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CHARACTERISATION OF COHESIVE POWDERS BY THE RAINING BED METHOD

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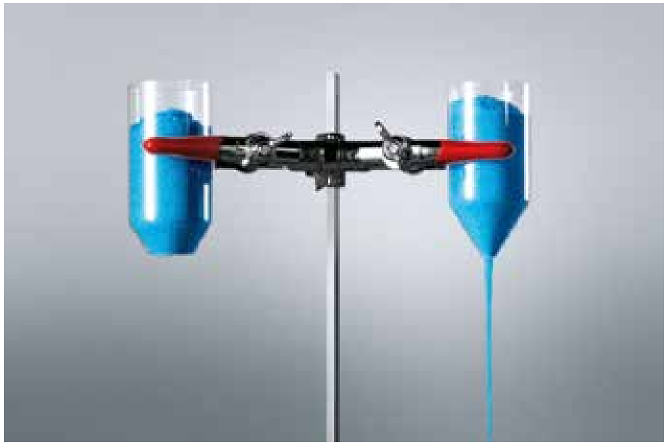
The flowability of a powder



- Flowability is not an inherent property of a particulate material; it results from a combination of various physical properties, environmental and processing factors.

- The capacity of a powder to flow under a specified set of conditions is highly dependent on the state of the powder and the application it is being used for.

- Owing to that, flowability cannot be described by any one value or any single index.

Methods of characterization

- The **‘right method’** to assess the powder flow properties depends greatly on the intended application. For these reasons most powder characterization equipment is designed to be application-specific.

- In general, it is advisable to use **multiple characterization techniques** to obtain information of powder flow under various sets of conditions.

- **The connection** among the various characterization methods available **is not well defined**.

Consolidation state

- Powder flow properties such as the yield strength depend, strongly and non-linearly, on the instantaneous degree of consolidation.
- For cohesive powders, the degree of consolidation is not an equilibrium property. Their instantaneous microstructure is typically non-uniform and is determined by their processing history.

- In some applications **the consolidation stress is very low** or virtually absent. In fluidization the interstitial gas flow acts on particles at any time.

Some popular experimental parameters and techniques for assessing powder flowability

Determination of

- Angle of repose (and other characteristic angles)
- Hausner and Carr ratios
- Compressibility indexes

Use of equipment and techniques like

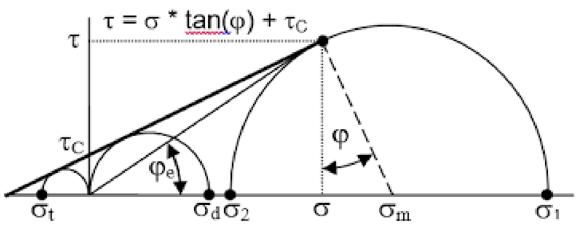
- Shear Cells
- Sevilla Powder Tester
- Ball Indentation

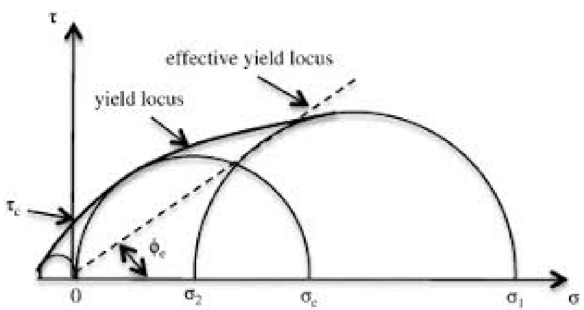
and……..

- **Raining Bed Method**

Although a correlation between the various techniques sometimes exists, a deeper understanding of how these methods relate to one another is still to be achieved.

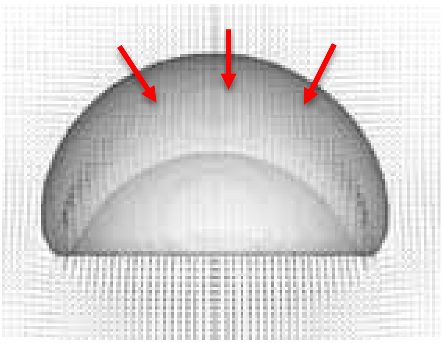
Systems with low consolidation stresses





- In a Shear Tester, solid bulk cohesion and tensile strength are determined by an **indirect measurement.**
- In particular the tensile strength of a powder is obtained by **extrapolating the yield locus to the tensile region.**
- Fitting a straight line to the yield locus may cause **overestimating s_t**.
- There is a need for methods of direct determination of s_t **at low consolidation stresses.**

Where the Raining Bed Method comes from



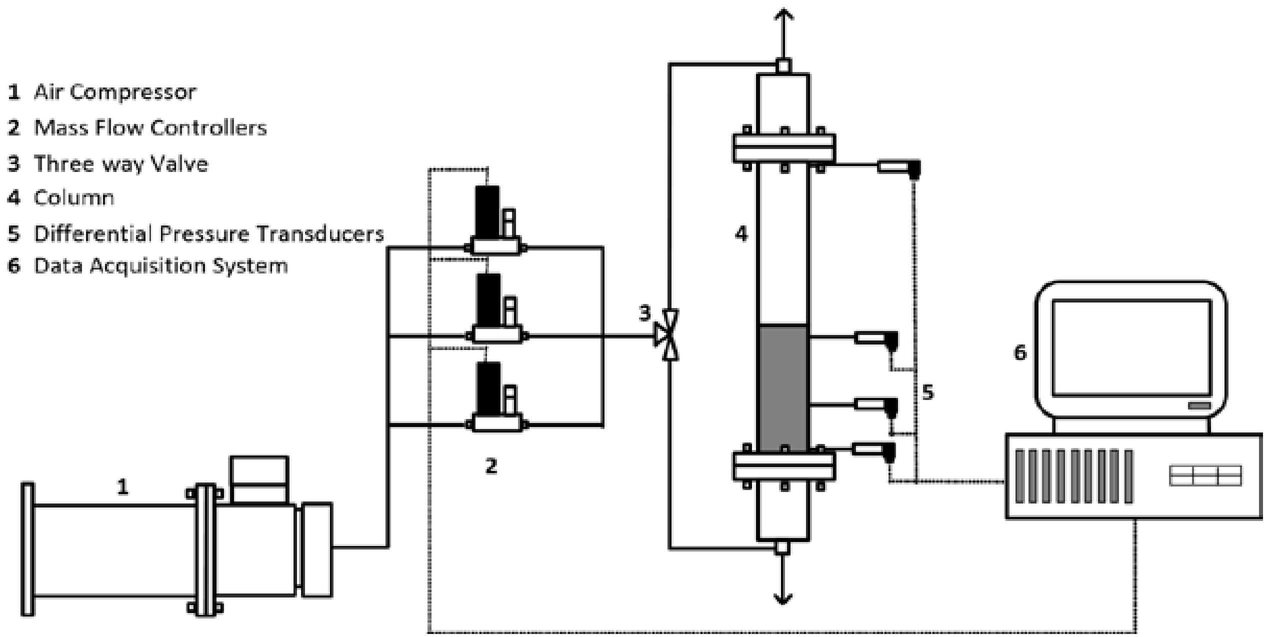
- The experiment on which the Raining Bed Method is based was first proposed by Buysman and Peersman:

  P. J. Buysman G. A. L. Peersman. *Stability of ceilings in fluidized beds.* In *Proc. Int. Symp. on Fluidization* , page 38, Eindhoven, 1967

- The technique was intended to give information on the stability of the roof of the bubbles flowing across a fluidized bed of either ‘free-flowing’ or ‘cohesive’ solids.

In spite of the efforts of (a few) other investigators, the experiment devised by Buysman and Peersmann did never undergo **significant developments.**

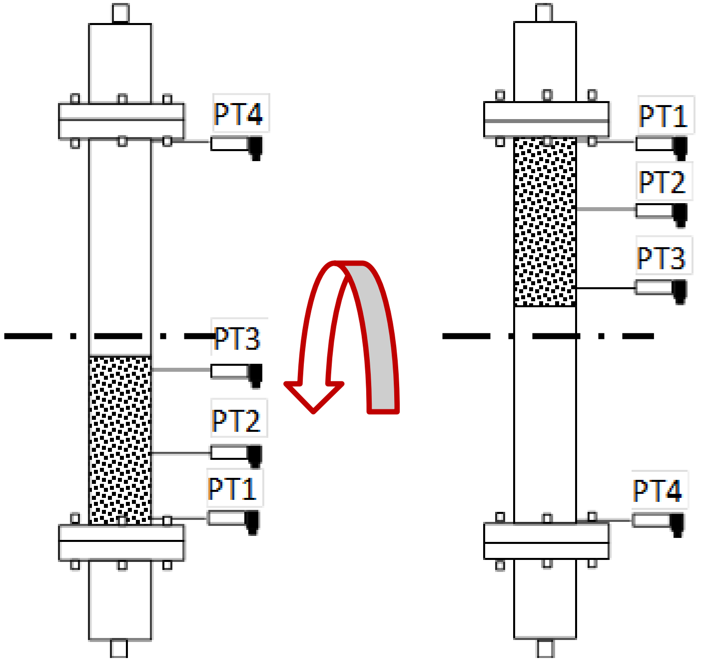
Apparatus for the rain-off experiment



Column diameter: 54 mm
Column height: 400 mm

- Air can be supplied to the column through either distributor
- Air feed and pressure lines need not be disconnected during rotation

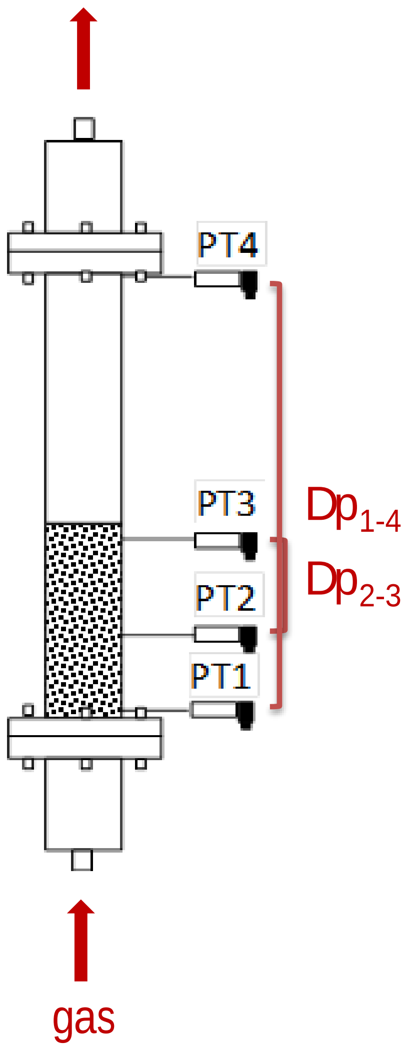
Fluidization and Raining Bed experiments



Same solid mass
Bed aspect ratio H/D @2.2 (to cover PT3)

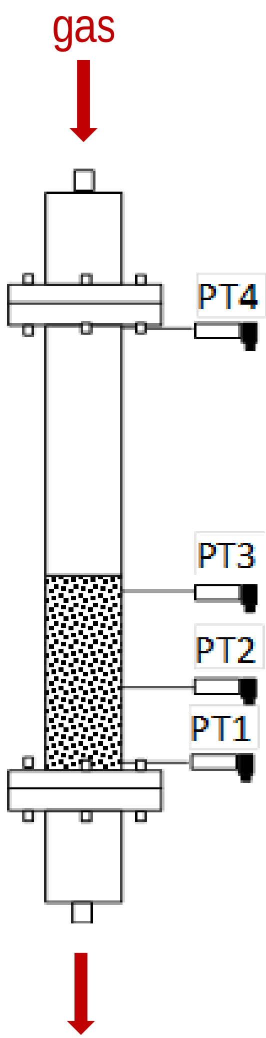
Same voidage (bulk density)

Fluidization experiments



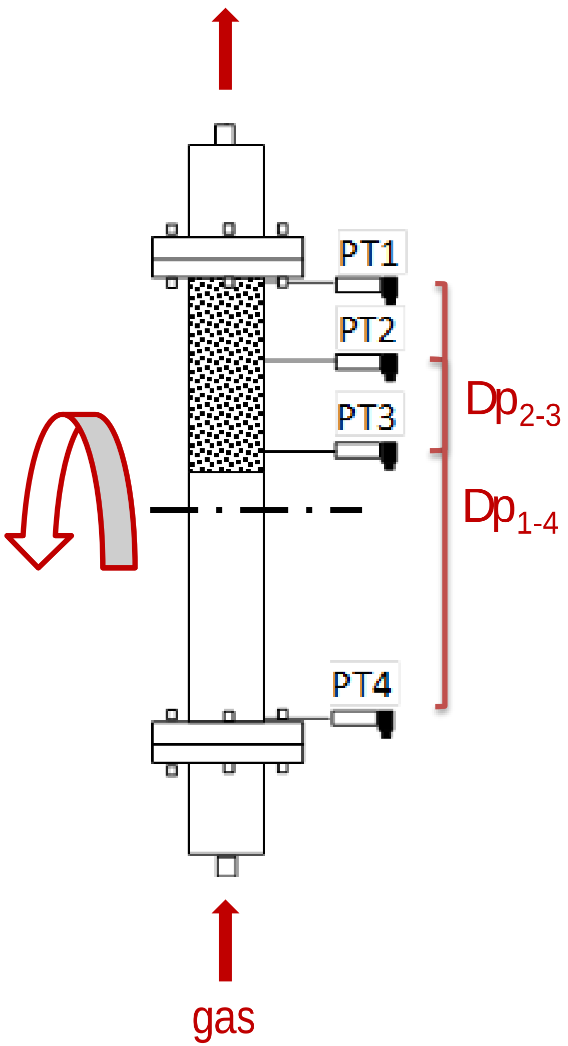
- The bed is first pre-fluidized to cancel its previous stress history
- The gas velocity is gradually increased until the bed achieves the fluidized state at u_{mf}
- Dp_{1-4} and Dp_{2-3} are measured (acquired) at each velocity value

Procedure of the Raining Bed experiment



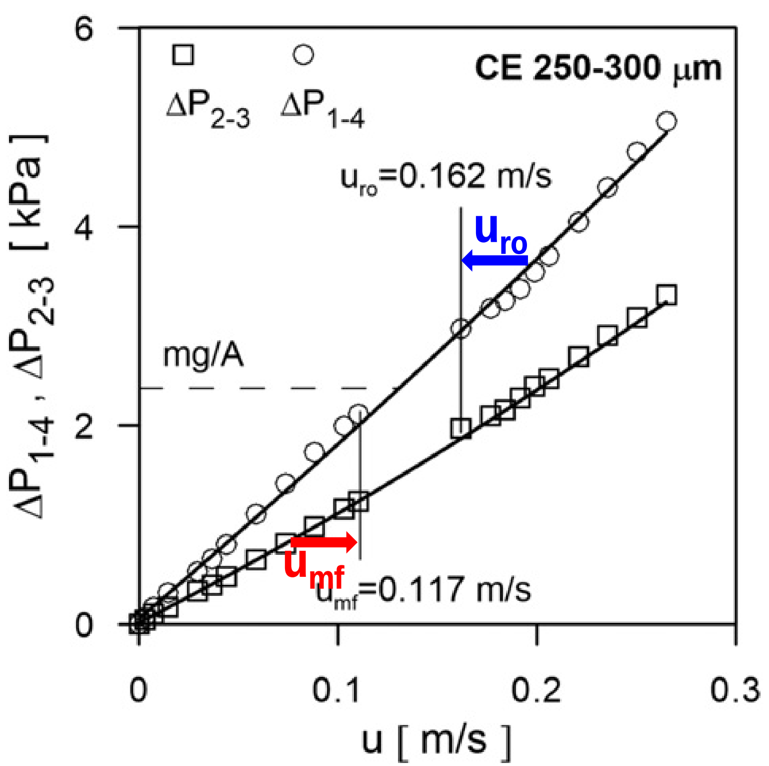
- Ø The bed is first pre-fluidized to cancel its previous stress history
- Ø The air flux is inverted by the three-ways valve (switched to the upper distributed)
- Ø Care is taken not to compress the bed to higher bulk densities
- Ø The column is gently rotated upside down

Procedure of the Raining Bed experiment (2)



- After rotation, the absence of unwanted variations of e_0 is checked by monitoring the value of Dp_{2-3}
- The gas velocity is reduced step by step
- Dp_{1-4} and Dp_{2-3} are measured (acquired) at each velocity value
- The velocity at which the bed falls down ('rain-off velocity', u_{ro}) is recorded

Fluidization vs Rain-off: free-flowing particles



- Ø Free-flowing materials give place to a rain of individual particles: the whole bed 'rains off' layer by layer

- Ø The superficial rain-off velocity is always higher than the minimum fluidization velocity:

$$u_{ro} > u_{mf}$$

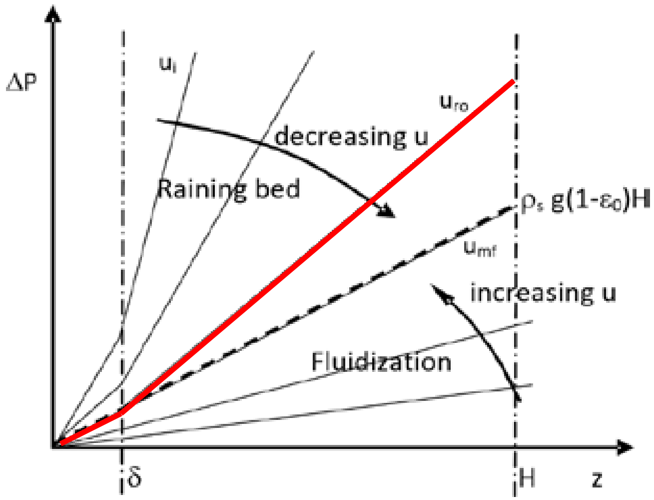
- Ø Both Dp_{2-3} and Dp_{1-4} are calculated by Ergun's equation :

$$\Delta p_{i-j} = \left[150 \frac{\mu_g u}{(\varphi d_P)^2} \frac{(1-\varepsilon_0)^2}{\varepsilon_0^3} + 1.75 \frac{\rho_f u^2}{\varphi d_P} \frac{1-\varepsilon_0}{\varepsilon_0^3}\right] H_{i-j}$$

Beds of free-flowing particles

As already observed by early investigators

- Free-flowing materials give place to a rain of individual particles: the whole bed ‘rains off’ layer by layer

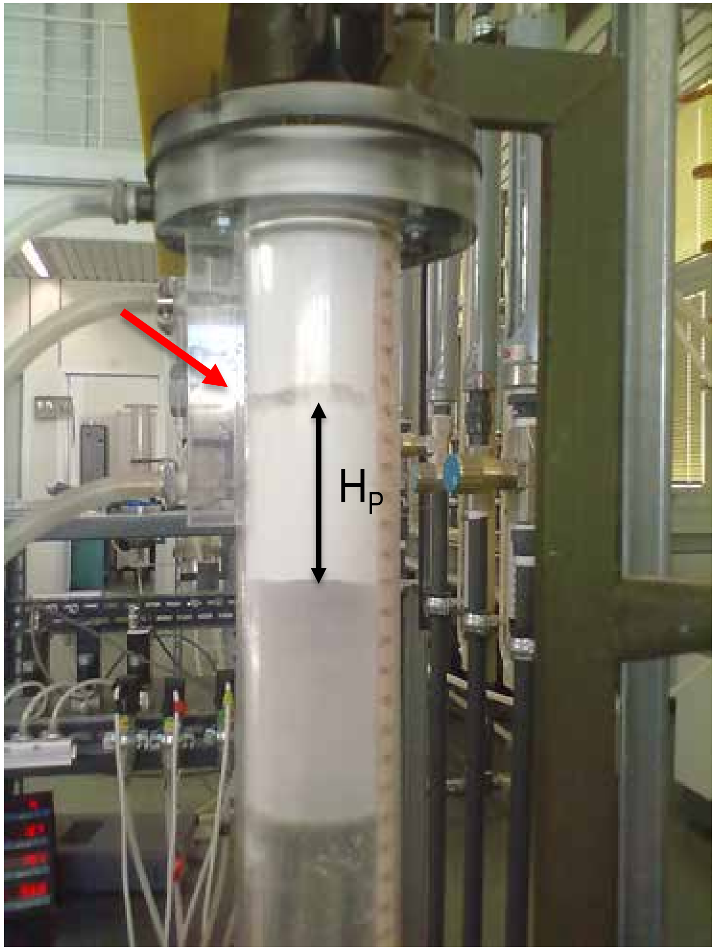


An equilibrium is established between drag and gravity at the external layer of the bed.

- In the fluidization experiment, the actual gas velocity at any height is the same, v=u/e

- In the raining bed test, the gas approaches the **external layer** of the bed at v=u. The drag force is much lower so that the superficial velocity required to support the bed is higher than u_{mf}.

Beds of cohesive particles

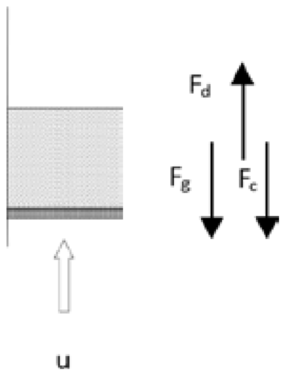


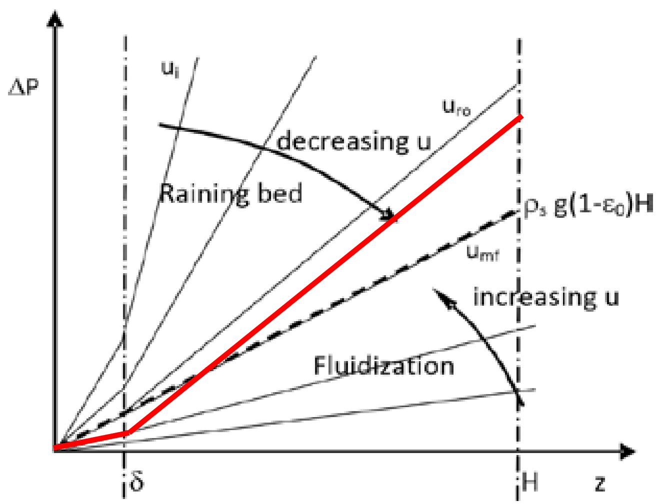
- Beds affected by interparticle forces undergo an internal fracture and fall down in plugs that break off in close succession

- By filming the experiment, the height of the plug is determined

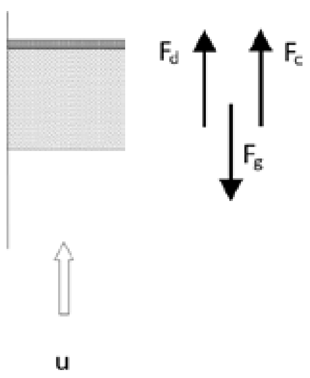
Effect of the cohesive force

Fluidization



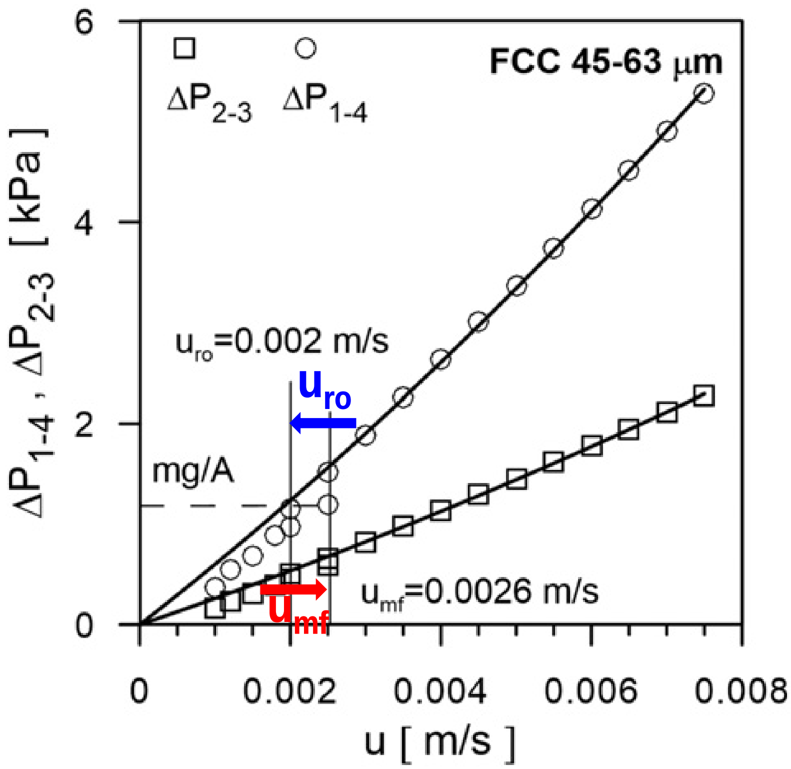


Raining bed



The presence of the cohesive force makes possible to the drag force decrease **under the value of the buoyant weight** per unit surface up to the fall of a plug .

Fluidization vs Rain-off: cohesive particles



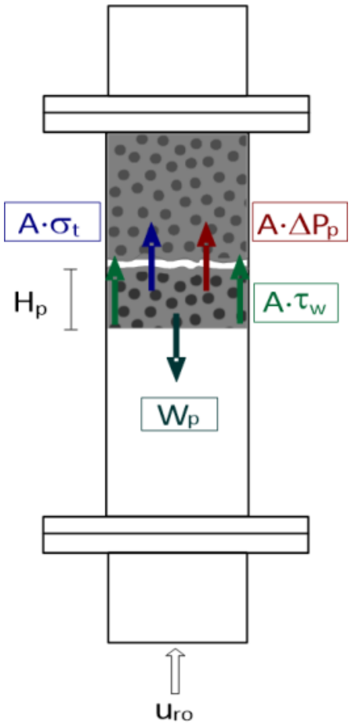
- Beds affected by interparticle forces undergo an internal fracture and fall down in plugs that break off in close succession

- The superficial rain-off velocity is now lower than the minimum fluidization velocity:

$$u_{ro} < u_{mf}$$

- Deviations of Dp_{2-3} and Dp_{1-4} from predictions of Ergun’s equation are observed

Tensile strenght of the solid bulk



At u_{ro} the tensile strenght s_t resists ‘raining’ even if the pressure drop across the plug is lower than its weight of per unit section:

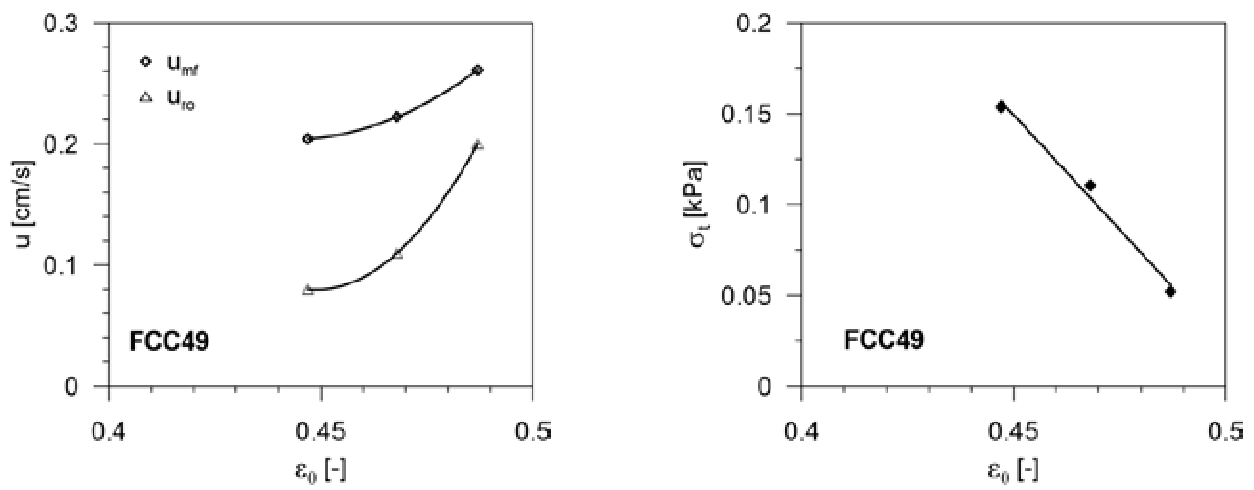
$$A\left[Dp_{P(u_{ro})} + s_t\right] = W_P - At_w$$

and it can be demonstrated that

$$s_t = \left[Dp_{2-3(u_{mf})} - Dp_{2-3(u_{ro})}\right]\frac{H_P}{H_{2-3}}$$

S_t at varying consolidation level: FCC

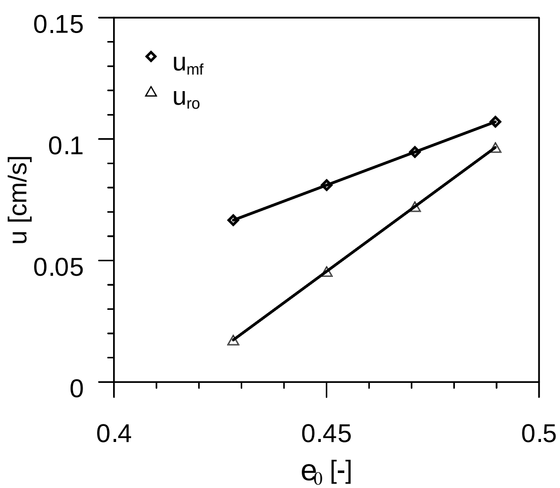
	Density ρ_P [kg/m^3]	Volume diameter d_V [μm]	Sauter diameter d_{SV} [μm]	Sfericity j [-]	d_{10} [μm]	d_{50} [μm]	d_{90} [μm]	Span	F_{25} [%]	F_{45} [%]
FCC catalyst	1800	52	49	0.99	24	50	70	0.92	22	32

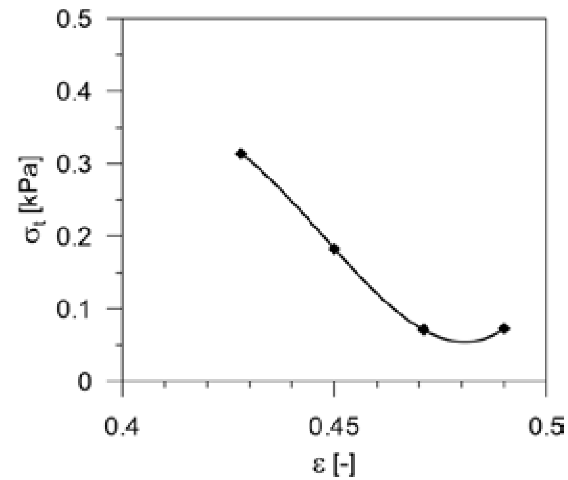


As expected, the tensile strenght of the bulk **decreases along with its bulk density**.

S_t at varying consolidation level: Respitose®

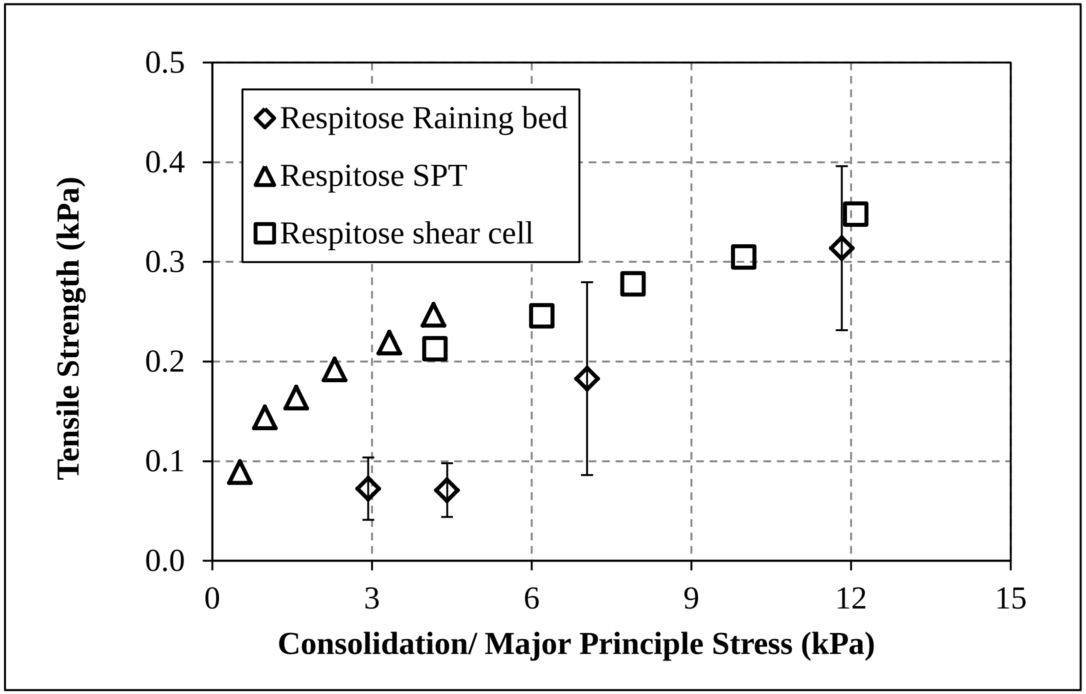
	Density ρ_P [kg/m³]	Volume diameter d_V [μm]	Sauter diameter d_{SV} [μm]	Sfericity j [-]	d_{10} [μm]	d_{50} [μm]	d_{90} [μm]	Span	F_{25} [%]	F_{45} [%]
RespitoseÒ	1515	69	28	0.70	38.2	66.3	105.9	1.022	4.5	18.2





A comparison with other techniques

Values of σ_t are compared with those provided by a Shear Cell and the Sevilla Powder Tester



- Lower values of σ_t are obtained.
- It is not clear whether the comparison is correctly made in terms of consolidation stress.
- Better control of the experimental conditions is required.

Achievements and problems

- After the original idea of Buysman and Peersman, the **Raining Bed Method** has been developed to measure the tensile strenght s_t of a particulate bulk by a direct experiment;

- The comparison between 'minimum fluidization' and 'rain-off' velocities provides a simple albeit approximate criterion for distinguish cohesive solids from free-flowing ones:

  if $u_{ro} / u_{mf} > 1$ the solid is **cohesionless**
  if $u_{ro} / u_{mf} < 1$ the solid is **cohesive**

- The amplitude of the deviation of the velocity ratio u_{ro} / u_{mf} from 1 is reflected by the thickness of the solid plug that falls down at u_{ro}. Its measurement leads to the quantitative determination of the **tensile strenght** of the particulate bulk.

- Improvements in the technique are needed as regards imposition and/or evaluation of the **consolidation state** of the solid bulk. This aspect is essential to integrate the Raining Bed Method into the set of techniques suitable for characterizing the flow properties of powders.