Powder Technology 253 (2014) 406–423

Contents lists available at ScienceDirect

Powder Technology

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

Characterization of flow properties of cohesive powders: A comparative study of traditional and new testing methods

M. Leturia [a,b], M. Benali [a], S. Lagarde [b], I. Ronga [b], K. Saleh [a,*]

[a] *Compiègne University of Technology, Chemical Engineering Department, Labo. TIMR-EA 4297, 60200 Compiègne, France*
[b] *AREVA–CEZUS Research Center, 291 Route de l'Electro-Chimie, 38560 Jarrie, France*

ARTICLE INFO

Article history:
Received 19 March 2013
Received in revised form 27 November 2013
Accepted 30 November 2013
Available online 12 December 2013

Keywords:
Fluidization
Flowability
Flow additive
Powder rheometer
Segregation
Self-agglomeration

ABSTRACT

The characterization of powder flow properties is often required for reliable design and proper operation of industrial processes. The effect of the state of compaction and bed voidage on bulk solids flowability is probably the most critical area of understanding. The goal of the present study is to compare traditional characterization techniques with methodologies provided by the FT4 Powder Rheometer (Freeman Technology). The data from six different methods, covering low to high stress levels, were compared to examine the hypothetical relationships between them. These techniques were also evaluated with regard to their ability to discriminate between different powders. To make a comparison of the testing methods, a range of seven materials was selected to cover the entire range of fine powders, i.e., from nanoparticles to group B powders.

The results showed that the characterization techniques clearly have different working ranges depending on the level of cohesiveness of the powder. The powder rheometer was found to allow quick and reproducible measurements of the powder response to various environments. The different blade testing methods provided data that were in good agreement with traditional characterization techniques. However, the powder rheometer measurements were difficult to interpret because they depend on many physical properties and environmental parameters. They were particularly useful to compare similar materials but did not allow good discrimination between very different materials. A more detailed understanding of the physical phenomena involved in blade testing techniques is still needed.

Finally, this study showed that powders and bulk materials cannot be viewed as invariant entities. Their flow properties cannot be predicted by only one indicator. The connection of several characterization methods is required to ensure a complete understanding of the powder flow properties over a wide range of conditions. This approach allows better insight into the powder/process relationship.

1. Introduction

It is estimated that approximately 60% of all products manufactured in the chemical industries in Europe are powders and an additional 20% of products use bulk solids in the processes [1]. Recently, there has been a general increase of interest in fine powders and nanoparticles because of their high surface area-to-volume ratio and other special characteristics that make them very attractive in the industries of advanced materials, foods, cosmetics, pharmaceuticals, plastics, paints, detergents, catalysts, coating, powder metallurgy, etc. [2–4]. Nevertheless, the ability of fine powders to flow and fluidize is complicated by the presence of interparticle forces (Van der Waals, electrostatic or capillary forces) which become preponderant compared with gravitational and/or hydrodynamic forces [2,5,6]. It should be noted that the term “nanoparticles” is generally used to refer to primary particles having a diameter smaller than 100 nm [7]. According to Geldart's classification [8], the group C powders correspond to particles less than 20 μm to about 80 μm depending on the density difference between the particle and the fluid.

The processing of many granular materials generally involves different unit operations such as aeration, fluidization, pneumatic conveying, blending, grinding, compaction and storage in bins or hoppers. The reliable flow of powders is an important issue since it can affect the final product quality and the efficiency of the processes. Poor flow leads to wastage, machinery maintenance problems and downtime, with associated costs [9]. As a consequence, the characterization of flow properties of powders is often required for reliable design and consistent operation of processes. One of the main difficulties is that the link between the physics of local grain interactions and their global mechanical behavior is still poorly understood, and thus empirical approaches prevail [10]. Several measuring techniques for the evaluation of the flow properties of bulk solids have been traditionally used: shear testers, uniaxial compression test, flow through a funnel, angle of repose, avalanching, consolidation test, determination of tapped density (Carr index, Hausner Ratio), fluidization test, bed collapse test, etc. [1,11]. Some of these methods yield quantitative results that can be used for equipment design (silos, hoppers, bins, fluidized beds, etc.). Other test results are used for quality control,

---

[*] Corresponding author. Tel.: +33 344 23 52 74; fax: +33 344 23 19 80.
E-mail address: khashayar.saleh@utc.fr (K. Saleh).

0032-5910/$ – see front matter © 2013 Elsevier B.V. All rights reserved.
http://dx.doi.org/10.1016/j.powtec.2013.11.045

comparison and ranking of different bulk solids. In the case of fine particles, tests are often applied to assess the degree of cohesiveness of different powders. These characterization techniques have numerous limitations including reproducibility, predictability, sensitivity and are often time-consuming and user-dependant. Hence, scattered results can be obtained, especially in the case of fine powders. The correlation of results between different empirical tests is lacking or insufficient. However, there is an increased interest in establishing some method comparisons (e.g. [12–17]).

The behavior of powders generally depends on three general features [18]:

- the intrinsic physical properties such as particle size, density, shape, roughness and porosity;
- the bulk powder properties such as size distribution, bulk density, distribution of forces as well as cohesive and frictional interactions;
- the external conditions or processing environment such as temperature and humidity as well as bed voidage and state of compaction.

Thus, it appears that powders and bulk materials cannot be viewed as invariant entities [19]. Since flowability is not an inherent property of the material, a single characteristic or index will not enable a complete understanding of the powder flow behavior [20]. In industrial processes, powders are submitted to a wide range of conditions that may affect their flow properties, from the highly dispersed state in fluidized beds to the highly consolidated state in roller compactors, as shown in Fig. 1 [19]. Individually, the available test methods do not represent all the conditions that powders undergo in their manufacture and application. As a consequence, a range of characterization methods is required to ensure a complete understanding of the behavior of a given powder in different unit operations of an industrial process. This approach, which consists in combining results from different tests, allows a better insight into the powder/process relationship. It leads to a more accurate prediction of the powder flow behavior as well as an improved understanding and efficiency of the processes.

The effect that bed voidage has on powder flow properties is probably the most critical area of understanding. Typically, a consolidated powder needs around 100 times more energy to make it flow than is required when the same powder is aerated [21]. The bed voidage may also impact on the ability to make reproducible measurements. Conditioning is essential to eliminate any packing history such as pre-consolidation or excess air and thus obtain repeatable data. Recently, Freeman Technology has developed a powder rheometer which includes various dynamic characterization methods. It allows measurement of the powder response to various environments, thus simulating the range of processing conditions more closely. From the measurement of the energy required to displace a sample of settled powder with a specifically designed blade, a series of indexes related to the flow properties of powders can be derived. In order to ensure repeatable and comparable data, a conditioning procedure allows the generation of a stable consolidation state that can be reproduced easily. In addition, the possibility to fully automate the testing procedure also minimizes the operator dependency and the time consumption.

The goal of the present study is to compare traditional testing techniques with methodologies provided by the FT4 Powder Rheometer (Freeman Technology). The data from different experimental techniques were evaluated to establish the relationship between the characterization tests. To make a comparison of these methods, a range of five materials was selected to cover the entire range of fine powders, i.e., from nanoparticles to group B powders. In addition, two binary mixtures were used in order to assess the ability of tests to discriminate between different mixing qualities. The main variable was chosen to be the stress level since the bed voidage is one of the most important factors affecting flowability. The flow properties of the powders were assessed using six different methods that can be classified into three groups, corresponding to different stress levels (Fig. 1):

- packed bed conditions: these tests are used to predict the flow (or no flow) of bulk solids from a storage vessel with a given outlet size. The flow properties are measured over a range of controlled stresses, from the stress generated by a few centimeters depth of powder to that generated by several meters depth of powder. It should be noted that the lowest stress may overlap with the tests below (free surface conditions). In this paper, traditional shear cell measurements were compared to a consolidation test provided by the FT4 Powder Rheometer;
- free surface conditions: these tests are representative of the filling of bulk solids into a small packing container. The flow properties are measured at low but uncontrolled and unknown stresses. Test stresses will be different between materials due to differences in their bulk density. Here, Hausner Ratio measurements were compared to a dynamic blade testing (FT4 Powder Rheometer);
- aerated conditions: these tests are representative of the fluidization behavior of bulk solids. Typically, the fluidization properties are evaluated by measuring the pressure drop across the bed. However, the degree to which it can be controlled, via the fluidization velocity, is limited depending on the materials fluidization properties. As a consequence, the level of control available reduces as a function of the ease of which the tested material becomes fluidized. In this paper, traditional fluidization measurements were compared to an aeration test carried out with the FT4 Powder Rheometer.

Tests belonging to the same group were evaluated with regard to their ability to discriminate between different powders.

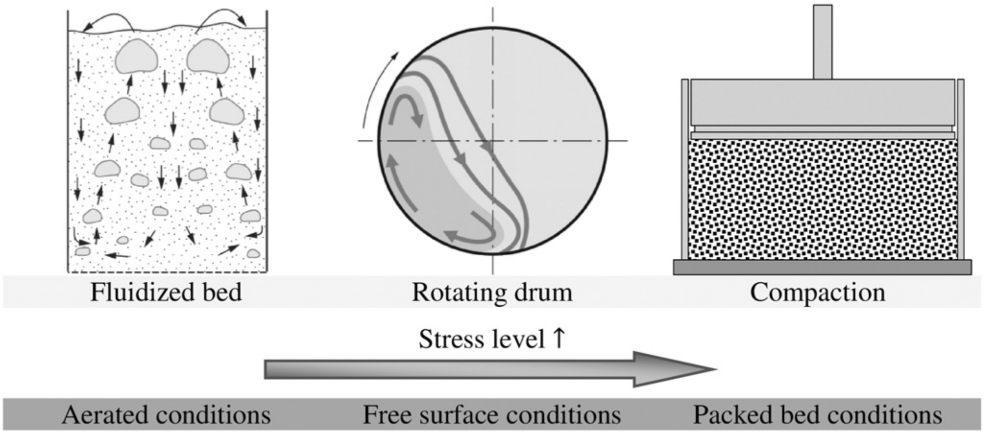

Fig. 1. Powder flow behavior over a range of stress levels.