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Comparison of three rotational shear cell testers: Powder flowability and bulk density

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ABSTRACT

Developed to aid in the design of hoppers and silos, the shear cell is now frequently used to rank the flowability of powders relative to one another. While standards, such as ASTM D6773 and D6128, exist for shear cell tests, there are still differences between commercially available shear cell testers, such as cell geometry and size. In this work, we used two materials, a free-flowing alumina and a cohesive alumina, to compare measurements from three commercially available rotational shear cells. Results were collected and compared for cohesion, unconfined yield stress, major principal stress, pre-shear stress, flow function coefficient, bulk density, effective angle of internal friction, and the angle of internal friction. ANOVA methods were used to determine the statistical significance and relative size of each of these effects. This work has found that while, as expected, the material type has the largest effect on the shear cell results, the consolidation at which the material was tested and the tester type are also statistically significant effects. These results indicate that care should be taken when comparing the results between different shear cells.

1. Introduction

Powder and granular materials are ubiquitous, appearing in both nature and industrial processes [1,2]. In fact, it has been reported that over 50% of all products manufactured are either in granular form or require the processing of granular materials during their production [3,4]. For example, Ennis et al. estimate that 40% of the value added in the chemical industry is the result of the use of particle technology [5]. The processing of granular materials, like many other processes, is done through a series of unit operations. Some examples of powder-based unit operations in industry are the use of fluidized catalytic reactors in the bulk chemical industry [6], freeze drying during the manufacture of food and pharmaceutical products [7], the compaction of granular materials into tablets in the pharmaceutical industry [8], and the blending of several powders to form cosmetic, personal care, and food products [9].

Despite the prevalence of granular materials, their behavior is poorly understood [10]. The flow properties of powders might change significantly throughout a process; these changes can be detrimental to product quality. The mechanisms of these changes might not be known and so many variables that may influence the manufacturing process are unmonitored [11]. This can manifest as major processing problems that adversely affect product quality. Processing problems are experienced by close to 94% of solid process plants [12]. This is especially visible in the pharmaceutical industry where 80% of products are formulated as tablets, pills, or capsules [11].

The flow of powders in process equipment is a complex and challenging area of study. Companies would like to predict how a given powder would flow in a given piece of process equipment or compare the flow of one powder to another powder. In the pharmaceutical industry, during the initial stages of development, one often has only a small amount available of active pharmaceutical ingredient, so companies would like to measure powder properties using lab scale tests to predict behavior in processing steps like agitated drying and blending [6,9,13–15]. In the catalyst manufacturing industry, new formulations may dictate the use of new powders in existing process equipment for steps like impregnation, drying, mulling, and calcination [16,17]. Again, being able to predict the flow of a powder using lab scale tests is desirable. In many cases an easy way to predict how a new powder may behave in a piece of equipment is to compare how that new powder flows relative to an existing powder that has already been used in that piece of equipment. Companies, therefore, often develop libraries of powder flow properties to compare new powders to existing powders [18].

A unified framework capable of describing powder flow behavior does not exist [19]. Therefore, experimental characterization techniques and empirical correlations must be considered. The capacity of a powder or granular material to flow under a specified set of conditions is referred to as the flowability of the material. This is a complex characteristic, dependent on not only material properties, but also the stress history the material has experienced and the processing equipment used [20]. As a result, many experimental characterization techniques

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have been developed [21]. One of the most common flow characterization techniques is the shear cell.

The shear cell testing methodology was originally developed by A.W. Jenike for the specific application of designing hoppers and silos from the principles of solid state mechanics [22]. Shear cells are now commonly used to rank granular materials according to their flowability [23]. The shear cell can also be used to measure the bulk density of a material as a function of applied normal stress. The bulk density of a material is indicative of a material's flowability and the degree to which the material may expand or consolidate under various conditions occurring during manufacturing [24,25]. The bulk density, defined as the ratio of the mass of powder sample to the volume of that powder sample, takes into account both the particle density as well as the packing of the powder bed [26]. From a series of shear tests, the angle of internal friction, the angle of wall friction, the slope of the hopper walls, and other design parameters can be extracted.

This methodology has since been more generally applied in the field of powder characterization. Powder flowability characterization has become so prevalent that international standards detailing the procedure have been defined [27,28]. Many studies have been conducted involving the shear cell [29,30]. Much of this work has been focused on determining whether shear cell measurements are applicable to specific situations [31–36]. In addition, changes in various aspects of the procedure (ones not dictated by the standards) have been studied [37–39].

However, while a number of different shear cells are available commercially, there is only a limited amount of published work that compares the measurements of a particular shear cell parameter between different types of shear testers. One such study by Pillai et al. compared an on-line wall friction tester and the Jenike wall friction tester. It was found that while some quantitative differences between the yield loci measured by the two testers were observed, each tester gave the same general trends [40]. Schulze has performed a round robin study of RST-XS and RST-01.pc shear cells using limestone CRM-116. Similar results were observed for the two shear cells, 30 and 900 mL in volume [41]. In comparison, our understanding of fluid rheological measurement has progressed to the point that we can readily expect that rheological measurements using a particular rheometric device will agree with those from another rheometric device. However, many unanswered questions remain as to whether the powder flowability measurements performed in one shear cell would agree with measurements of the same powder in a different shear cell. Considering how common it is to measure powder flow properties in shear cells, we believe that it is important to answer these questions.

This paper examines the effect of consolidation stress and tester type on eight responses obtained using the shear cell: cohesion, unconfined yield stress, major principal stress, pre-shear stress, flow function coefficient, bulk density, effective angle of internal friction, and angle of internal friction. Three different shear cells are used. The effect of the shear cell tester (differing in cell size and geometry) and the initial consolidation stress on the results obtained from a shear cell were studied to determine if results from various shear cells measured under a range of experimental conditions can be directly compared. The rest of the paper is organized as follows. The materials characterized and the three shear cell testers studied are described. The results of the eight responses are discussed in the Results and discussion section. The results include statistical analysis used in determining the statistical significance of the material, consolidation stress, and tester factors. It was found that each of these factors was generally statistically significant. Therefore, the results from shear cell tests executed under varying initial consolidation stresses using various shear cells should be compared only with caution.

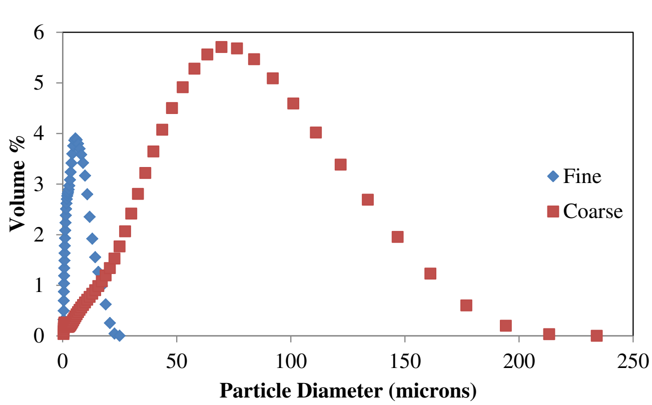

Fig. 1. Particle size distributions of the fine and coarse grade of γ-alumina powder.

2. Materials and methods

2.1. Materials

Two materials, a cohesive and a free-flowing powder, were used in this study. These materials were two grades of γ-alumina supplied by Albemarle (Amsterdam, The Netherlands). The particle size distributions of the powders were measured using a Beckman-Coulter LS 13 320 series laser diffraction particle size analyzer (Pasadena, CA, USA) and are shown in Fig. 1. The coarse grade had a d_{10} of 11 μm, a d_{50} of 59 μm, and a d_{90} of 122 μm. The fine grade had a d_{10} of 1 μm, a d_{50} of 4 μm, and a d_{90} of 11 μm.

2.2. Methods

2.2.1. Procedure

The standard shear cell procedure involves three steps: pre-compaction of the powder bed, pre-shearing of the powder bed until steady state flow is achieved (the bulk density is constant), and shearing until the powder yields. The pre-shearing/shearing process is repeated 4–5 times using normal stresses 20–80% of the consolidation stress. The result of the above process is a yield locus (see Fig. 2). The yield locus is fit with a best-fit line that is extrapolated to the y-axis. This corresponds to the shear stress at zero normal stress, or cohesion, τ_c. The angle that the best-fit line creates with the x-axis is the angle of internal friction.

In addition, Mohr circle analysis is performed on the yield locus. Mohr circle analysis is a geometric representation of a coordinate transformation to identify the principal stresses. Two circles are used. The first goes through the origin and is tangent to the best-fit line through the yield locus. This circle represents the conditions present at the free surface of an arch (as is present in hopper flow) and represents the conditions for critical failure. The second circle is tangent to the yield locus and passes through the pre-shear (steady state flow) point. This circle represents the conditions for the critical state. The principal stresses extracted from this analysis are called the unconfined yield stress, UYS, and the compacting stress (major principal stress, MPS), as shown in Fig. 2. The effective yield locus passes through the origin and is tangent to the greater Mohr circle. The angle that the effective yield locus creates with the x-axis is the effective angle of internal friction.

The yield locus is measured at several consolidation stresses and the unconfined yield stress and compacting stress are extracted from each yield locus. The unconfined yield stress–compacting stress pairs are plotted from each yield locus to give the flow function. The slope of the flow function indicates how well-flowing a powder is.

The bulk density of the powder bed is also measured at each initial consolidation stress. The bulk density of a material is indicative of a material's flowability and the degree to which the material may expand or consolidate under various conditions occurring during manufacturing [24,25]. The bulk density, defined as the ratio of the mass of powder sample to the volume of that powder sample, takes into account both the particle density as well as the packing of the powder bed [26].