DETERMINATION OF THE TENSILE STRENGTH OF A PARTICULATE SOLID BY THE RAINING BED METHOD

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Abstract – Based on an experiment first devised to investigate the stability of bubbles in a fluidized bed (Buysman and Peersman, 1967), an experimental technique is presented, capable to determine the tensile strength of a powder bed subjected to interparticle gas flow.

As a major difference with the usual fluidization procedure, in the Raining Bed Method (RBM) the particulate solid is held against a porous plate located on top of the column, supported by air flowing upwards from its bottom. The air flow rate is gradually reduced until the fall of the bed (rain–off) occurs at the moment at which the equilibrium condition between fluid drag, gravity and interparticle cohesion is attained. Determination of the superficial velocity and of the pressure drop across the bed at the moment of particle fall gives a valuable insight into the behaviour of systems affected by interparticle forces, in that it provides a criterion for distinguishing cohesive solids from cohesionless ones. Coupled with other experimental data, it eventually leads to quantitative determination of the tensile strength of the bed.

The paper illustrates the main features of the RBM technique; although still under development, it seems capable to support the effort of developing a better representation of the fluidization properties of cohesive materials.

INTRODUCTION

Fine solids are employed in a large number of fluidized bed processes but 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 particulate bulk. For particles 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 often 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, (Nase et al., 2001) is considered sufficient to classify solids into the two large groups of cohesive and non-cohesive materials (Castellanos, 2005).

In the presence of interstitial flow, instead, the flow properties of solids are also influenced by gas particle interaction. In fluidization, for instance, the simultaneous action of gas-particle and particle-particle forces superimposed to that of gravity is the origin of the different behaviours reflected by Geldart's classification (Geldart, 1973), for which A solids (aeratable or homogeneously fluidizable) are distinct from type C (non-fluidizable) in that interparticle forces can still be overcome by gas drag.

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 in a specified set of conditions. The possibility of determining, predicting or improving solid flowability is of crucial importance for solid processing: 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.

So, both in fluidization of fine particles and in the wider field of solid processing, methods for measuring the macroscopic cohesive force acting in a particulate phase constitute a matter of great interest, as they should help throwing light on the relationship between the degree of cohesiveness of the solid bulk and its flow properties. Of these, the Raining Bed Method (RBM), recently proposed by the authors (Zafar et al., 2015) after an original idea by Buysman and Peersman (1967) 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. For this reason, this paper illustrates both the characteristics of the technique and the results obtainable from its application.

TECHNIQUE OF THE RAINING BED TEST

The facility used for developing the 'Raining Bed Method' (RBM) consists of a transparent plexiglas column with an internal diameter of 5.4 cm, 40 cm high. As sketched in Figures 1 and 2, both ends of this column are connected to a plenum chamber bearing a high pressure drop porous plate, so that air can be admitted to the facility through either of these distributors by acting on a three-way valve. Fig. 3 shows the whole apparatus with two mass flow controllers on the air feed line, covering the range 0-6000 Nl/h, which regulates the flow rate of the fluidizing gas. Bed height values are determined thanks to two graduate scales set at 180° on the transparent wall of the facility.
The column is also provided with four pressure taps connected to as many transducers, to allow pressure drop measurements: PT1 and PT4 are placed symmetrically, level with the two gas distributors; as regards PT2 and PT3, they are located 4.6 and 11.2 cm apart from PT1, respectively. In order to skip from fluidization to rain-off experiments, the column can be rotated upside down around a horizontal axis passing through its centre of gravity.

Measurements of the differential pressure drop were carried out across both the whole height of the bed (ΔP_{1-4}) and an inner part of it 66 mm high (ΔP_{2-3}). The latter is a measurement of ΔP within the solid bulk far from the distributor region as well as from the free surface of the bed. For this reason, ΔP_{2-3} is the only variable whose values determined during the fluidization and the rain-off tests can directly be compared.

Each rain-off test is run with an amount of solids equal to that used in the corresponding fluidization experiment. Moreover, after pre-fluidization of the particle mass, bed voidage is brought down to the value measured in the fluidization experiment by gently tapping the particle assembly. Then, the direction of the gas feed is inverted thanks to the solenoid valve in a way that it is let flow across the bed from top. On doing that, the gas flow rate is set at a value (variable with the material at hand) suitable for preventing the fall of the solid bed after column rotation. The column is then rotated and the absence of unwanted changes of the bed voidage is checked by monitoring the internal pressure drop ΔP_{2-3} before and after rotation. Whenever a variation of ε_0 is observed, the whole experimental procedure is repeated from the beginning.

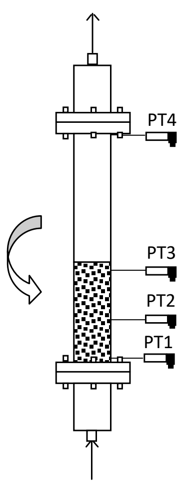


Fig. 1 – Fluidization test.

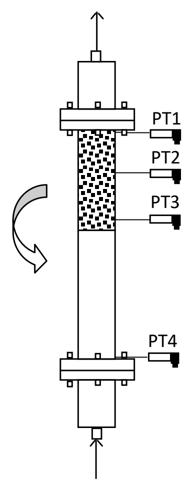


Fig. 2 – Raining bed test.

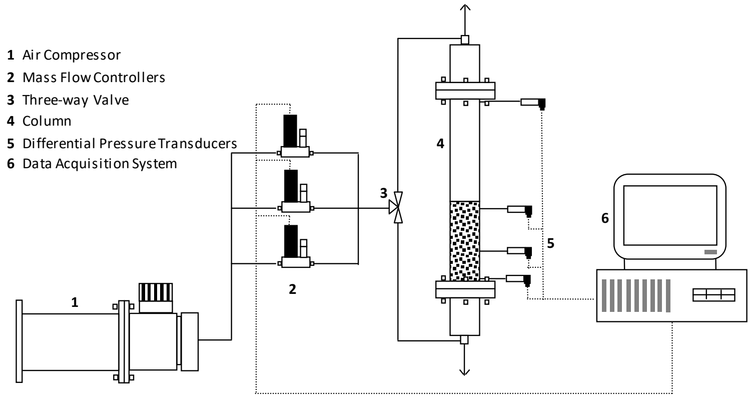


Fig. 3 – Experimental apparatus.

After column rotation, the bed is standing upside-down, held against the upper porous plate by the air that crosses it from the bottom at the superficial velocity u_{ie} that marks the beginning of the experiment (Fig. 2). The gas flow rate is then gradually reduced by a fixed amount until the fall of the bed ('rain-off') occurs at a velocity u_{ro}. A video of the position/plane of failure is recorded. At each value of u between u_{ie} and u_{ro}, both ΔP_{1-4} and ΔP_{2-3} are automatically acquired by the PC (Fig. 3) and stored for post-processing.

The aspect ratio H/D of the beds subjected to the rain-off experiments was never lower than 2.2 so that the solid mass was always sufficient to cover the pressure tap PT3 and to allow the measurement of ΔP_{2-3}.

COHESIVE AND NON-COESIVE SOLIDS

Although performed on systems having opposite configurations, both the fluidization and the bed support experiments analyse the effect of the variation of the gas flow rate on the stability of a packed bed of particles crossed by a gas stream. To check this similarity, the respective series of data of the total pressure drop ΔP_{1-4} at varying superficial gas velocity, as well as those of the pressure drop ΔP_{2-3} across an internal portion of the bed of known height, are reported in the same diagram. When this is done for a bed of non-cohesive particles, as for example it done in Fig.4 for ceramic spheres, CE300, (ρ_s=3850 kg/m^3; d_{SV}=300 μm), the comparison of the hydrodynamic features of the two experiments free from the effects of interparticle cohesion reveals, as expected, that both the data of the partial pressure drop and those of ΔP_{2-3} are represented by the same curve, regardless of the type of test they are relevant to. Both the curves of ΔP_{1-4} and ΔP_{2-3} are given by the same relationship, like that of Ergun.

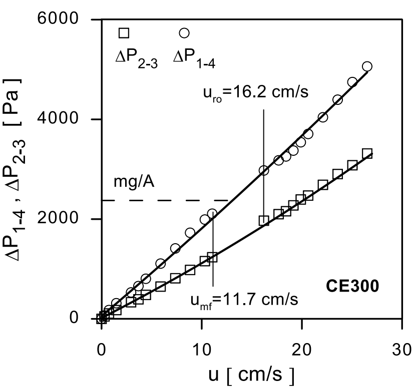


Fig. 4 – Typical diagram of comparison between fluidization and rain-off tests for a non-cohesive solid.

Fig. 4 reports also the experimental values of u_{mf} and u_{ro} of the particle bed; as orderly found in any fluidization test, the experimental value of ΔP_{1-4} at u_{mf} is nearly equal to the bed weight per unit area, mg/A.

The fact that an excess drag force is needed to sustain the suspended bed of a free-flowing material is signaled by the circumstance that the "rain-off" velocity u_{ro} is always higher than the corresponding u_{mf} in spite of the coincidence of the two trends of the pressure drop.

The common feature of the state of incipient fluidization and of that at which the supported bed "rains off" is that the drag force exerted on particles by the up-flowing stream must balance gravity. However, as first pointed out by Buysman and Peersman (1967), in the former case the fluid particle interaction has place everywhere at the interstitial gas velocity; in the latter, instead, that occurs only in the solid bulk, since the velocity at which the gas flows against the bottom layer of the bed coincides with its superficial velocity. A further difference is that during the process of fluidization any velocity rise over u_{mf} is followed by the commencement of bubbling (or the growth of voidage), whereas during bed support it causes a proportional increase of the pressure drop. Such considerations are outlined in Fig. 5, where the evolution of the axial pressure profile at varying superficial velocity is schematically illustrated.

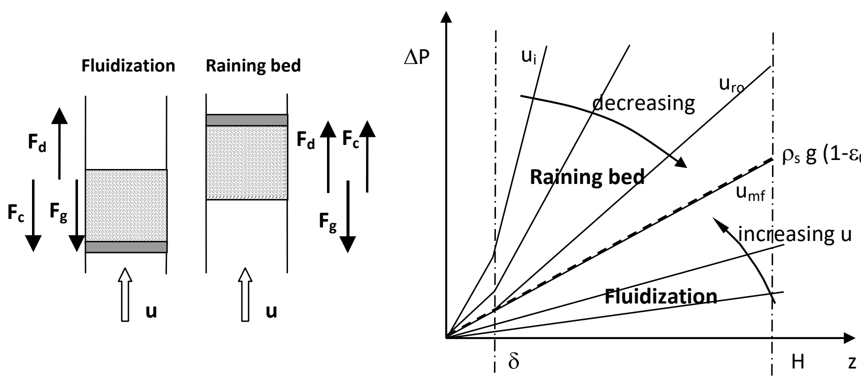


Fig. 5 – Axial pressure profiles during fluidization and raining bed experiments.

During the fluidization procedure, the slope of ΔP versus z increases along with u until, at $u=u_{mf}$, it equals the ultimate value $\rho_s g(1-\varepsilon_0)$. As regards the raining bed test, instead, the pressure profile is characterized by two distinct slopes, both of which decrease as much as the gas flow rate is reduced. Given the difference of velocity at which the gas flows across the bottom layer (of thickness δ) and the solid bulk, on reducing the supporting flow rate the critical value of ΔP slope at which rain-off occurs, $\rho_s g(1-\varepsilon_0)$, is first reached within the bottom layer, while that relevant to the solid bulk is necessarily steeper. The fall of the outer particles uncovers the contiguous region of the bed which, in turn, undergoes the same dynamics; by this mechanism, the whole solid mass eventually collapses, layer by layer. These considerations explain why, for any free-flowing material, the total pressure drop across the reversed bed at its rain off velocity is always higher than that measured at incipient fluidization (see again Fig. 4).

The comparison of results of Fig. 4 is now repeated in Fig. 6 for the case of the Respitose® (a sieved grade α-lactose monohydrate used in the pharmaceutical industry as an excipient), whose behaviour has been found to represent that of a cohesive solid. Its granulometric characteristics, as determined by a laser diffraction using the Mastersizer 2000 laser diffractometer, are reported in the following table:

Table.1: Properties of the experimental material.

	Density ρ_P [kg/m^3]	Volume diameter d_V [μm]	Sauter diameter d_{SV} [μm]	Sfericità ϕ [-]	d_{10} [μm]	d_{50} [μm]	d_{90} [μm]	Span	F_{25} [%]	F_{45} [%]
Respitose®	1515	69	28	0.7	38.2	66.3	105.9	1.022	4.5	18.2

The differences with what observed with free-flowing materials are evident: typical of cohesive solids is that their experimental u_{ro} is lower than u_{mf} or equal to it so that the velocity intervals covered by the two experiments partially overlap.

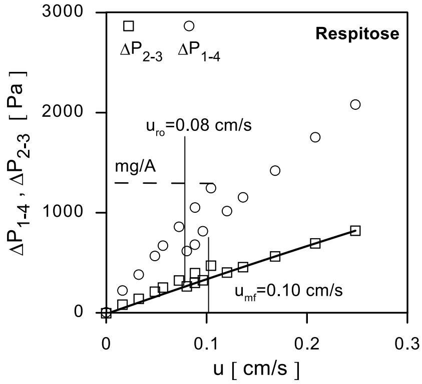


Fig. 6 – Typical diagram of comparison between fluidization and rain-off tests for a cohesive solid.

Such a finding can be explained by what highlighted in Fig. 5, i.e. that during the process of fluidization the cohesive force F_c opposes to the drag F_d exerted on particles by the up-flowing gas, whereas it adds up to drag in balancing the action of gravity force F_g when the bed is subjected to the rain off experiment.

The analysis of a large number of results relevant to solids of various nature and size reveals that what shown by the two materials referred to in this paper represents a result of general validity, so that to a first approximation a value of the ratio u_{ro}/u_{mf} greater than 1 is typical of cohesionless particles, whereas values of the velocity ratio lower than unity are obtained with cohesive bulks.

THE MEASUREMENT OF THE TENSILE STRENGHT OF THE SOLID BULK

As originally observed by Buysman and Peersman (1967) and subsequently confimed by Seville and Clift (1984), the fall of the bed at u_{ro} may occurr by either of two different mechanisms: free flowing materials give place to a rain of individual particles or tiny agglomerates, whereas beds of solids endowed with interparticle cohesion show a mechanism of fracture into horizontal layers that fall in rapid succession at a critical value of the gas flow rate or at slightly lower values.

At u_{ro}, if cohesion is present, the tensile strength resists raining even when the pressure drop is less than the bed weight. As sketched in Fig. 7, at the bed failure point, the upward fluid drag, F_{drag} and tensile force F_c balance the weight of the plug that falls down,W_{plug}, i.e.,

$$A[\Delta P_{\mathrm{plug(uro)}} - \sigma_{\mathrm{t}}] = W_{plug} \tag{1}$$

where σ_t is the macroscopic tensile strength acting on the horizontal failure plane of cross-sectional area A and $\Delta P_{plug(uro)}$ is the pressure drop across the plug height H_{plug} at the rain-off point. W_{plug} can be measured by collecting the plug and weighing it, or alternatively, as used here, it can be determined from the pressure drop across bed height H_{2-3} at minimum fluidization conditions, corresponding to u_{mf}, i.e.,

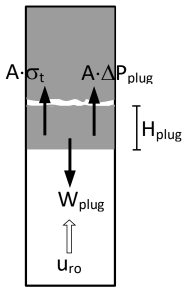
$$A[\Delta P_{\mathrm{plug(umf)}}] = W_{plug} \tag{2}$$

and

$$\Delta P_{\mathrm{plug(umf)}} - \Delta P_{\mathrm{plug(uro)}} = \sigma_{\mathrm{t}} \tag{3}$$

Now, considering that the pressure drop per unit height is constant, Eq. (3) can be expressed in terms of ΔP_{2-3}, (which is actually measured) as given by Eq. (4)

$$\sigma_{\mathrm{t}} = [\Delta P_{\mathrm{2\text{-}3,(umf)}} - \Delta P_{\mathrm{2\text{-}3,(uro)}}]\frac{H_{plug}}{H_{2-3}} \tag{4}$$



H_{plug} is the height of the falling plug, whose value is obtained from the analysis of the images recorded by a video camera (Sony HDRHC7E) during the experiment. Although not perfectly even, the newly formed bed surface after the fall of the plug is smooth, planar and horizontal, so that evaluation of the mass of H_{plug} proves relatively easy.

At u_{ro}, the tensile strength resists raining, even when the pressure drop is less than the bed weight. At the bed failure point, the cohesive force $F_c=\sigma_t \cdot A$ directed upwards is added to the drag to balance the weight of the falling plug, W_{plug}, as illustrates in Fig. 7.

Fig. 7 – Typical equilibrium in the raining bed test.

Beds of fine, moderately cohesive solids are orderly subjected to a certain variation of their consolidation state, a fact associated with the parallel variation of the void fraction of the particulate phase. If a series of experiments is performed on the same sample at various levels of voidage, as done with Respitose, the minimum fluidization and the rain-off velocity, u_{mf} and u_{ro}, do change, as here shown in Fig. 8: u_{ro} is constantly lower than u_{mf} and the difference between the two paramenters grows, as expected, with the degree of compaction of the sample.

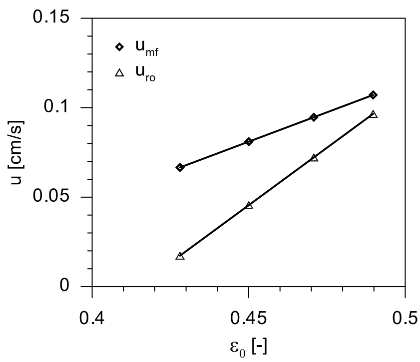


Fig. 8 – Minimum fluidization velocity and rain-off velocity at varying bed voidage.

The variation of ε_0 is accompanied by that of the height of the falling plug H_{plug}, so that also the tensile strength of the particle bulk is found to increase with the reduction of system voidage (see Fig. 9).

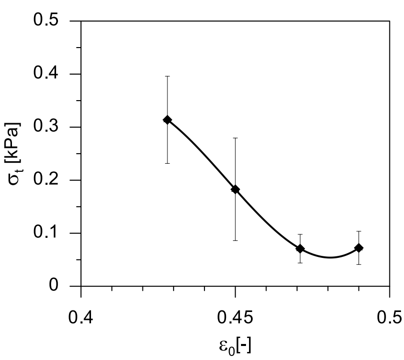


Fig. 9 – Tensile strength at varying bed voidage.

CONCLUSIONS

Further to providing a criterion based on the value of the ratio u_{ro}/u_{mf} for distinguishing between cohesive and non-cohesive solids, the Raining Bed Method is a technique potentially capable of allowing quantitative determination of the tensile strength of a bulk of solids of group A of Geldart classification. To this goal, the experimental determination of the height of the plug detaching from the particle bed is essential to calculate the tensile strength of the solid phase. Although its technique is only partially developed, RBM seems already capable to give an account of the variation of the tensile strenght with the consolidation state of the material.

NOTATION

A	column section, cm^2	H_{2-3}, H, H_{plug}	internal, total and plug height, cm
D	column internal diameter, cm	m	bed mass, kg
d_{SV}	Sauter's diameter of particle, μm	u	superficial gas velocity, cm/s
d_V	volume diameter of particle, cm	u_{ie}	initial gas velocity in the RBM, cm/s
d_{10}, d_{50}, d_{90}	size under 10, 50 and 90 %, μm	u_{mf}	minimum fluidization velocity, cm/s
F_c ,F_d, F_g	cohesive, drag and gravity force, N	u_{ro}	rain off velocity, cm/s
F_{25}, F_{45}	fine fraction under 25 and 45 μm, %	W_{plug}	weight of the plug, N
g	acceleration of gravity, m/s^2		

Greek symbols

δ,	thickness of external layer, cm
ΔP,	pressure drop, Pa
ΔP_{2-3}, ΔP_{1-4}, ΔP_{plug}	internal, total and in the plug pressure drop, Pa
ε_0	voidage of the bed, -
ϕ	particle sphericity, -
ρ_s	particle density, kg/m^3
σ_t	tensile strenght, Pa

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