



Contents lists available at ScienceDirect

## Chemical Engineering Journal

journal homepage: [www.elsevier.com/locate/cej](http://www.elsevier.com/locate/cej)Chemical  
Engineering  
Journal

## Fluidized-bed measurements of carefully-characterized, mildly-cohesive (Group A) particles

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## HIGHLIGHTS

- High-fidelity fluidized bed experiments are reported for mildly-cohesive glass beads.
- Characterized particle properties include friction, shape and restitution coefficient.
- Surface roughness is measured with AFM and is important to predicting cohesive forces.
- Defluidization experiments (pressure drop and bed density vs velocity) are reported.
- Bubble chord-length, velocity and frequency are presented for various axial and radial positions.

## ARTICLE INFO

## Article history:

Received 8 July 2016

Received in revised form 24 October 2016

Accepted 26 October 2016

Available online xxx

## Keywords:

Multiphase flow

Fluidized bed

Bubbling bed

Cohesion

van der Waals

Validation

## ABSTRACT

Fluidization is sensitive to mild cohesion levels, as evidenced by the different flow behaviors of Geldart Group A and Group B particles. Additionally, cohesion is extremely sensitive to surface roughness. Here, mildly cohesive (Group A) spherical particles were carefully characterized and used in high-fidelity experiments of defluidization, bed expansion, and bubble characterization. The experiments were conducted such that van der Waals forces were the only source of cohesion, and thus the results presented can be used for validating Discrete Element Method (DEM) and/or continuum models. Particle characterization includes surface roughness and adhesion (pull-off) force, which can be used for modeling the van der Waals force. Additionally, the particle friction coefficient, coefficient of restitution, shape (sphericity), and diameter were measured via a suite of simple experiments. This level of particle characterization, which is critical for the validation of cohesive models (DEM and continuum), has not been reported in previous fluidization studies. Accordingly, the characterization experiments employed here are described in sufficient detail for straightforward adaptation elsewhere.

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### 1. Introduction

Unit operations involving solid particle flows (fluidized beds, hopper flow, pneumatic transport lines, etc.) are characterized by complex hydrodynamics such as bubbling, clustering, and species segregation. Modeling frameworks such as the two-fluid (continuum) approach and Discrete Element Method (DEM) can increase the understanding of macro-scale behavior, which is linked to micro-scale (particle) properties [1]. For instance, DEM simulations have shown [2] particle-particle cohesion to be critical to homogeneous bed expansion of mildly cohesive (Geldart Group A) particles. Also, past studies have found other particle properties, e.g., friction

coefficient [3], coefficient of restitution [4], and roughness [5], significantly affect the behavior of many-particle systems. A significant amount of recent DEM and experimental work has been targeted at understanding the effects of such properties on, for instance, fluidization behavior [2,6–16]. Nonetheless, although a large body of experimental data exists using cohesive and non-cohesive particles (e.g. Refs [17–32]), many of these studies do not report or include direct measurements of the particle properties necessary for a robust validation of models [17]. Accordingly, a detailed, comprehensive data set of fluidization behavior with carefully characterized particles is necessary, especially for mildly cohesive particles, which are particularly sensitive to micro-scale properties like roughness [5,6].

Numerous past works have demonstrated the important impact of particle properties on the fluidization behavior of non-cohesive systems. For example, for different types of glass (soda lime,

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borosilicate, etc.), tabulated values of kinetic (sliding) friction coefficient span 0.1 to 0.3 [24,33], and the defluidization curve (pressure drop vs. gas velocity) is known to change within this range [33]. Furthermore, several groups have found that the coefficient of restitution significantly affects bubble properties [4,34,35], but it negligibly affects the defluidization curve [33]. It was also found that accounting for even a slight non-sphericity (sphericity > 0.92) is necessary to predict defluidization accurately with DEM [33]. Additionally, previous works have indicated that particle size [36] and the width of the particle size distribution [37] impact bubble properties. Collectively, these studies point to the importance of particle properties on fluidized bed behavior and the corresponding need for carefully characterized particles.

In addition to the particle properties mentioned above [16,32,36], mildly cohesive (Group A) particles are also affected by other particle and system properties that are generally negligible for non-cohesive particles. The mild cohesion associated with Group A particles is attributed to the van der Waals force between particles [38], which is extremely sensitive to surface roughness; namely, the van der Waals cohesion is proportional to the square of the separation distance [39]. This sensitivity has been observed both in micro-scale (single-particle) [40] and macro-scale (many-particle) experiments [7,10,41]. Furthermore, recent work [6] indicated that correctly accounting for surface roughness is critical to accurate defluidization predictions. Even though the dependence of cohesive particle behavior on surface roughness is well-documented, surface roughness measurements are rarely reported for many-particle experiments or reported in a form that is not sufficient for inputs into simulations. As an example of the latter, earlier studies provided scanning electron micrograph (SEM) images of their particles [7,42–44]. Such images provide high-resolution information about surface roughness in the lateral direction, but only qualitative information normal to the surface (since it is a two-dimensional projection of the surface) [45]; it is the normal direction that is critical to predicting van der Waals forces since it dictates the minimum separation distance between particles.

To account for the effects of surface roughness in cohesive models for DEM and continuum frameworks, roughness must be measured accurately. Atomic Force Microscopy (AFM) can be used to quantify surface via topographical maps with nanoscale resolution [46]. The links between surface roughness and adhesion forces have been investigated in several studies, e.g. [47–51]. Two approaches are available to incorporate such roughness measurements into models for van der Waals forces: (i) analytical solution of single-equation theories that incorporate roughness (e.g. the Hamaker theory adjusted to account for surface roughness [40,47,52–55]), and (ii) computational methods that sum the van der Waals force between adjacent points on discretized meshes that reproduce the measured surface roughness [56–60]. Regarding the former, a method was recently developed to extract roughness parameters from the measured particle topography, without resorting to visual or arbitrary means, that can be used as input to the analytical theory [47].

The main aim of this work is to provide high-fidelity experimental data that can be used for validating DEM and/or continuum models that incorporate van der Waals forces. To achieve this aim, a twofold approach is implemented: (i) careful characterization of particles that are mildly cohesive (microscopic), and (ii) corresponding fluidized-bed measurements (macroscopic). Particles are characterized by measuring properties such as coefficient of friction, shape, coefficient of restitution, and surface roughness using straightforward techniques. Pressure drop and bed expansion are reported with increasing and decreasing gas velocity (fluidization and defluidization). Furthermore, bubble size, frequency and velocity are presented for particles with increasing cohesion at various gas velocities and axial and radial positions.

## 2. Materials and experimental methods

### 2.1. Particles

Soda-lime glass spheres from Mo-Sci Corp were used in this work. To ensure the particles are close to monodisperse, they were sieved to the smallest size range possible using standard sieves. Specifically, the three size ranges implemented here are 20–38  $\mu\text{m}$ , 45–53  $\mu\text{m}$ , and 63–75  $\mu\text{m}$ . Fig. 1 shows that the particles are relatively monodisperse (narrow particle size distribution) and nearly spherical, making them close to ideal for validating DEM and continuum theories. Table 1 provides the particle properties specific to each size range, namely, the mean diameter ( $d_p$ ) and particle shape (sphericity,  $\phi$ ); the technique used to measure the sphericity will be detailed in the following section.

#### 2.1.1. Particle characterization: sphericity, restitution coefficient, and friction coefficients

Particle size and material density ( $\rho_p$ ) are the most commonly reported material parameters in past works, likely because they are reported by manufacturers and/or straightforward to measure (e.g. sieve analysis for determining particle size). For this work, simple, and relatively inexpensive methods were developed to measure the other relevant particle properties (shape (sphericity)  $\phi$ , restitution coefficient  $e$ , static friction coefficient  $\mu_s$ , and kinetic friction coefficient  $\mu_k$ ) with the intent of making these techniques easily adoptable in most labs. As detailed below, the coefficients of friction and restitution were measured using a DVC (model 340 M) high-speed camera, and the acquisition rate used varied between 100 and 300 frames per second (fps) based on the requirement of the system (i.e. faster particle velocities required faster frame rates) and the amount of light available in a given experiment. The image resolution was generally 320 pixels x 240 pixels. This high-speed camera is the most expensive piece of equipment for this suite of measurements; however, many current point-and-shoot cameras offer comparable high-speed video acquisition with frame rates of around 250 fps. In order to eliminate cohesion in the restitution and friction measurements, tests were performed using 2 mm diameter soda-lime glass spheres from the same distributor as those used in the fluidized-bed experiments.

Fig. 2 provides images that overview these straightforward characterization techniques. As pictured in Fig. 2a–c, the coefficient of restitution is measured using a laboratory ring stand with several clamps used to hold a pair of tweezers. The tweezers clamp onto a particle (a 2 mm glass bead is held by the end of the tweezers in Fig. 2a), which uses a spring-based clamp to hold the particle, allowing the tweezers to open wider than the particle when unclamped. The particle motion is recorded with the high-speed camera to obtain the height when released,  $h_o$  (Fig. 2a) and the maximum height after rebound,  $h_f$  (Fig. 2c) using a ruler in the frame of the image. The restitution coefficient is then determined according to  $e = \sqrt{h_f/h_o}$ . To ensure an accurate measurement of  $e$ , only normal particle-wall collisions with no particle rotation can be considered. Accordingly, if the particle bounces at an oblique angle, the trial is rejected as it indicates significant spin was imparted on the particle during release; usually this behavior is associated with incorrectly releasing the tweezers.

Fig. 2d shows the method to measure particle shape, or sphericity, using images taken with an optical microscope, which is the particle projected area diameter divided by the projected perimeter diameter [33,61].

The particle-particle and particle-wall coefficient of friction measurements are described in detail elsewhere [62], with an image of the apparatus provided in Fig. 2e. To measure the kinetic (sliding) friction coefficient, a sled is made of particles glued to a

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