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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 mm^3. 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.

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 and 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).

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

where A is obtained using Eq. (2);

$$A = \pi\left(d_b h_c - {h_c}^2\right) \tag{2}$$

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_{min}/x_{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