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Flowability assessment of weakly consolidated powders

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Abstract. The inability of cohesive powders to flow consistently and reliably is a major cause of process downtime and reduced efficiency across a wide range of powder processing industries. Most methods to assess powder flowability fail at low consolidation pressures (<1 kPa). In this paper, the ball indentation technique is used to assess the flow behaviour of two powders at low stresses by determining the bed hardness. In parallel, the powders are subjected to shear testing in a range of high stresses, with the derived unconfined yield strength used, along with the indentation hardness to define the constraint factor (*C*). By using the latter, which is considered independent of the preconsolidation stress applied, the unconfined yield strength of the powders at low stresses are inferred from the penetration hardness measurements.

1 Introduction

Nowadays, a large proportion of the materials processed and the products manufactured in industry are in the form of particulate solids. Several industries, such as pharmaceuticals, petrochemicals, cosmetics, food, materials, detergents and metallurgy comprise of processes that involve granular materials, such as fluidization, pneumatic conveying, blending, and storage in bins or hoppers. One of the major concerns regarding bulk solids handling is the flowability of powders in order to properly design the process and the equipment needed.

There have been various techniques developed for assessing powder flowability over the years, based on a variety of different principles. Some of them are quick and easily operable, such as the angle of repose, which are suitable only for a rough classification of flowability for quality control, while others, such as the uniaxial compression test and most notably shear testers are well established, providing quantitative results and offering stress control to the operator [1]. When it comes to evaluating the flow behaviour of cohesive powders at low consolidation pressures, which are involved in processes of great industrial interest e.g. in filling and dosing of powders in capsules, in feeding powders for packing and tableting machines, and dispersion in dry powder inhalers (DPI), all of the aforementioned methods fail. Furthermore, they are incapable of assessing flow at strain rates above the quasi-static regime as defined by Tardos *et al.* [2]. To address these issues, Hassanpour and Ghadiri introduced the ball indentation technique [3], which is based on penetration tests, with its operational window established by Zafar *et al.* and Pasha *et al.* [4, 5]. It involves a powder bed that is prepared inside a cylindrical die and is pre-consolidated by a piston. Then, the compressed bed is penetrated by a spherical indenter at a specified strain rate, until a certain load is reached, and then unloaded (Fig.1).

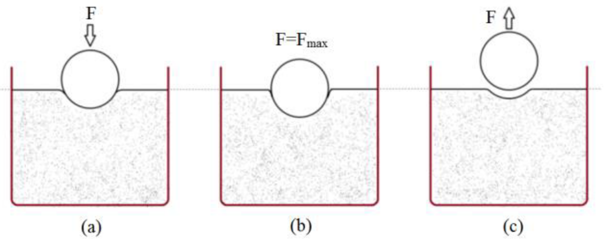

Fig. 1. Indentation step of the ball indentation technique [5].

Flowability is evaluated by calculating hardness, *H*, representing flow stress, from the force-displacement response of the bed. Hardness can be linked to the unconfined yield strength, *Y,* which is derived from shear testing or uniaxial compression tests, via a constraint factor, *C*, which is dependent on particle properties, although cannot yet be determined *a priori*. Shear cells are not consistent and reliable, and are often unable to generate steady-state shear in the region of low stresses, below 1 kPa, therefore a common practice is to extrapolate the yield locus to zero major principal stress, which often leads to an overestimation of unconfined yield strength [4]. On the contrary, ball indentation enables unconfined yield strength at low stresses to be inferred from hardness measurements via *C* at low stresses, providing invaluable data for hopper and silo design.

In the light of the above, this work has been centred on the evaluation of flowability of two different powders at low stresses by using the ball indentation technique to infer their unconfined yield strength.

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2 Materials and methods

2.1 Materials used

For this experimental investigation two different powders were chosen. The first powder was a model material, soda lime glass beads (Sigmund Lindner GmbH), which were sieved to obtain four consecutive single sieve cuts: 53-63, 63-75, 75-90 and 90-106 µm. The beads were made cohesive by applying a commercially available silane coating, known as Sigmacote (Sigma-Aldrich), as conventional glass beads have very low interface energy. The second material that was studied was titanium dioxide (TiO_2), also known as titania. The specific powder sample used was Tiona AT-1 (98.5% Anatase, Cristal Global), a technically pure, dry milled anatase pigment. It is made of smooth, rounded primary particles with a size of roughly 100 nm. The appearance of the powder is white and cohesive, with loose clusters easily forming during powder handling and storage. Titania has the ability to auto-agglomerate, due to possible agitation of the powder bed, such as mechanical vibration [6].

2.2 Methods used

In this work two methods were used to assess powder flowability, ball indentation and shear testing. Shear testing was performed using an automated Schulze Ring Shear Tester. A detailed description of the principles of shear testing can be found elsewhere [1]. From shear testing, the unconfined yield strength, *Y*, of a powder can be determined, which is the stress required to fail a consolidated mass of material to initiate flow. All the shear cell measurements are repeated three times at the same conditions and error bars indicate standard deviation of the measured values.

Ball indentation was investigated using an Instron 5566 mechanical testing machine (Instron Corp., USA). The samples were pre-consolidated to different levels by uniaxial loading in a die by a stainless steel piston using a 10 N load cell, which has a resolution of 0.25 mN. The cylindrical die used here is made of stainless steel and has a height of 20 mm and an internal diameter of 20.5 mm. The loading rate was kept constant at 1 mm/s, therefore testing at quasi-static conditions. The pre-consolidated samples were then subjected to indentation using a high precision glass spherical ball indenter of 3.969 mm diameter, supplied by Sigmund Lindner GmbH. The indenter was fixed to a small loading rod using super glue. Indentations were carried out only at the central zone on each specimen in order to avoid wall friction effect and increase the reliability of the data for different compression levels. The criteria for choosing bed diameter, bed height and indenter's radius are chosen based on the standardisation of the method by Zafar *et al.* and Pasha *et al.* [4, 5].

The flowability is determined by the force-displacement response of the powder bed. The applied load (*F*) and the displacement of the piston/indenter were continuously recorded throughout the process. Hardness, *H*, is given by the ratio of the maximum indentation load, *F*, to the projected area of the impression, *A*:

$$H = \frac{F_{max}}{A} \tag{1}$$

where *A* is the projected area of the impression of the indenter, which can be expressed in terms of the size of the indenter, *d*, and final depth of impression, *h*:

$$A = \pi(dh - h^2) \tag{2}$$

Tabor established a relationship between indentation hardness and yield stress for continuum materials that is represented by the following equation [7]:

$$H = CY \tag{3}$$

where *Y* is the yield stress and *C* is the constraint factor.

The value of C for a given powder is not known *a priori*. Zafar reported values of the constraint factor for various materials, ranging from 1.5-3 for silanised glass beads, to 6-8 for different lactohale particle sizes [4].

In this work all the hardness measurements are repeated five times at the same conditions and error bars indicate standard deviation of the measured values. Also, throughout the experiments of the current research, relative humidity and temperature were recorded and were in the range 15-25°C and 40-65% RH, respectively.

3 Results and discussion

3.1. Shear Testing

The two powders under investigation were subjected to shearing for a range of pre-consolidation stresses. Unconfined yield strength is investigated as a function of major principal stress as presented in Figures 2 and 3 for the two materials.

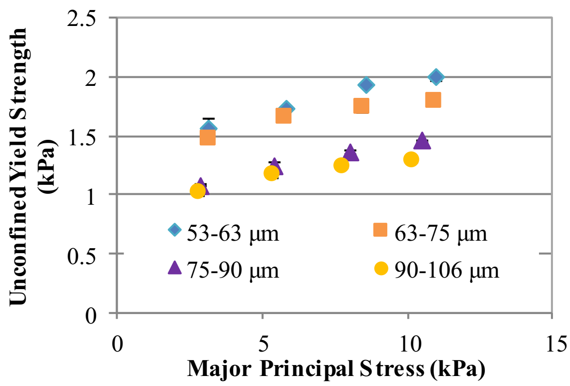

Fig. 2. Shear cell results for silanised glass beads.

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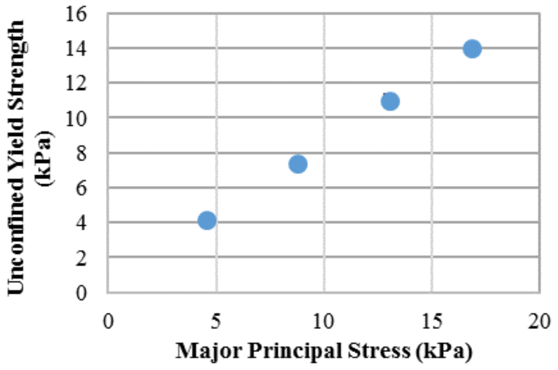

Fig. 3. Shear cell results for titania.

It can be seen from the data obtained from shear testing that the unconfined yield strength increases linearly with the increase in major principal stress due to an increased packing fraction, most notably for titania.

3.2 Ball Indentation

Hardness must be independent of indentation load in order to represent the plastic yield stress, therefore a range of penetrations depths that provide reliable hardness measurements has to be established first for both materials. The depth is presented as dimensionless penetration depth, which can be calculated from the following equation:

$$h_d = \frac{h}{R_i} \quad (4)$$

where R_i is the radius of the indenter.

For silanised glass beads, dimensionless penetration depths of 0.3-0.7 are found to give reliable hardness measurements (Fig. 4). At very low indentation loads/penetration depths, the force on the indenter is due to its interactions with only a few particles, and hence the calculations of the projected area of the impression, and therefore hardness, are inaccurate, since plastic deformation is not initiated.

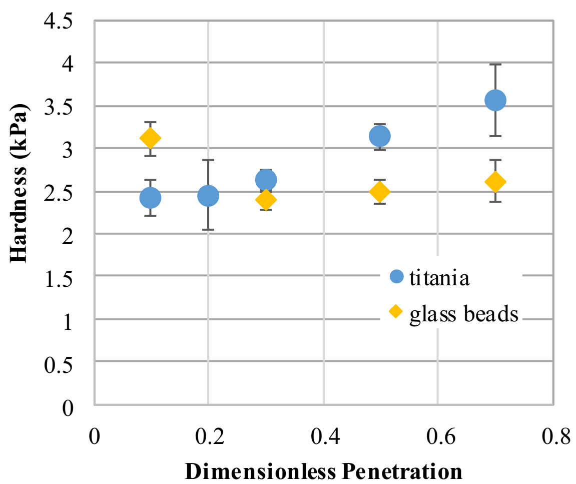

Fig. 4. Hardness vs dimensionless penetration at 1 kPa.

In contrast, for titania, it is observed that dimensionless penetration depths of 0.1-0.3 provide reliable hardness measurements. Titania's particle size is significantly smaller than the glass bead sieve cuts, therefore the indenter contacts a sufficient number of particles to cause plastic deformation and to obtain a reliable hardness measurement a short distance after penetrating the bed.

Now that a range of penetration depths, within which hardness remains stable has been established for both materials, indentation tests at dimensionless penetration depths of 0.2 for titania and 0.5 for silanised glass beads are conducted in a range of stresses with the results presented in Figures 5 and 6, respectively.

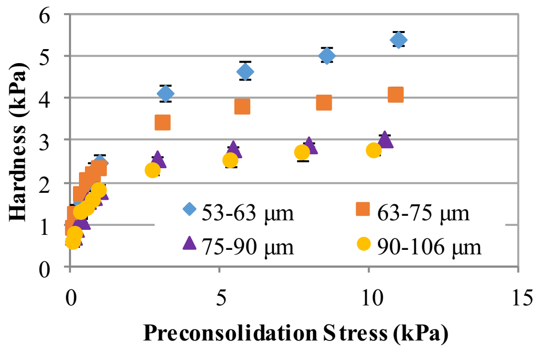

Fig. 5. Ball indentation results for silanised glass beads.

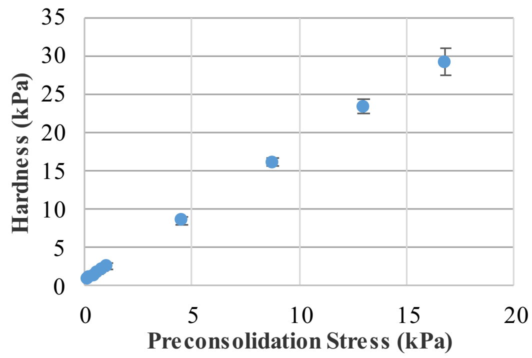

Fig. 6. Ball indentation results for titania.

Hardness is observed to increase with pre-consolidation stress, for both materials tested. Powders which have been pre-consolidated to lower pressure levels have a weaker resistance to plastic deformation at the same penetration depth, due to a decreased packing fraction. For titania, hardness increases linearly with pre-consolidation stress throughout the range of stresses applied. In contrast, for all the single sieve cuts of coated glass beads the increase is much more sharp in the range of low stresses (<1 kPa). It can also be seen that the smaller the particle size, the larger the exhibited hardness. This trend is clear among consecutive sieve cuts at high stresses, whereas at low stresses the difference is clear among alternating single sieve cuts.

3.3 Combined methods

The constraint factor is calculated using Eq. 3, and is found to be almost constant for both materials (Fig. 7). Also, in the case of silanised glass beads, the smallest particles were found to have the greatest value.

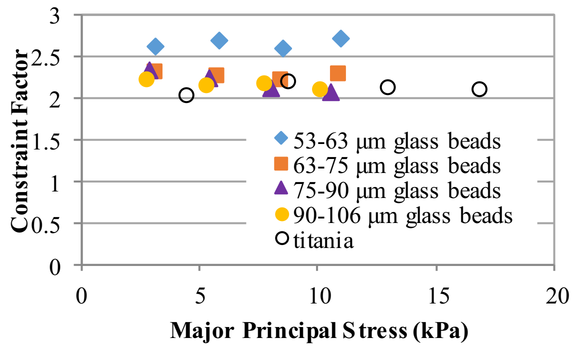

Fig. 7. Constraint factor for silanised glass beads and titania.

In order to calculate the unconfined yield strength at low consolidation pressures, it is hypothesized that the constraint factor remains constant throughout the whole range of stresses. Using the ball indentation hardness values, the unconfined yield strength is calculated at low consolidation levels using Eq. 3. (Figs. 8-9).

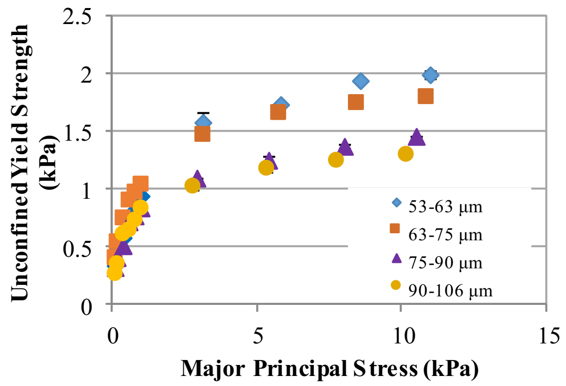

Fig. 8. Unconfined yield strength for silanised glass beads.

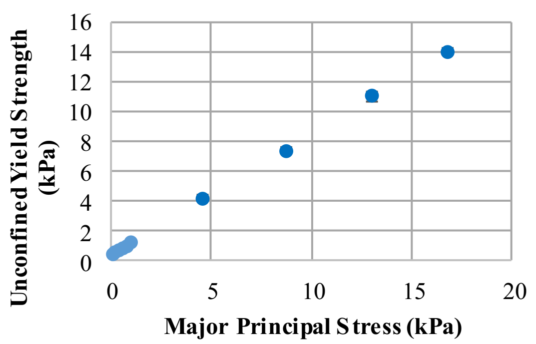

Fig. 9. Unconfined yield strength for titania.

In the case of silanised glass beads, shear tests predicted greater values of unconfined yield strength at low stresses than ball indentation, whereas for titania the extrapolation of the yield locus from shear cell measurements correlates well with the ball indentation findings.

4 Conclusions

Flowablity of titania and four consecutive silanised glass bead sieve cuts was assessed using the ball indentation technique and shear testing. Ball indentation results exhibited a very good reliability of the technique at both high and low stresses. Hardness was found to increase with pre-consolidation stress for both materials, but much more sharply for coated glass beads at low compaction levels. Also, ball indentation provided a very good sensitivity of hardness to particle size; the smaller the particle size, the greater the hardness. The constraint factor was found to be constant throughout the range of stresses for which it was calculated, having different values for different materials. Finally, by hypothesizing that the constraint factor remains constant at low stresses, for which it cannot be determined experimentally, the results of the yield stress inferred from ball indentation measurements showed that in the case of silanised glass beads shear cell extrapolation would result in an overestimation of yield stress. On the contrary, titania's inferred yield stress follows the same trend as the derived from yield locus extrapolation.

Future work will broaden the range of tested materials, as well as utilising the Distinct Element Method (DEM) to investigate the influence of particle properties on constraint factor and examine its variation at low stresses.

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