (-)	0.768 (0.0008)	0.526 (0.0009)	0.514 (0.0005)	0.721 (0.0002)	0.606 (0.0003)	0.601 (0.0004)
$1/b$ (MPa)	6.86 (0.052)	14.5 (0.12)	14.6 (0.14)	12.8 (0.04)	20.2 (0.10)	19.8 (0.06)
EM						
A (MPa)	110 (1.0)	89 (0.8)	89 (0.6)	234 (1.9)	199 (1.8)	188 (2.1)
E (GPa)	38.6 (2.43)	33.5 (2.04)	37.7 (0.90)	19.8 (0.50)	21.7 (0.55)	17.8 (0.51)
α (-)	1.39 (0.015)	1.41 (0.015)	1.44 (0.012)	3.08 (0.010)	3.14 (0.008)	3.29 (0.007)
H_0 (mm)	3.99 (0.015)	4.19 (0.020)	4.14 (0.020)	3.45 (0.005)	3.42 (0.004)	3.37 (0.005)
Exp. P at H_0 (MPa)	4.18 (0.055)	1.67 (0.171)	1.57 (0.165)	5.37 (0.062)	12.03 (0.091)	11.98 (0.117)
H_{min} (mm)	2.28 (0.008)	2.31 (0.005)	2.30 (0.002)	2.33 (0.002)	2.33 (0.003)	2.35 (0.003)