Powder Technology 211 (2011) 189–198

Contents lists available at ScienceDirect

Powder Technology

journal homepage: www.elsevier.com/locate/powtec

Density behavior of cohesive granular materials

Rafael Mendez [a], Francis S. Romanski [b], M. Silvina Tomassone [b,*]

[a] Department of Chemical Engineering, University of Puerto Rico at Mayaguez, Puerto Rico
[b] Department of Chemical and Biochemical Engineering, Rutgers University, 98 Brett Road, Piscataway, NJ 08854, 98 Brett Rd. C-118 Piscataway NJ, 08854, United States

ARTICLE INFO

Article history:
Received 15 June 2010
Received in revised form 22 September 2010
Accepted 21 November 2010
Available online 3 December 2010

Keywords:
Particle technology
Powder flow
Bulk Density
Tap Density

ABSTRACT

A new experimental methodology for the characterization of density in a powder bed utilizing X-ray micro-computerized tomography (micro-CT) was developed to quantify the density fluctuations in three common pharmaceutical powders (α-Lactose Monohydrate, Lactose 310, and Avicel 102). The method begins by filling an acrylic cylinder with powder and subsequently subjecting the system to vibrations using a mechanical shaker while monitoring the density at predetermined bed heights. Three key parameters were isolated including frequency, amplitude, and number of strokes. It was found that the three powders exhibited different packing rates and final states. It was also found that the density increased in the powder bed as a function of the number of taps, frequency, and amplitude. Additionally, a more uniform density profile was achieved by utilizing higher amplitudes. The cohesive properties of the three powders were investigated using the FT4 powder rheometer and correlated with the results found with the micro-CT scanner. It was found that changes in density were more significant in less cohesive powders, such as Avicel. As powders increase in cohesion, it was found that more mechanical energy was required to alter the agglomerated powder bed. Additionally, the density at the top of the powder bed was significantly more dense than that at the bottom for Avicel, however, the results were directly opposite for the other more cohesive powders. The results have indicated that micro-CT may be used as a more comprehensive and higher resolution technique for analyzing the density of powders and provide a unique insight to packing at different powder bed heights.

Published by Elsevier B.V.

1. Introduction

The mechanical properties of granular systems are of great importance to many chemical industries including petrochemical, specialty chemical, food sciences, catalysis, and most abundantly, the pharmaceutical industry [1]. Intrinsic properties, such as cohesion and density have a direct impact on the performance, flowability, and blending processes commonly used in powder manufacturing. However, cohesion and density are neither simple, nor easy to quantify. Despite the tremendous relevance and wide applicability, the flow properties of powders are not well understood [1,2]. Unfortunately, the behavior of granular systems, in particular cohesive granular systems, is intrinsically more complex than the flow of fluid media. Complexities such as yield stress, failure surfaces, the coexistence of multiple constitutive regimes, and a density hysteresis all add to the complex nature of granular systems. Furthermore, as a direct result of the complex behavior of granular systems, the experimental methodologies used to observe the systems are significantly more rudimentary than those used for fluid media. In addition, the computational work in granular system is also in relative infancy when compared to fluid mechanics [3]. Therefore, there exists a significant gap in the ability to predict not only the intrinsic properties, such as density and cohesion, but the overall flowability and behavior of granular systems.

In general, there are two accepted classifications for characterizing the flowability of powders. Direct methods characterize the granular system in a consolidated state, such as in a shear cell, while indirect methods characterize powder in a flowing, loosely packed state, such as the measure of the angle of repose [2,4–6]. Additionally, the packing behavior of powders is typically represented using densities and density ratios and is generally used as an additional classification for the behavior of granular systems. For example, both the bulk and tap density of powders are individually useful, however, they become much more useful when combined to form flow indices such as the Hausner ratio or the Carr index [7]. An example of packing behavior is exhibited in the Hausner ratio (HR), which simply is the ratio of the tap density to the bulk density of the powder. Generally, if the Hausner ratio is high, the granular system is assumed to exhibit poor flowability [8]. While the Hausner ratio and Carr index are widely used both academically and industrially due to the relative ease of analysis, they are not easily validated and are subject to much debate [5].

In contrast to traditional methods to estimate the tap density, which rely on measuring the volume and mass of a sample, a novel methodology utilizing an X-ray CT scanner is proposed. In an X-ray micro-CT scanner, X-rays are used to create a cross section image by

* Corresponding author. Tel.: +1 732 445 4949.
E-mail address: silvina@soemail.rutgers.edu (M.S. Tomassone).

0032-5910/$ – see front matter. Published by Elsevier B.V.
doi:10.1016/j.powtec.2010.11.024

the projection of a thin-beam X-ray through an object, in this case, the powder bed. The X-rays are then absorbed, scattered, and transmitted. Those that are transmitted through the object at each predetermined angle are subsequently measured and retained as attenuation data. Specifically, this measured attenuation data is a measure of the reduction in X-ray intensity that results from the absorption and scattering caused by the granular system. Subsequently, the collected data is summed over the various angles and reconstructed computationally, thus building a CT image that represents a cross section of the granular system. Differences in density are seen as a grey-scale contrast. Since the local rate of attenuation is directly proportional to the mass density along the given path, the output results are a representation of the density of the granular system. Experimentally, this system can now be subjected to various forces, i.e. "tapping" and the density recalculated, eventually giving a representative profile of the density as a function of the number of taps, the amplitude, and the frequency of taps. This method of using micro-CT has been used previously to establish density profiles for many powder systems, most notably, glass spheres, metals, salts, and ceramics [9–12]. Unfortunately, this method has seen limited use in the characterization of pharmaceutical powders [13], of which many are extremely cohesive; in fact, the most use of X-ray tomography has been in the analysis of tablets and pharmaceutical ribbons [13–15].

This work focuses on the development of an experimental method for the characterization and understanding of density and density fluctuations in granular beds as a function of cohesive powder properties. In particular, the use of X-ray micro-CT scanning was implemented and used to study three widely used pharmaceutical powders with varied, well established, cohesive properties (α-lactose monohydrate, lactose monohydrate 310, and Avicel 102) [3,16]. The effect of various forms of mechanical energy through the variation of frequency, amplitude, and number of impacts on the density profiles of the powders were established and compared with those obtained through conventional methodologies. The remainder of this paper is organized as follows: Section 2 describes in detail the methodology used to incorporate X-ray CT-scanning to study the density of a pharmaceutical granular system. Section 3 exhibits the results and includes a detailed discussion. Section 4 contains the conclusions of the study and areas where the technique can be further explored.

2. Materials and methods

2.1. Materials

α-Lactose Monohydrate (Lacto Chem., later referred to as alpha lactose), Lactose Monohydrate 310 N.F. (regular lactose, Foremost Farm), and microcrystalline cellulose (Avicel 102; FMC) were used in this study. Each was obtained from Fisher Scientific (USA), and used as received. Powders were kept in the same location and at a constant temperature and relative humidity. The particle size distributions of the three powders, as measured using the Beckman-Coulter LS 13-320 Laser Diffraction powder module are shown in Fig. 1. Selected relevant physical properties of the three excipients are shown in Table 1; the cohesion parameter and bulk density data were established using the FT4 Powder Rheometer.

2.2. Equipment summary

Particle size analysis was performed using the Beckman-Coulter LS 13 320 Laser diffraction particle size analyzer (Beckman-Coulter, Brea, CA). Bulk density and cohesion data were obtained using the FT4 Powder Rheometer (Freeman Technology, Worcestershire, UK). The mechanical shaker was constructed using the following components: Function generator from Global Specialties 105-2001 (Wallingford, CT), a Techron 5515 amplifier (AE Techron, Elkhart, IN), a 110 Volt, 1/14 Amp power supply amplifier, a PCB 48712 10–10–100 mV/G accelerometer (PCB Group, Inc, Depew, NY), and a Vibration Test Systems VG-100-6 shaker (Vibration Test Systems, Aurora, OH). The transparent acrylic cylinder containing the granular material to be tested was constructed with a 3.8 cm internal diameter and a 3 cm fill height. An image of the setup is shown in Fig. 2. For operation, a signal of step input was given to the shaker, which was amplified by the amplifier. A step signal was used to resemble a "tap" as close as possible. A mounted piezoelectric device on the base of the shaker was connected to an accelerometer recording acceleration, displacement, and velocity movement in the shaker. The SkyScan 1172 high-resolution micro-CT system was used for X-ray tomography (SkyScan, Kontich, Belgium).

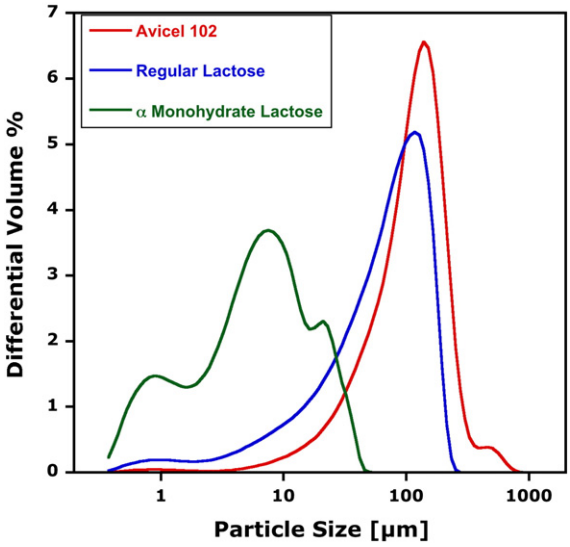

Fig. 1. Particle size distribution.

2.3. Experimental procedure

Initially, the acrylic cylinder was filled to roughly 80% of its total volume. It is important to note that the powder was always poured from an identical height to minimize pouring effects. After filling, the sample was weighed and recorded. The initial state was scanned using the SkyScan 1172 micro-CT. Following the initial scan, the powder was shaken using the mechanical shaker varied by number of strokes (or "taps"), frequency, and amplitude. Three frequencies were used: 2 Hz, 4 Hz, and 8 Hz, as well as, two distinct amplitudes 4 a.u. (3 mm) and 5 a.u. (6 mm). The samples were carefully removed from the shaker and analyzed in the micro-CT after 30, 120, 250, 500, 750, 1000, 2000, 4300, and 8000 taps.

The SkyScan 1172 used for scanning was optimized for the appropriate resolution by using a pixel size of 28″ in a 1000×500 pixel mode with a close proximity, filter-less camera. The reconstruction of the images was conducted at a grey-scale threshold from 0 to 0.6; the beam

Table 1
Key properties of pharmaceutical excipients.

Material	Conditioned bulk density[a] (g/cm^3)	Cohesion parameter (kPa)	Mean particle size (μm)
Avicel 102	0.381	0.110	132
Regular lactose	0.393	0.384	81
Alpha lactose	0.629	0.774	9.1

[a] The conditioned bulk density (CBD) was measured by the FT-4 Rheometer after the pre-treatment. In the pre-treatment, a 45° angle impeller blade was introduced into the powder column in clockwise direction and moving out in the opposite direction three times. The measure cylinder is a split vessel assembly designed to allow a precise volume of powder to be sampled, by rotating the upper part of the vessel away from the lower part. The powder remaining in the lower vessel can be weighed using the FT4 in-built balance and the conditioned bulk density can be calculated.

Download English Version:

https://daneshyari.com/en/article/237489

Download Persian Version:

https://daneshyari.com/article/237489

Daneshyari.com