



Ball indentation on powder beds for assessing powder flowability: Analysis of operation window



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ABSTRACT

The characterisation of bulk behaviour of cohesive powders is very important in processing of particulate solids, e.g. for reliable powder flow out of storage vessels. For filling and dosing of small quantities of powders in capsules and for dispersion in dry powder inhalers, the interest is on the behaviour of loosely-compacted powders in small quantities and under very low applied loads. Furthermore at the early stages of drug development, the quantity of the powder available is often very small and the traditional bulk testing methods are neither possible nor applicable. In this work we evaluate a method to infer powder flowability by ball indentation. This technique provides a measure of flow resistance which can be related to the unconfined yield stress. It can be applied at very low loads and requires only a small sample quantity, typically a few mm³. The operational window in the ball indentation method in terms of minimum sample size, penetration depth and indenter properties (such as size, shape, friction and Young's modulus) has been analysed and reported here.

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1. Introduction

Processing of fine and cohesive powders is difficult and marred by inconsistencies in powder flow, which adversely affect manufacturing reliability and productivity. The flowability issues are often attributed to the cohesive nature of fine powders (typically <100 µm), due to attractive interparticle forces [16]. For example in the case of powder discharge from silos or hoppers, ratholes and arches may be formed, especially in the presence of humid air, resulting in poor flow of the powder. On the other hand, uncontrollable flooding of fine powders can also occur due to aeration.

Consistent and reliable powder flow is critical in a number of industries such as the pharmaceutical industry. For tableting dry powder blends must flow easily into the compression dies in order to obtain a consistent weight and homogeneous product quality. In healthcare technologies dosing of small quantities of cohesive powders is technologically very challenging. For instance, for drug delivery via the lungs the functionality of dry powder inhalers (DPIs) is strongly dependant on the flowability of weakly compacted bulk powders. Also in the nuclear industry, the production of fuel rods relies on precise dosage of powder for compaction. Therefore, it is important to characterise the physical properties relevant to powder flow as a function of consolidation stress. There are several techniques available for assessing the

flow behaviour of powders such as the uniaxial test e.g. Edinburgh Powder Tester [1], shear cells, e.g. Jenike [8] or the Schulze ring shear tester [14]. However, these tests are generally not capable of handling measurements at consolidation stresses much <1 kPa, which are applicable to the above processes. More recently developed techniques for assessing the flow behaviour of powders focus on low stress ranges including SSSpin Tester - based on science of centrifugal force to the measure of unconfined yield strength [9], Sevilla Powder Tester and Raining Bed Method, which measures direct tensile yield stress of the powder [19] and FT4 powder rheometer of Freeman Technology [5]. These tests require relatively large amounts of powder [13], which are highly undesirable for industries such as nuclear and pharmaceutical due to toxicity, cost of drugs and lack of material availability at the early stages of the development.

Hassanpour and Ghadiri [6] introduced a new method for assessing the cohesive bulk powder failure based on indentation hardness measurement carried out on compacted powder beds. They showed that for the indentation test results to be correlated with the common unconfined compression test method, the characterisation of yielding by the material underneath the ball has to be done in the same way as of the indentation of solid materials. The constraint factor, C , is defined as the ratio of indentation hardness, H , to the yield stress, Y , i.e. H/Y . For solid materials C depends on the indenter geometry and elastic modulus of the material [10,15]. For particulate solids, it is expected to be dependent on the single particle properties such as particle shape, roughness and friction coefficient [12]. However, the operational window in terms of ball size, powder quantity and pre-consolidation stress range is yet to be identified.

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In this study, the ball indentation test is carried out on cohesive powder beds of various materials to investigate the effects of powder filling method, indenter size, minimum sample quantity and penetration depth required to ensure a reliable hardness measurement. In addition the effect of indenter Young's modulus and container wall material on the hardness measurement is investigated.

2. Materials and methods

Spherical glass beads with three different sieve cuts (45–63, 75–90 and 90–125 μm) were used as model materials. Glass beads were treated by a silanisation process to make them cohesive, since normal glass-beads are very free flowing [2]. The process of silanisation can be carried out with coatings containing different functional groups, which are capable of bringing about surface chemical modifications. In this work, glass beads were made cohesive by applying a commercially available silane coating, known as Sigmacote®, supplied by Sigma-Aldrich®. Sigmacote is a clear, colourless solution made of the chemical 1,7-Dichloro-1,1,3,3,5,5,7,7-octamethyltetrasiloxane with heptane. The procedure reported by Zafar [18] for silanisation, drying time and temperature was followed. The size distributions of the selected test materials were measured by laser diffraction using the wet dispersion mode of the Malvern Mastersizer 2000. Multiple measurements were taken (10 for each sample) and average particle sizes are given in Table 1.

Ball indentation experiments were carried out using the Instron 5566 mechanical testing machine (Instron Corp., USA). The samples were first poured into a die and pre-consolidated by a stainless steel piston using a 10 N load cell which had a resolution of 0.25 mN. The strain rate was kept constant at 10^{-3} s^{-1} , therefore ensuring quasi-static test conditions prevailed. The pre-consolidated samples were then subjected to indentation using high precision spherical ball indenters supplied by Sigmund Lindner GmbH (Warmensteinach, Germany). The properties of the glass indenters used in this research work are shown in Table 2.

The applied load, F , and the displacement of the indenter, h , were continuously recorded throughout the indentation process. The approach outlined by Hassanpour and Ghadiri [6] was followed for determination of sample hardness based on maximum indentation load, F_{max} , and projected area of the impression after load was removed, A . The hardness of the powder bed is calculated using Eq. (1).

$$H = \frac{F_{\text{max}}}{A} \quad (1)$$

where A is obtained using Eq. (2);

$$A = \pi (d_b h_c - h_c^2) \quad (2)$$

where d_b is the diameter of the indenter and h_c is the plastic depth, determined by the intercept of the tangent to the unloading curve [6,12]. All experiments in this work were repeated three times for each condition and error bars indicate the standard deviation of the measured values. The experiments reported in this study were carried out at 37–50% RH and 17–24 °C.

Table 1
Particle size distributions obtained by wet dispersion (volume basis).

Materials	d_{10} (μm)	d_{50} (μm)	d_{90} (μm)
Glass beads (45–63 μm)	34.6	55.4	87.2
Glass beads (75–90 μm)	60.2	83.2	115.6
Glass beads (90–125 μm)	77.4	101.7	138.0
Durcal 15	1.8	14.7	30.3
Limestone	4.8	7.1	23.8

Table 2
Properties of glass indenter used in this study as given by the manufacturer.

Indenter properties	
Sphericity	>0.99 (ratio width/length ($x_{\text{min}}/x_{\text{max}}$))
Mean bulk density	1.53 kg m^{-3}
Young's modulus	65 GPa
Hardness	>6 GPa
Roughness, R_a	0.08 μm

3. Results and discussion

3.1. Filling method

In addition to powder properties such as particle size, bulk cohesion, shape, density etc., the structure of a bed formed by pouring powder into a container also depends on the stresses due to gravity, external loading and vibration. Furthermore the procedure by which the powder is introduced into the container is also strongly influential. For example the flow behaviour of formulated powders during die filling influences significantly the packing fraction and its uniformity throughout the powder bed. This affects the strength, homogeneity and dosage variations. In this work, the effect of the method of filling of powder in the die on hardness measurement by ball indentation is investigated. Three different die filling techniques were used: (1) tapped method, (2) poured method, and (3) sieved method. The sample powder used in this investigation was silanised glass beads of 45–63 μm sieve cut, as a model material with well-defined shape. The volumetric size distribution of the sample obtained from the Malvern Mastersizer 2000 is given in Table 1. The pre-compression bed height of the material used in this study was kept constant. The test consisted of three stages: (i) initial filling of the sample material into a 20 mm diameter PTFE cylindrical die, (ii) uniaxial compression of the sample to a pre-consolidation pressure of 5 kPa, (iii) ball indentation. The indenter was a spherical glass bead of 1.588 mm diameter. It was driven at a constant speed of 1 mm/min.

In the tapped method, a fixed mass of powder was poured into the die and was tapped for 10 times at certain amplitude manually. In the poured method the powder was poured in to the die from a central zone, thus allowing the sample powder to fill under its natural flow. In the sieved method, the sample material was passed through a sieve directly placed above a funnel on top of the die. The sieve had a mesh opening of approximately five times the mean particle diameter. The particles then fall into a funnel with a discharge opening of 20 mm which is the same opening size as the inlet of the die. The schematic diagrams of all the mentioned methods are shown in Fig. 1. Indentation hardness measurements as a function of pre-consolidation pressure were carried out for all the die filling methods and the results are shown in Fig. 2.

It can be seen that the hardness increases with an increase in the pre-consolidation pressure. Surprisingly, the sieved method gives the largest highest hardness, followed by the tapped, and poured methods. This shows that sieved method yields the most uniform packing on consolidation even at a low pressure, compared to the other two methods. With the sieved method it was observed that the loose cohesively-bonded agglomerates broke on sieving and the particles fell uniformly the die area. In the tapped method, the powder bed experienced extra consolidation due to manual tapping and therefore the flowability assessment would not be representative of the applied pre-consolidation stress. This was observed by Freeman and Fu [3] for tungsten powders, for which the bulk density increased by 16% on tapping, hence showing the undesirable effect of tapping method. Xie and Puri [17] and Härtl and Ooi [7] also highlighted the densification of powder samples upon vertical vibration or tapping of the die.

To explore differences amongst the die filling methods, the apparent structure porosity of the powder beds was observed by extruding the

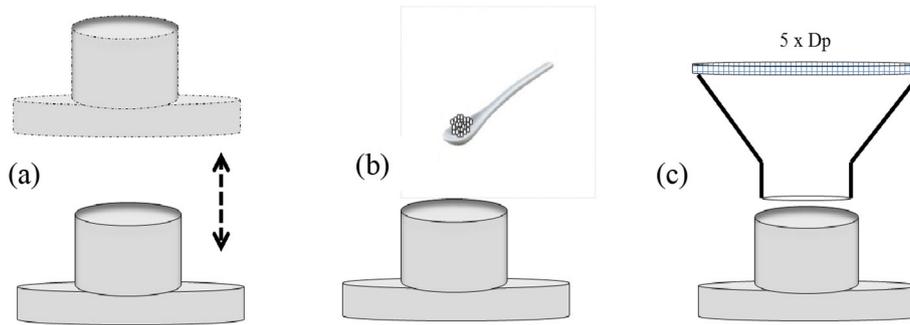


Fig. 1. Schematic diagram of the three filling methods: of (a) tapped, (b) poured, c) sieved.

samples out of the die. The poured filling method gave rise to large cavities or holes. These can be seen in Fig. 3. The uniaxial test on this powder bed gives rise to large fluctuations in load displacement relationship, leading to a lack of repeatability and less packed powder bed and least hardness. These defects have clearly formed during filling, but they have survived the compression stress by cohesive arching and particle-wall friction.

A similar test was performed on a compacted powder bed that was filled using the sieved method. Upon removal of die walls, a well packed powder bed was observed and is shown in Fig. 4. The load-displacement curve was smoother as compared with the poured filling method. Hence this method was used for all the tests for hardness and uniaxial compression measurements. In a series of experiments sieves with three different mesh openings were used to prepare the samples of silanised glass beads. Ball indentation hardness tests were then carried out on the samples using various pre-consolidation pressures. The results are shown in Fig. 5. It can be seen that the influence of mesh openings is relatively small although the trend is clearly discernible.

Clearly the hardness measurement and uniaxial tests carried out on the samples prepared by the sieved method are more consistent. A well-packed structure is obtained even at a low pressure without visible density variations. It is important to know the particle size distribution of the samples, so that an appropriate sieve mesh opening can be selected for sieving to achieve a uniform packing of a powder bed in the die. All the further experiments reported in this study have been carried out using the sieved method with mesh opening of five times the mean particle size of the sample.

3.2. Penetration depth

In the ball indentation method used here the depth is measured as a function of the applied load for both loading and unloading cycles, from which the plastic deformation and elastic recovery are calculated. For continuum solid materials the penetration depth range is well

established in the literature [4]. However for particulate systems, this range has yet to be established. There is a range of penetration depths below which the hardness is unreliable, as insufficient particles have been displaced and consequently the plastic depth is insufficient.

To investigate the effect of penetration depth on hardness measurement by the ball indentation technique, the silanised glass beads of 45–63 μm sieve size were pre-consolidated to 5 kPa and indented using two indenter sizes: 1.1 and 1.6 mm, which correspond to 10.3 and 14.8 times the mean particle size, respectively. The experiments were carried out at indentation loads 5, 7, 9 and 11 mN in order to vary the penetration depth and the results are shown in Fig. 6. The hardness measurement should be independent of the indentation load (or penetration depth) if deformation is plastic. The penetration depth is non-dimensionalised in order to facilitate comparison of different indenter sizes. The dimensionless penetration depth, h_d , is given by Eq. (3);

$$h_d = 2h/d_i \quad (3)$$

where h is the penetration depth, d_i is the diameter of the indenter. Fig. 6 shows the hardness as a function of dimensionless penetration depth, using two different indenter sizes.

A very high value of hardness is obtained at very low indentation loads/penetration depths. This is due to the fact that at small penetration depths the indenter is in contact with only a few particles. This does not provide bulk plastic deformation in the bed. The calculation of the projected area is not reliable in this range since the deformation is not sufficiently large to be accurately approximated by a spherical cap. It should be noted that at low penetration depths a large standard deviation is obtained. This can be attributed to the fact that with a low number of contacts between the particles and the indenter, the rearrangements at the particle level introduce fluctuations. The hardness, however, decreases to a constant stable value for dimensionless penetrations of >0.4 . This shows that the dimensionless penetration at a

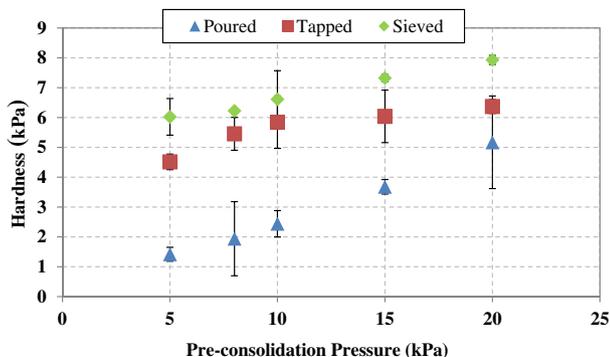


Fig. 2. Hardness as a function of pre-consolidation pressure for different filling methods for 45–63 μm silanised glass beads.

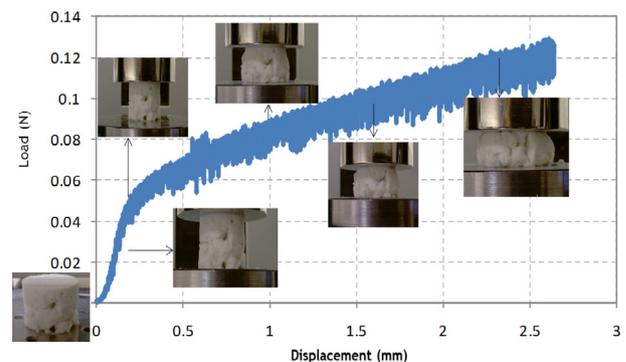


Fig. 3. Load-displacement behaviour of the poured filling sample at 5 kPa during uniaxial compression test.

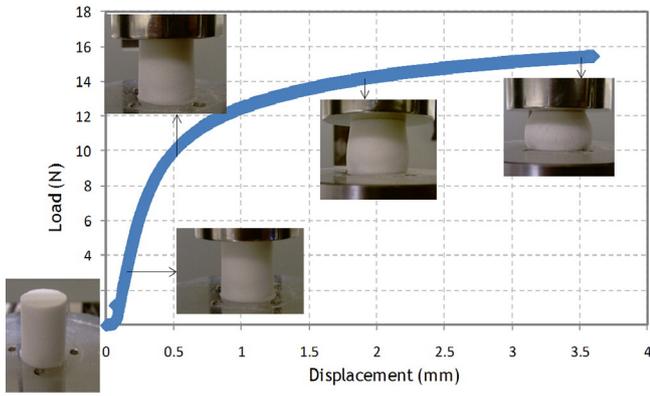


Fig. 4. Uniaxial compression test at 5 kPa with sieved filling method.

given consolidation pressure must exceed $0.4R_i$ to give a reliable measure of hardness.

3.3. Indenter size

To determine the indenter size range which can be used to achieve a reliable hardness measurement, experiments were carried out for two different sieve cuts of silanised glass beads (75–90 μm and 90–125 μm) using a 5 kPa pre-consolidation pressure. The indentation load was kept constant at 7 mN, which provided a dimensionless penetration depth in the range, as previously stipulated. Fig. 7 shows the hardness as a function of normalised indenter size (normalised by d_{50}).

There is clearly a lower limit for indenter size, above which the hardness measurements are reproducible, since the hardness remains relatively constant beyond this point. Below this limit, hardness increases with decreasing indenter size. The use of small indenters leads to local consolidation of the bed, which might result in the observed overestimate of bed hardness. Based on these results, the indenter radius needs to be at least 16 times the mean particle radius in order to cause significant bulk plastic deformation without further consolidation. The maximum limit of indenter size is dependent on the bed height and diameter. Another observation from Fig. 7 is that hardness decreases with particle size. This is expected since smaller particles provide increased number of contacts per unit volume of the bed and hence larger cohesion and reduced flowability.

To quantify the upper limit of indenter size, 75–90 μm silanised glass beads and limestone were used. They were poured separately in to cylindrical dies of 10 mm diameter; one made of PTFE and another made of stainless steel. The results of hardness measurements as a function of indenter diameter (normalised with respect to the bed diameter) are shown in Figs. 8 and 9 for silanised glass beads and limestone respectively. The hardness remains unchanged at low ratios, but it increases at indenter to bed diameter ratios greater than approximately

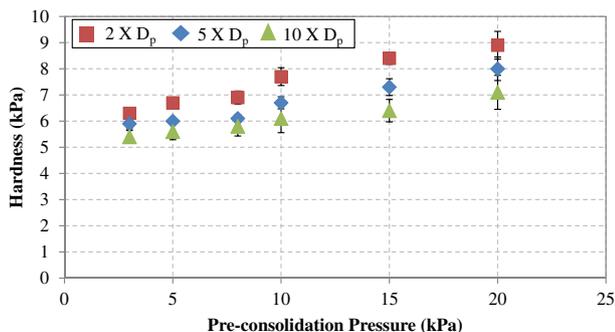


Fig. 5. Hardness measurement as a function of consolidation pressure for samples of silanised glass beads sieved with different mesh openings.

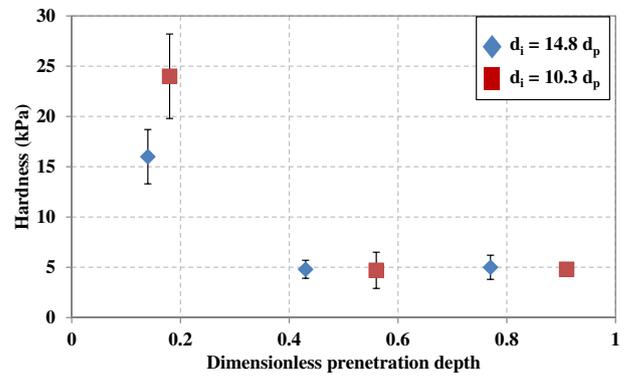


Fig. 6. Relationship between hardness and penetration depth for 45–63 μm silanised glass beads, where d_p is the particle diameter based on d_{50} .

0.65. This is because around this ratio the wall interacts with the plastically deforming zone around the indenter, providing extra constraint [11]. It can also be seen that for a ratio above 0.65, the steel die gives a larger hardness as compared to the PTFE die. This is due to the fact that steel has a greater particle-wall friction than PTFE. It is noteworthy that the ball indentation technique is able to capture this effect of wall friction to a good degree of sensitivity. The same trend was observed in the case of limestone powder as shown in Fig. 9. Therefore, based on these measurements, the indenter size should not exceed $0.65d_b$.

3.4. Bed height

In order to investigate the effect of powder bed height, silanised glass beads (75–90 μm) and limestone were again used. The PTFE dies were filled with varying amounts of materials and pre-consolidated to 5 kPa. The hardness measurements obtained as a function of bed height normalised by mean particle diameter are shown in Figs. 10 and 11, respectively.

At shallow bed heights the hardness is significantly higher than deeper beds for both materials, due to the interaction of the base with the plastic zone. For the silanised glass beads, the hardness is relatively constant for bed heights larger than about 28 times d_{50} . In the case of limestone, the minimum bed height for measuring a constant hardness is around 40 times d_{50} , as shown in Fig. 11. This difference may be due to the effect of shape and the wide size distribution of limestone particles (see Table 1). The limestone sample contains a fraction of coarse particles, forming the bed; therefore the use of d_{50} by volume to represent particle size may not be a good representative measure to be used. In any case, the bed height required for limestone (40 times d_{50}) still corresponds to an extremely low sample mass requirement.

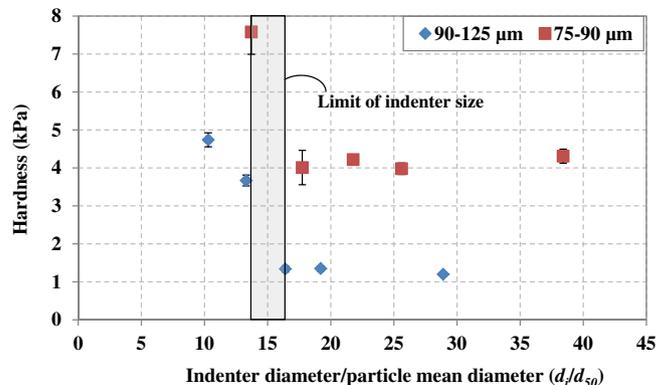


Fig. 7. Relationship between hardness measurement and indenter size at 5 kPa pre-consolidation pressure and 7 mN indentation load, for two sizes of cohesive glass beads.

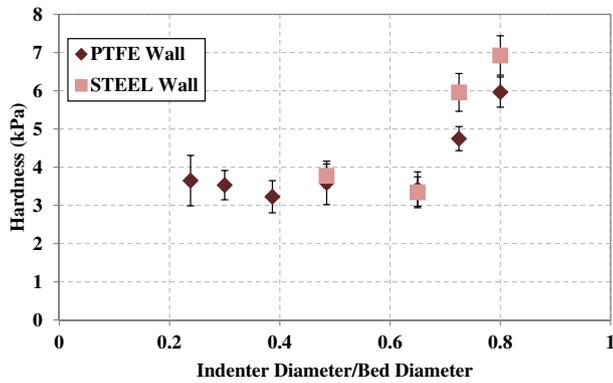


Fig. 8. Hardness measurement of silanised glass beads as a function of indenter diameter at 5 kPa pre-consolidation pressure and 7 mN indentation load.

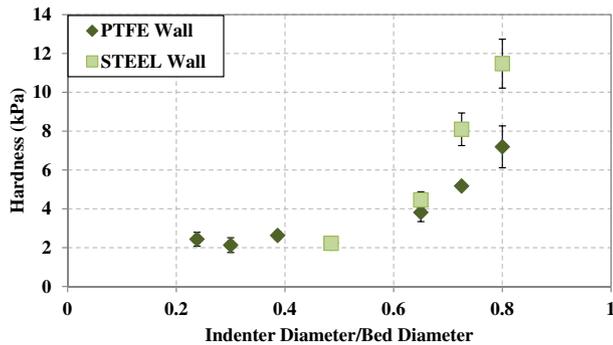


Fig. 9. Hardness measurement of limestone as a function of indenter diameter at 5 kPa pre-consolidation pressure and 7 mN indentation load.

3.5. Indentation position

The effect of indentation position on material hardness was investigated by using a 2.38 mm spherical glass indenter, based on the criteria of indenter size identified above. The indentations were carried out on powder beds of 45–63 μm silanised glass beads, limestone and Durcal 15, pre-consolidated to 10 kPa and indented using a 7 mN load. The results are shown in Fig. 12, where the fractional radial positions 0 and 1 correspond to the centre of the die to the wall boundary, respectively. There is a slight increase in the hardness towards the wall in the case of silanised glass beads and limestone, whereas no change is observed for Durcal. The increased hardness near the wall suggests a higher particle-wall friction as compared to internal angle of friction for silanised glass beads and limestone. It could also suggest exactly the

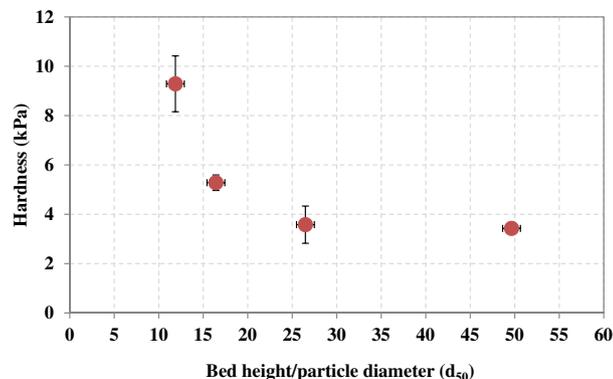


Fig. 10. Hardness variation with bed height for silanised glass beads at 5 kPa pre-consolidation pressure and 7 mN indentation load.

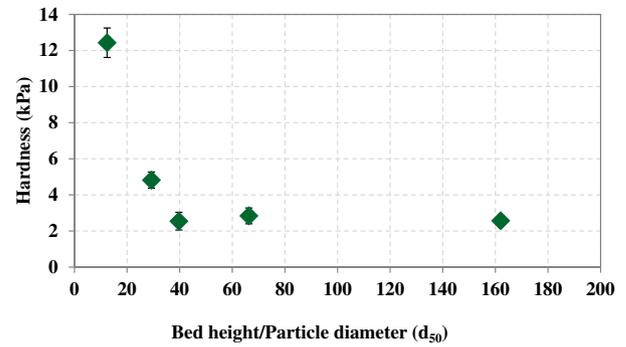


Fig. 11. Hardness variation with bed height for limestone at 5 kPa pre-consolidation pressure and 7 mN indentation load.

opposite, in which case a better compaction is achieved near the wall, and hence higher hardness. So it depends on the die material. Therefore, to be consistent for comparative analysis for further experiments, it is essential that all indents are made near the centre of the compacted powder bed.

To further investigate the capability of performing multiple tests on a sample, experiments were carried out to determine the minimum spacing required between the indents. A sample of silanised glass beads of 45–63 μm was consolidated to 5 kPa and subjected to constant indentation load of 7 mN. A spherical glass indenter of 1.8 mm diameter was used to ensure small separation distances could be achieved, whilst adhering to the minimum indenter size criteria discussed above. The indents were made at different positions ranging from 1 to 3.5 mm spacing, measured by a calibrated gauge. The hardness as a function of the separation distance between the indents, normalised by indenter diameter is shown in Fig. 13. It can be seen that the hardness is significantly higher at low separation distances as compared to large distances, presumably due to the constraint of the plastic zone in the vicinity of the indentation impression, the size of which is dependent on the indenter size. However, the value of hardness seems to reach an asymptote beyond 2.5 mm spacing. Therefore, based on the spacing between the indents, the minimum separation distance required for a reliable hardness measurement is approximately 1.5 times indenter diameter.

3.6. Indenter properties

Spherical indenters made of different materials (steel, glass, Polymer 1, Polymer 2) were used to investigate the effect of indenter Young's modulus. The mechanical properties of the indenters including Young's modulus were determined by nano-indentation using the NanoTest instrument (Micro Materials, Wrexham). The spherical indenters were glued to a stainless steel surface and placed on to the stage of NanoTest. They were indented using a Berkovich pyramid indenter

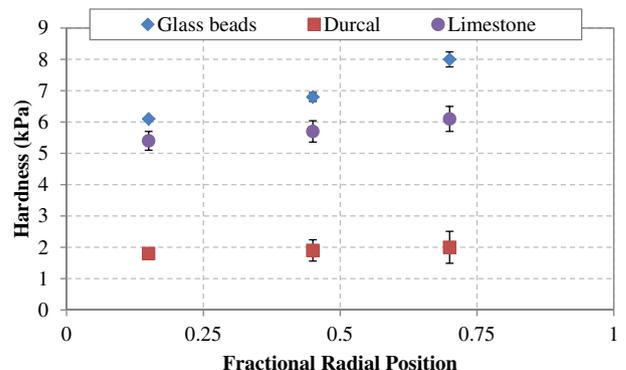


Fig. 12. Hardness comparison for different sample materials as a function of radial position at 10 kPa pre-consolidation pressure and 7 mN indentation load.

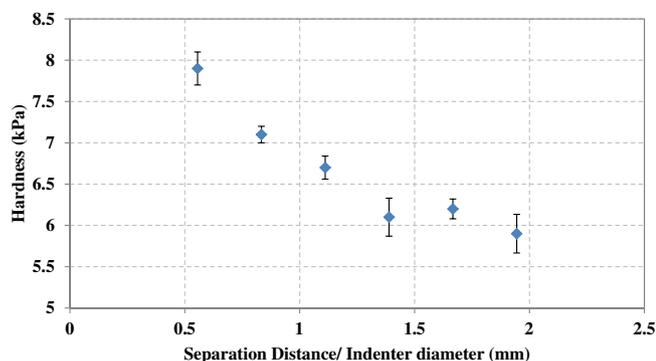


Fig. 13. Hardness measurement for different separation distance normalised by indenter diameter between each indents for 45–63 μm silanised glass beads using 1.8 mm diameter indenter at 5 kPa pre-consolidation pressure and 7 mN indentation load.

with a diamond tip. The measured mechanical properties of the indenters are summarised in Table 3. According to the measurements, the steel indenter has the largest Young's modulus followed by glass, Polymer 1 and Polymer 2, respectively.

The indentation tests with these indenters were carried out on 45–63 μm silanised glass beads using an indentation load of 7 mN. This provided a dimensionless penetration depth >0.4 . The hardness measurements obtained as a function of pre-consolidation pressure for these four types of spherical indenters are shown in Fig. 14. At low pre-consolidation pressures the measured hardness values are all close to each other, with the hardness measured by Polymer 1 indenters being marginally greater than the rest. At pre-consolidation pressures of 15 kPa and beyond, there is a widening of difference in the hardness measurements. In this range the hardness increases with indenter Young's modulus. This was in fact predicted by Pasha et al. [12] from their numerical simulations by DEM for a wide range of indenters of varying Young's moduli. It should be noted that Young's moduli of the tested indenters are significantly greater than that of the powder bed.

4. Conclusions

The ball indentation method for measuring powder flowability has been rigorously assessed in order to determine a standard operating procedure. This analysis included an investigation of die filling method, penetration depth, indenter size, sample quantity, bed height and indenter Young's modulus. Amongst the three die filling methods tests, i.e. tapped, poured and sieved, the latter gives a uniform packing with minimum voids, leading to a higher packing fraction. To have a reliable hardness measurement it is necessary to have a dimensionless penetration depth >0.4 . The minimum indenter size which can be used should be >16 times the mean particle diameter to avoid the influence of individual particles on the hardness measurement and localised consolidation. The upper limit of the indenter size depends on the size of the bed, such that the ratio of indenter to bed diameter should be <0.65 .

One major advantage of the ball indentation technique compared to conventional methods for assessing powder flowability is that a sample size of only a few mm^3 of material is required. Based on the particle size of the sample and parameters which were investigated for the ball

Table 3

Mechanical properties of indenters used for the evaluation of indenter stiffness.

Material	Hardness (GPa)	Young's modulus (GPa)
Steel	3.8–11.2	200–210
Glass	6.3–7.5	78–83
Polymer 1	0.3–0.4	5.5–6.5
Polymer 2	0.2–0.3	5–6.5

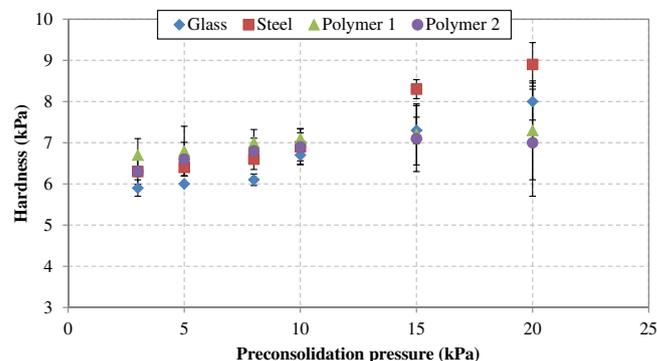


Fig. 14. Hardness measurement of 45–63 μm silanised glass beads as a function of preconsolidation pressure for different spherical indenters using 7 mN indentation load.

Table 4

Minimum indenter and sample size requirement for ball indentation.

Size d_{50} (μm)	Minimum diameter (mm)		Bed height (mm)	Bed volume (mm^3)
	Indenter	Bed		
50	0.85	1.7	2	5
100	1.7	3.4	4	40
200	3.4	6.8	8	300
500	8.5	17	20	4500

indentation technique, the minimum indenter size and sample volume required for a given test can be defined as shown in Table 4. A further advantage of the ball indentation technique is that it enables multiple hardness measurements on the same sample to be made. Multiple indents should be made with a minimum separation distance of approximately 1.5 times the indenter diameter.

Acknowledgments

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