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# Compaction of bidisperse cohesive powders

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**Abstract** The relationship between an external stress applied to a bulk and its resulting porosity—i.e. its compaction behavior—is studied in this work, varying the median particle diameter by using different batches of carbonyl iron powder (CIP) as well as by mixing them. Firstly, compaction equations of other authors and an algebraic relation suggested by us are presented. Secondly, these descriptions are compared to data obtained by compacting CIP. Last, for powder mixtures, the role of the particles' median and mean diameters is studied in experiments and DEM-simulations, respectively. We show that they take on the role of the relevant length scale appearing in the algebraic compaction law.

**Keywords** Bidisperse cohesive powders · Compaction · Porosity · Experiments · Contact dynamics simulation

## 1 Introduction

The understanding of the influence of microscopic parameters on the macroscopic behavior of powders is essential when aiming at developing physically based material models for powders. In experiments, only few microscopic parameters can be set with a reasonable expenditure such as the particle size, the amount of contact liquids etc.

The influence of the microscopic—but averaged—median particle diameter $x_{50}$ on the powder's compaction behavior [1], tensile strength [2] and shearing behavior [3] has been the topic of several works carried out by the authors of this paper [4]. The approach is to investigate the microscopic and macroscopic behavior of several batches of identical powders which present a narrow particle size distribution and which only differ from each other by their median particle diameter. Batches of powders with these properties such as limestones, zinc dusts and carbonyl iron powders are commercially available.

A further step when varying the median particle diameter is to mix the batches of identical powders—differing in their median particle diameter—and to hereby obtain a blend—which will be referred to as a "bidisperse powder"—with an intermediate median particle diameter and a broader particle size distribution. The primary powders necessarily need to be cohesive, as non-cohesive powder blends tend to segregate during the experiment (a problem which can be dealt with in simulations, though, cf. [5]).

The mechanical properties of bidisperse powders have been the topic of simulations investigating e.g. the segregation dynamics [6,7] and shear [8] but seldom for cohesive materials [9]. Experimental investigations have been carried out, analyzing e.g. the geometrical [10] and structural [11] properties of cohesion-less bidisperse media. In this work, the compaction of cohesive powders (primary and bidisperse powders) is investigated in experiments and simulations. The findings are compared to existing compaction equations.

## 2 Experimental setup

### 2.1 Compaction tests

The compaction of cohesive powders had been investigated at stress levels up to 30 kPa using the true biaxial shear

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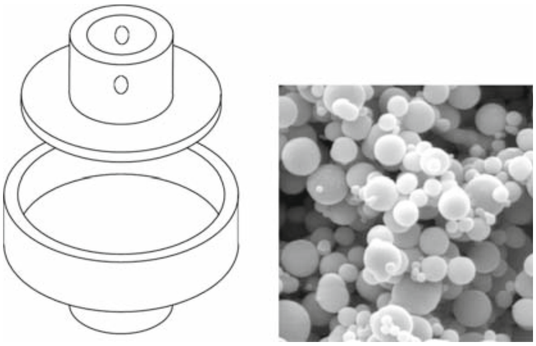

**Fig. 1** Cylindrical container $d = 60\text{mm}, h = 20\text{mm}$ and piston mounted on the testing machine and used for compaction experiments (*left*), and SEM-photograph of carbonyl-iron-powder, image width 9 µm (*right*)

tester, which allows for homogeneously compacting the powder sample minimizing shear stresses at the specimen's borders, cf. [12]. In the present paper, we will use experimental results which had been acquired over two orders of magnitude using a uniaxial compaction tester, based on a standard testing machine, used in materials sciences e.g. for tensile and compression tests. For the experimental work carried out here, it was equipped with a cylindrical container with a diameter of 60 mm and a height of 20 mm, see Fig. 1 (left). The powder was sieved and then compacted in direction of sieving (i.e. vertically) by a piston with a speed of 0.1 mm/min. The maximum load of about 7 kN, which could be reached with this setup, corresponds to a compaction stress of about 2 MPa.

### 2.2 Powders

The experiments have been carried out using carbonyl-iron-powder (Fig. 1 right), which has been used as model powder both for microscopic and for macroscopic investigations in the past years [4].

Here, experimental results of the compaction of three batches of narrowly size-distributed carbonyl-iron-powders ($x_{50} \approx 2, 4$ and 6 µm, respectively) as well as of a mixture (of the 2 micron and the 6 micron sized powder) with a mass ratio of 1 : 1 are presented, cf. Fig. 2. The mixing procedure had been optimized for two batches of limestone presenting similar median particle sizes [13] and has been carried out by means of a Turbula mixer at 64 rpm during 2 min.

## 3 Simulation setup

### 3.1 The contact dynamics method

In principle, the stress/porosity-relation is a combination of the contributions of particle rearrangements and of the particle deformations. To focus only on the former, it is desirable to employ rigid particles, as done by the Contact Dynamics method (cf. e.g. [14]). It implements volume exclusion, Coulomb friction ($|F_t| \leq \mu F_n$, $F_n$ being positive for repulsion), Coulomb-like rolling friction ($|T_{\text{coul}}| \leq \mu_r F_n$) and an attractive normal force ($F_n \geq -F_c$) within a cohesion range $d_c$, in which the cohesion force $F_c$ is present. The technical subtleties of rolling friction in Contact Dynamics will be discussed elsewhere [15].

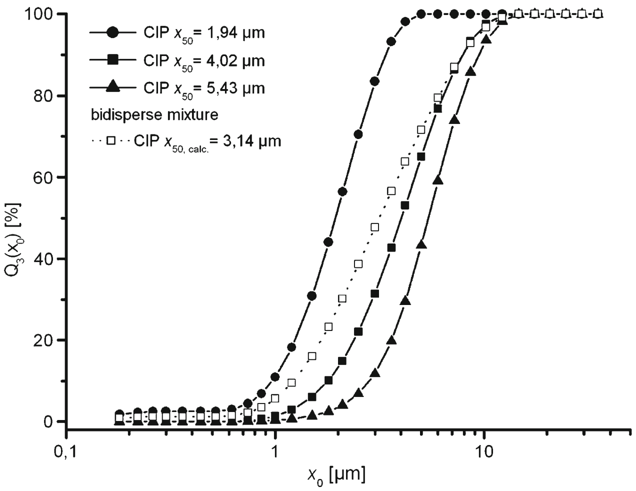

**Fig. 2** Particle size distributions of primary batches and mixture of CIP. When assuming perfect mixing, the distributions can be linearly combined and yield a median of $x_{50} = 3.14$ µm

### 3.2 Simulation of bidisperse powders

In our two-dimensional simulations, a perfectly bidisperse powder was investigated, i.e. a powder composed of discs of two different diameters $x_{1,2}$ with $x_2 = 2x_1$ (and correspondingly two different masses $m_2 = 4m_1$).

As initial configurations, highly porous systems of dendritic structure (cf. Fig. 3) with periodic boundary conditions in the lateral direction were used, where a certain amount of small particles (diameter $x_1$) was randomly replaced with larger particles (diameter $x_2$), the total number of particles being kept constant $N = 6503$. The friction coefficients used were $\mu = 0.2$ and $\mu_r = 0.1x_1$.

The uniaxial compaction was performed by means of a piston with a small, fixed velocity

$$v \ll \sqrt{\frac{F_c d_c}{m_2}}, \tag{1}$$

where $m_2$ is the mass of the larger particle. The force $F_p$ on the piston was measured and yielded, after division by the lateral system size $L = 150x_1$, the piston stress $\sigma$ which in turn was regarded as a function of the porosity $E$.

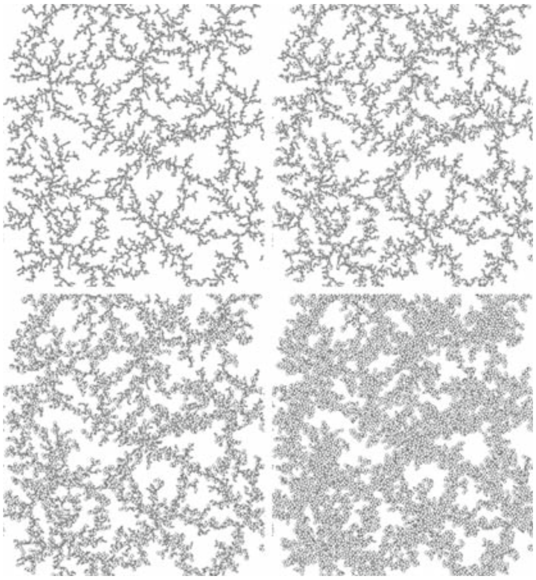

**Fig. 3** Initial configurations for the uniaxial simulation, containing (from *left* to *right*), 0, 10, 30 and 60% of large particles

## 4 Description of the compaction process

### 4.1 Existing compaction equations

In this paper, we will compare our findings both to the widely used, so called Kawakita-equation [16] as well as to a more modern approach given by Grossmann et al. [17].

#### *4.1.1 Kawakita-Equation*

A description widely used [18] in the field of practical powder handling stems from the 1970's, when Kawakita [16] gave a review and comparison over not less than 15 compaction equations and promoted the following for metallic and pharmaceutical powders (here formulated in terms of porosity $E$):

$$E = E_\infty + \frac{E_0 - E_\infty}{1 + \frac{\sigma}{\sigma'}\frac{1-E_0}{1-E_\infty}} \tag{2}$$

Here, $E_0$ is the initial porosity for zero (or rather the small, weight induced) pressure, and $E_\infty$ the porosity obtained in the limit of very high stresses, while $\sigma'$ is a characteristic stress of the Kawakita description. In terms of the volume fraction $\nu = 1 - E$ it reads

$$\frac{\nu}{\nu_0} = \nu_* - \frac{\nu_* - 1}{\frac{\sigma}{\sigma' \nu_*} + 1}, \tag{3}$$

where $\nu_* = \nu_\infty/\nu_0$ is the maximal relative increase in density.

#### *4.1.2 Gas-Law-Type equation*

A recent approach of an equation relating the bulk porosity to compaction stresses (of magnitudes from 10 to 1000 kPa), inspired by the ideal gas law, is given in [17] as

$$\frac{\nu}{\nu_0} = \frac{1-E}{1-E_0} = \left(1 + \frac{\sigma_M}{\sigma_0}\right)^n, \tag{4}$$

where $\sigma_0$ denotes the isostatic tensile strength of the powder and $n$ the so-called compression index $0 \leq n \leq 1$. The comparison of Eq. (4) to other descriptions (e.g. Eq. (2) or Eq. (5)) is hampered by the fact Eq. (4) is formulated for a powder during steady state flow, $\sigma_M$ being the corresponding center stress, and not for an uniaxial compression with piston stress $\sigma$. A relation between $\sigma_M$ and $\sigma$ lacking in [17,19], we assume at least proportionality between them.

### 4.2 Algebraic relation

It is plausible not to expect a compaction relation to be applicable to the whole range of stresses which can occur in experiments. For small stresses (not more than about 5 kPa in the present case), effects of the sample preparation may still be present (cf. also [1]), while for very large stresses the compaction mechanism must change from particle rearrangement to (elasto-plastic) particle deformation. Since we exclude the latter from our description, the asymptotic porosity $E_\infty$, a hypothetical quantity, must be non-zero and cannot depend on the cohesive properties of the powder anymore. Thus, it can only be a function of the particle friction (cf. [20] for that) and size distribution and can be expected to differ even between different batches of the same powder material. On the other hand, $E_0$, occurring in the descriptions Eqs. (3) and (4), is not a material parameter.

In several experiments and simulations of dry cohesive powders (cf. e.g. [1,3,21–23]), we found an algebraic dependence of the remainder $E - E_\infty$ as

$$E - E_\infty = \left(\frac{\sigma}{\sigma_*}\right)^{-\alpha}, \tag{5}$$

which interestingly appeared in the context of consolidating clays and silts as well [24].

To get rid of one of the three parameters ($E_\infty$, compaction exponent $\alpha$ and characteristic stress $\sigma_*$) one can take the derivative with respect to the stress:

$$E' \equiv \frac{dE}{d\sigma} = \frac{1}{\sigma_*}\left(\frac{\sigma}{\sigma_*}\right)^{-(1+\alpha)} \tag{6}$$

An intriguing property of $E'$ is the empirical, rather robust finding of $x_{50} \cdot E'$ to yield a data collapse. For a discussion of the implications for the compaction exponent, which was experimentally found to range between 0.1 and 0.5 and to

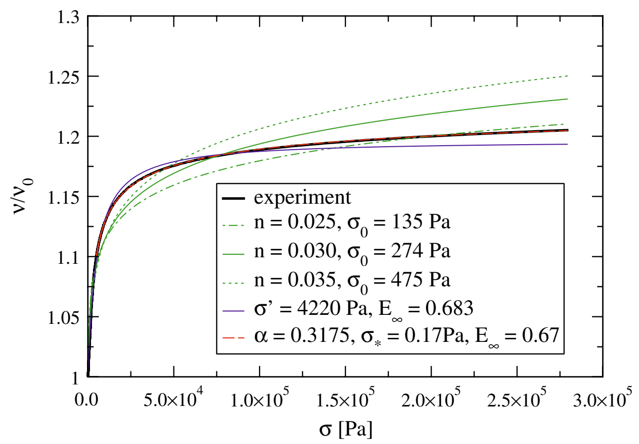

**Fig. 4** Experimental compaction data for CIP6 (*thick black*) compared to the descriptions Eq. (4) (*grey*: *solid*, *dotted and dot dashed*), Eq. (3) (*black*), and Eq. (5) (*grey dashed*). For Eq. (4), three suitable values for $n$ were fixed and $\sigma_0$ kept as fitting parameter, while for Eq. (3) the two fitting parameters were $\sigma'$ and $\nu_*$ (the latter providing $E_\infty = 1 - \nu_0\,\nu_*$). For Eq. (5), $\sigma_*$ and $E_\infty$ were fitted while $\alpha$ was extracted from Eq. (6), cf. also Fig. 5

mostly be close to 0.3, confer [4]. A key point of this work will be the role of $x_{50}$ in powder mixtures.

## 5 Results

### 5.1 Experiments

Just as there are many proposed relations between compaction and stress, there are many ways to display them. E.g. in the context of the Kawakita equation, data was originally plotted as

$$\frac{E_0 - E}{1 - E}\,\sigma \quad \text{vs.} \quad \sigma \tag{7}$$

For a powder obeying the Kawakita equation, this yields a straight line with

$$\text{slope} \quad \frac{1 - E_\infty}{E_0 - E_\infty} \equiv \frac{1}{a} \quad \text{and offset} \quad \frac{\sigma'}{a}, \tag{8}$$

with a certain lack of sensitivity for extracting the parameters $a$[1] and $\sigma'$, though ($\sigma'/a$ being small compared to the stresses $\sigma$).

Another possibility is to plot the volume fraction $\nu = 1 - E$ normalized to the initial one $\nu_0$ as done in [17] and in Fig. 4, which shows experimental compaction data for CIP6. The regime of our values found for the exponent $n$ can be read off in [17,19] as corresponding to "free flowing, fine sand". This rather surprising outcome may be attributed to the oversimplified relation between steady state flow center stress $\sigma_M$ and piston stress $\sigma$.

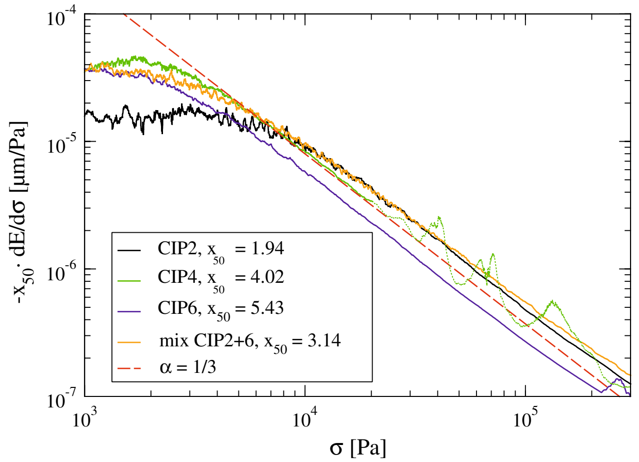

**Fig. 5** Numerically obtained derivative $dE/d\sigma$, rescaled by the particle median $x_{50}$. For the mixture, this value was calculated according to the mass ratio and the distributions of the primary batches (cf. Fig. 2). The CIP4 suffered from severe stick-slip above a stress of 20 kPa

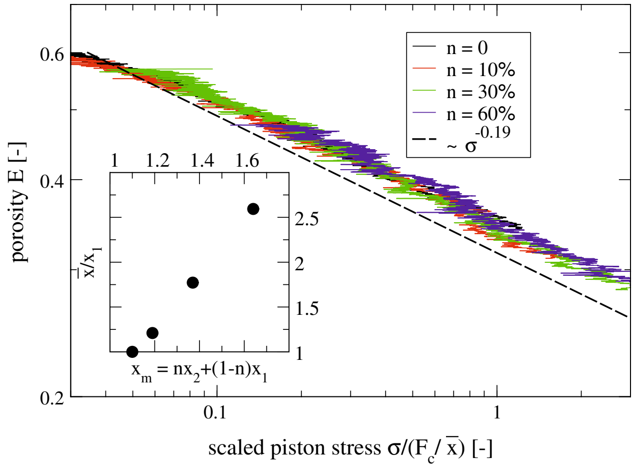

**Fig. 6** Compaction curve for simulations of bidisperse systems with a relative amount of $n$ large particles. A data collapse of fair quality is obtained when rescaling the piston stress with the characteristic stress $F_c/\overline{x}$ where the dedimensionalized length scale $\overline{x}/x_1$ is a linear function of the mean diameter

To check the role of $x_{50}$ in the case of mixed powders, we used the calculated value $x_{50} = 3.14\,\mu\text{m}$ for rescaling the derivative $dE/d\sigma$ in Fig. 5. As can be seen, it fulfills its role as the relevant length scale.

### 5.2 Simulation

The simulation results are displayed in Fig. 6. Due to the high noise, numerical differentiation did not produce reliable results even after averaging.

Thus, the porosity itself instead of its derivative is plotted (disregarding the offset $E$). As can be seen, the data is compatible with a power-law with an exponent $\alpha \approx 0.19$, and

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[1] Note, that only setting $E_\infty = 0$ yields $a = E_0$ as done in [18].

does essentially collapse onto one curve when rescaled by a factor λ which is proportional to the mean diameter $x_m$.

## 6 Conclusion

We addressed the role of the particle size in constitutive equations for the compaction of cohesive powders. The question whether the law (5) found in [3] could be extended in a simple way for cases without a well defined particle size (non-monodispersity) was answered positively by experiments as well as Contact Dynamics simulations. For the former, the particle size $x_{50}$ had to be substituted by the median of the mixture, while for the perfect bidisperse mixtures in the simulations (where no median is defined) the mean size defined as the amount weighted sizes took on this role. Up to which size ratio $x_2/x_1$ this convenient, simple scheme is valid, remains to be clarified in the future.

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