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Experimental characterization of the cohesive behaviour of fine powders by the raining bed test

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ABSTRACT

The paper analyses and discusses the possibility of using the so-called 'raining bed experiment' to achieve quantitative characterization of the macroscopic cohesion force that affects fluidization of group A solids. This procedure, originally devised by Buysman and Peersman in 1967 [1], is based on measuring the minimum velocity required to an up-flowing gas stream for an up-flowing gas stream to hold a particulate bed against a porous plate at the top of a column, before bed failure occurs, either as a rain of particles ('rain-off') or as a fall of plugs. Rain-off velocities and bed pressure drops relevant to different cuts of glass ballotini, ceramics spheres, alumina, FCC catalyst, Ludox catalyst, silica sand and other powders, with average particle size ranging from 25 to 465 μm, are determined. These data are compared with the corresponding ones obtained by performing fluidization experiments on the same materials. Such a comparison allows a classification of the powders among cohesive and free-flowing solids. Moreover, the raining bed technique is able to provide valuable information about the relationship between macroscopic cohesion and fluidization properties of fine solids. Its development is thus likely to allow evaluating the excess drag force required to overcome interparticle forces in the transition from the fixed to the fluidized state.

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1. Introduction

Fine solids are usually employed in fluidized bed processes like catalytic cracking or polymerization, whenever large specific surfaces of contact between fluid and solid enhance the overall operation efficiency. In other cases, the presence of a fine powder is the consequence, at some stage of a process, of the formation of a solid (by crystal nucleation, spray-drying, etc.), subsequently subjected to treatment to achieve different goals (granulation, coating, etc.).

In spite of the frequency with which fine solids are encountered it is generally agreed that a satisfactory knowledge of their fluidization behaviour has not yet been achieved due to the strong influence exerted by interparticle forces on the properties of the solid bulk [2]. For particles with a density lower than 2000 kg/m^3 and smaller than 100 μm the forces originating from particle-particle interaction are comparable with their weight so that a tendency to form solid aggregates is sometimes observed. For the same reason, when subjected to flow cohesive powders fracture in coherent pieces much thicker than a mono-particle layer.

A relatively simple case is that of particulate bulks not subjected to gas flow across their interstitial voids; for these systems the ratio between interparticle force and particle weight, which defines the cohesive granular Bond number, [3] is considered sufficient to classify solids into the two large groups of cohesive and non-cohesive materials [4].

In the presence of interstitial flow, instead, the flow properties of the solids are also influenced by gas-particle interactions. In fluidization, for instance, the simultaneous action of gas-particle and particle-particle forces superimposed to that of gravity is the origin of different behaviours reflected by Geldart's classification [5], for which A solids (aeratable or homogeneously fluidizable) are distinct from C type (non-fluidizable) in that the gas drag is insufficient to overcome interparticle forces. The importance of the interparticle cohesive forces in determining the fluidization behaviour of a bed of particles is further demonstrated by the possibility of using very fine particles as additives capable to act as a flow aid for the fluidization process of cohesive solids [6] or, on the contrary, the observation that a significant rise in fluidization temperature may prevent suspension of a fine solid otherwise fluidizable at ambient conditions [7–9].

As far as macroscopic behaviour is considered, out of the field of fluidization interparticle forces are closely related to the concept of 'flowability', a complex of variously defined characteristics related to the ability of a granular material or a powder to flow under a specified set of conditions [10]. The possibility of determining, predicting or improving solid flowability is of crucial importance for the development of solid processes as well as for their regulation and control. Operations like solid dosage, blending or separation and many other aspects of solid handling are all facilitated by the ability of particulate phases to flow regularly under the action of gravity or other forces applied to them.

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For this reason, methods for measuring the flow properties of particulate materials as determined by the macroscopic cohesive force acting in the solid bulk constitute a matter of great interest in the field of fine solid processing. In a recent paper [11] a comparison has been made of the measurement results of the tensile strength provided by different methods and apparatuses, namely the Raining Bed Method [12], Sevilla Powder Tester [13] and the Ball Indentation Method [14]. Of these, the Raining Bed Method, recently developed by the authors after an original idea by Buysman and Peersman [1] is perhaps the less popular; for this reason, this paper will illustrate in detail both the characteristics of the technique and some of the indications obtainable from its results.

2. Parameters related to powder flowability

To evaluate the ability of a powder to flow, various parameters have been proposed together with the procedures for their measurement. Among them, widely used are the angle of repose (AOR), the compressibility index (CI) (also called Carr index) [15–16] and the Hausner ratio (HR) [17], as their definition is simple and they are rather easy to handle. In spite of that, each of these parameters is but indirectly related to the macroscopic cohesive force acting in the solid bulk so that also its relation with the flow behaviour of the solid is not straightforward.

An AOR value lower than 30° indicates ‘excellent’ flow properties whereas values higher than 56° are associated to a ‘very poor’ attitude to flow. With respect to its flowability, at intermediate levels the solid may be classified as ‘good’ (AOR between 31 and 35°), ‘fair’ (AOR between 36 and 40°), ‘passable which may hang up’ (AOR between 41 and 45°) and ‘poor which must be agitated or vibrated’ (AOR between 46 and 55°) [18]. The angle of repose is not an intrinsic property of the powder: as it may happen to keep memory of the stress history of the solid, many researchers do not consider it a valid parameter for determining powder flowability. However, the method is still in use thanks to its simplicity.

The ‘Carr index’ (CI) of a powder is used as a measure of its tendency to undergo consolidation and is defined by the following relationship [15]:

$$CI = \frac{\rho_t - \rho_a}{\rho_t} \tag{1}$$

The Carr index of a powder is inversely related to its flowability: solids that flow well are very densely packed, so that they can hardly undergo further compression; on the contrary, poorly flowing materials are rather loosely packed and can be further compressed easily. A powder with a compressibility index lower than 20% is considered to have a good flowability.

Also popular is the Hausner ratio [17], an index referred to the compressibility of the material, which is defined as the ratio between the tapped and the aerated bulk density:

$$HR = \frac{\rho_t}{\rho_a} \tag{2}$$

This ratio is a useful measure of cohesion that somewhat accounts for interparticle friction: ‘*if powders pack well, they flow well*’ [4].

In Table 1 the various types of behaviour are classified in reference to these two indexes as well as to the value of angle of repose of the material.

Different empirical techniques have been developed for measuring bulk flow properties of powders, varying with the nature of the solids and the field of application (pharmaceutical, metallurgical, xerographic industry, etc.) [19]. Even though some of them are allow rather easy measurements, a frequently encountered problem is that related to the initialization of the experiment, as the ‘history’ of the powder (filling procedure, previously applied stresses, interaction with the mechanical parts of the equipment, etc.) may have an influence on the results. Furthermore, it is not sure that the macroscopic properties of solid phase determined from measurements on a static sample can safely be utilized in applications like fluidization or aerated flows, where the gas-solid interaction is thought to play a major role.

Table 1
Behaviour of the powders at varying Hausner Ratio, Carr Index and Angle of Repose.

HR [−]	Behaviour classification	CI [%]	Flow description	AOR [°]
>1,4	Cohesive	>40	Cohesive (very very poor flow)	>66
		35–38	Cohesive (very poor flow)	56–65
		28–35	Cohesive (poor flow, vibrate)	46–55
1.25–1.4	Semicohesive	23–28	Easy fluidizable (poor flow)	41–45
		18–21	Fair to passable powdered granule flow	36–40
<1.25	Free-flowing	12–16	Free-flowing (good flow)	31–35
		5–15	Free-flowing (excellent flow)	<30

For these reasons, while they are closer to the conditions of a large number of applications, techniques that contemplate the presence of gas flow across the particle bed subjected to the determination of its internal cohesion seem more suitable because conditioning removes the stress history of the solid bulk. This is the case, for instance, of the Sevilla Powder Test [20] and of the Raining Bed Method (RBM) referred to in the present paper [11]. The latter technique, recently proposed by the authors after an original idea by Buysman and Peersman [1] and still under further development is perhaps the less popular, although it exploits experimental conditions (presence of interstitial gas flow, low consolidation level of the solid bulk, etc.) very close to the conditions of the fluidization process. Owing to that, this work illustrates both the characteristics of the technique and the results obtainable from its application.

3. Experimental facility and technique of the raining bed test

The fluidization and rain-off experiments of this paper were performed in a transparent column of Perspex with an internal diameter of 54 mm, 400 mm high. As sketched in Fig. 1, both ends of this column

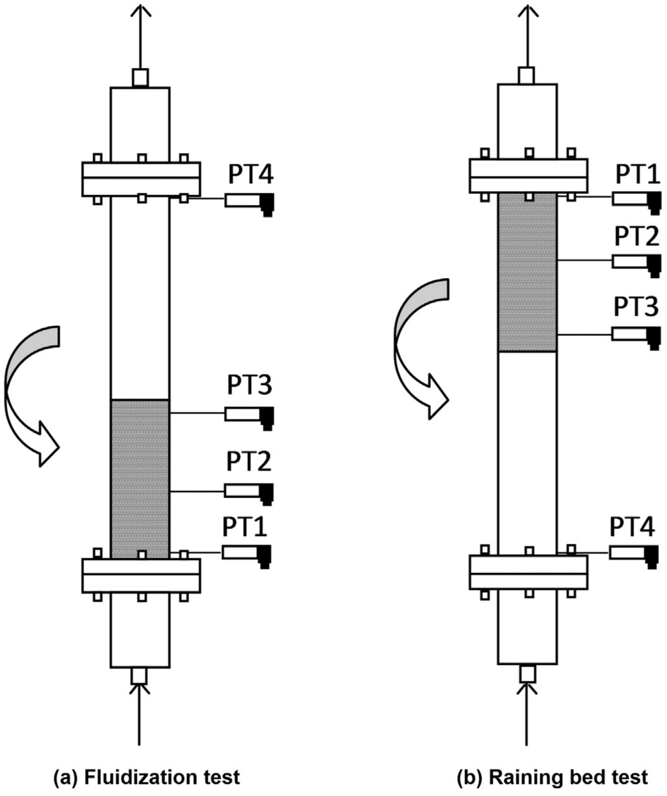

Fig. 1. Experimental arrangements.

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