

Particle and Powder Flow Properties- Part II

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Notes include material adapted from:

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Topics/Outline

- Sampling (Wassgren)
- Particle size (Wassgren)
- Granular Material (Valverde/Castellanos)
- Particle-Particle Interactions
- Dry Particle Coating – Nano-additives
- Cohesion, Flow and Roles of Nano-additives (Valverde/Castellanos)
- Cohesion/Flow Characterization using several powder testers (Dave, Sanchez-Quintanilla, Valverde, Wassgren)
- Contact Modeling – Influence of Nano-additives (Yuhua Chen and Dave)
- Appendices
 - Plasticity Theory for Powders (Sanchez-Quintanilla)
 - Mechanical Properties (Hancock and Morris)
 - Review on Powder Testing Equipment (Sanchez-Quintanilla, Lauren Beach, Yuhua Chen, Laila Jallo)
- Reading material
 - Key papers as PDF files

* Names in blue are students who assisted with notes



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Relevance for testing

There is not a single physical model capable of explaining the behaviour of a granular material in the four dynamical regimes

THUS

- ✓ A single testing device can not give meaningful results for the flowability of a powder all the four regimes
- ✓ Before committing time for testing, the dynamical regime of the powder in the plant process needs to be identified.
- ✓ A testing device in which the powder is in the same dynamical regime as in the plant process must be selected.



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Types of testers

- **Shear tests**

In these tests the stresses applied to the powder are controlled to ensure the velocities of the particles are small and the powder is always in the plastic regime. They are the only tests backed by a well established physical theory.

- **Rheometers**

They borrow methods and ideas developed for fluid testing, but they lack the backing of a physical theory because the fluid mechanics does not directly apply to powders.

- **Tests based on mechanical stability**

In these tests the stresses applied to the powder are not controlled. Therefore, the powder may experiment transitions from the plastic regime to the granular or the fluidized regime.

- **Tests based on the solid fraction**

- **Tests based in fluidization**

These tests determine the properties of the sample in the fluidized regime.



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Tests based on mechanical stability

In this type of tests the stresses inside granular material are driven to the yield condition in an uncontrolled way and it is allowed to flow until the stresses are relaxed and the flow stops.

- Because the initiation of the flow is determined by the plastic yield condition, the results of these tests is influenced by the flowability of the powder in the plastic regime.

- However, since the velocities attained in the flow are not controlled, it may happen that the material enters the inertial or fluidization regimes. Hence the flowability of the powder in these regimes may affect the results of the test.

There are two types of this tests: tests based on avalanches and tests based on the angle of repose



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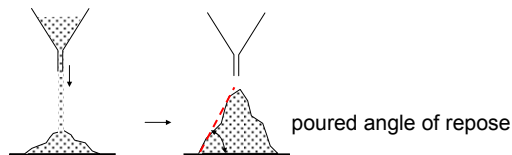
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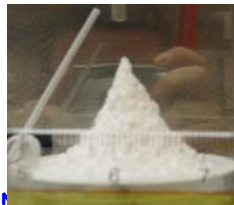
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Angle of Repose

- Poured angle of repose
 - Pour a powder from some elevation onto a flat plate and measure the angle that the powder slope makes with respect to the horizontal.



- The angle of repose is does not have a unique value, especially for cohesive powders.



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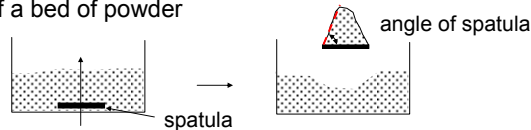
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Angle of Repose...

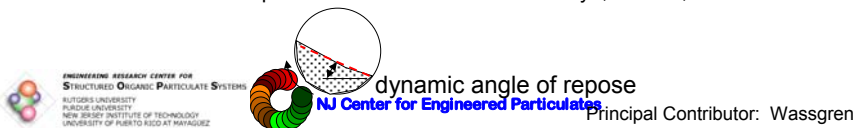
- Angle of fall
 - the angle of repose for a powder mound that has been impacted



- Angle of spatula
 - the angle of repose for a powder that has been formed by lifting a spatula out of a bed of powder



- Dynamic angle of repose
 - the angle of repose for a continuously avalanching powder
 - this concept is utilized in several testers- Kaye, Muzzio, etc.



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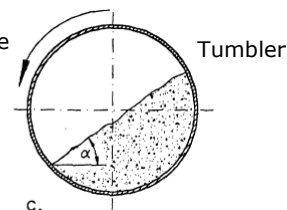
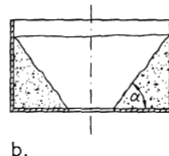
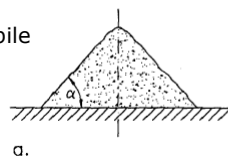
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Angle of repose

The tests based on the angle of repose characterise the flowability of a powder by the angle of the free surface of the material when the granular flow stops under different configurations.

Example of configurations: Hopper discharge

Conical pile



- When the unconfined yield strength of the material is not negligible, the stability of the free surface depends on the consolidation stresses acting on the surface.
- Since the consolidation stresses depend on the geometry and scale of the test, for cohesive materials these tests show **scale and geometry dependence**

No real problems can be solved by only knowing the angle of repose



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Angle of Repose...

- Segregated powder blends may have different angles of repose at different locations in the bed.



- A smaller angle of repose generally implies better flow characteristics.
- Angle of repose as a measure of flowability is most useful (only) for free flowing powders.
 - Hiestand (1991) states that “for pharmaceutical materials the angle of repose [as a flowability measure] is satisfactory only with powders whose flow characteristics are so good that one seldom needs the measurement.”

Material	Angle of Repose [deg.]
Sodium chloride	34 ± 1 (Lavoie <i>et al.</i> , 2002)
Sucrose	35 ± 1 (Lavoie <i>et al.</i> , 2002)
Lactose 100	38 ± 2 (Lavoie <i>et al.</i> , 2002)
Lactose 325	41 ± 1 (Lavoie <i>et al.</i> , 2002)



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Results using the Angle of Repose (I)

- Thalberg et al. Powder Technology 146 (2004) 206–213

Apparatus: PharmaTest Flow-time and Cone Angle Tester

Materials used:

Ordered mixtures of a carrier (Pharmatose 325M, $d_p \approx 50 \mu\text{m}$), intermediated sized particles (Pharmatose 450M, $d_p \approx 20 \mu\text{m}$) and micronized lactose ($d_p \approx 2 \mu\text{m}$)

Composition of ordered mixtures

Denomination	Pharmatose 325M, % w/w	Pharmatose 450M, % w/w	Micronized lactose, % w/w
[100/0/0] (Pure carrier)	100	0	0
[95/5/0]	95	5	0
[95/2.5/2.5]	95	2.5	2.5
[90/7.5/2.5]	90	7.5	2.5
[95/0/5]	95	0	5
[90/5/5]	90	5	5
[90/2.5/7.5]	90	2.5	7.5
[85/7.5/7.5]	85	7.5	7.5
[80/10/10]	80	10	10

Conclusions:

- ✓ The more cohesive powders, as determined by other techniques, have a higher angle of repose.
- ✗ For the most cohesive powders, the differences between the angle of repose tend to decrease
- ✓ There is a linear correlation between angle of repose and the modified Hausner Ratio



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Results for the Angle of Repose (II)

•Räsänen et al. *AAPS PharmSciTech* 2003; 4 (4) Article 53

Apparatus: Custom made: Funnel discharging into a plate

Materials used:

Active ingredient: paracetamol

Excipients: microcrystalline cellulose (MCC, Avicel PH101, PH102 and PH200 and a mixture of MCC101/MCC200 (75%/25%) and silicified micro-crystalline cellulose

Conclusions:

- ✓ The angle of repose increases with increasing concentration of the poorest flowing ingredient (paracetamol).
- ✗ For the largest concentrations of paracetamol, the angle of repose tends to a plateau (implies that one cannot really get very meaningful results for very cohesive powders)
- ✓ Samples with larger angles of repose showed larger minimum fluidization velocity and increasingly poorer fluidization in a fluidized bed



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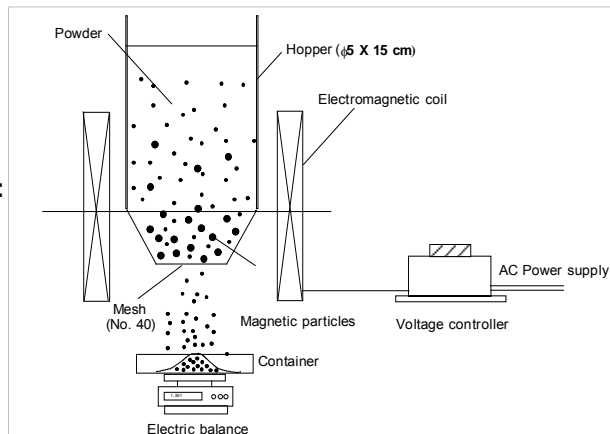
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A New Device Schematic of AOR Equipment

Magnetically Assisted Particle Flow (MAPF):
Use magnetic particles to agitate host powders, inducing flow.



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Parameters Studied

- ➡ Height from which the powder is dropped
- ➡ Mass of powder
- ➡ Magnetic field strength (field voltage)
- ➡ Hopper/funnel position within magnetic field
- ➡ Mass of magnets
- ➡ Outlet area of hopper/funnel
- ➡ Outlet mesh size
- ➡ Effects of different powders



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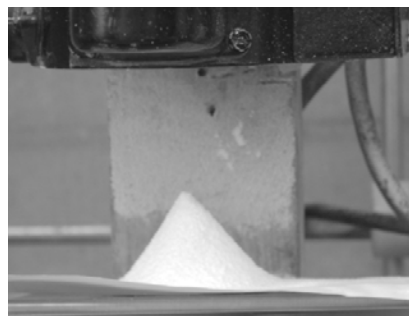
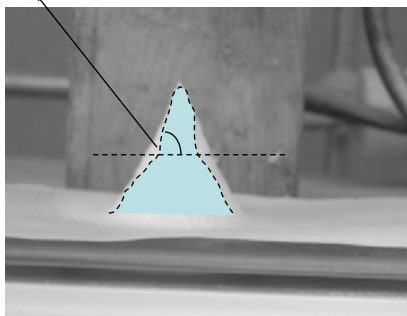


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Examples of piles obtained using this device

*False angle of
repose*



Clean heaps are obtained - **no false peaks** are observed



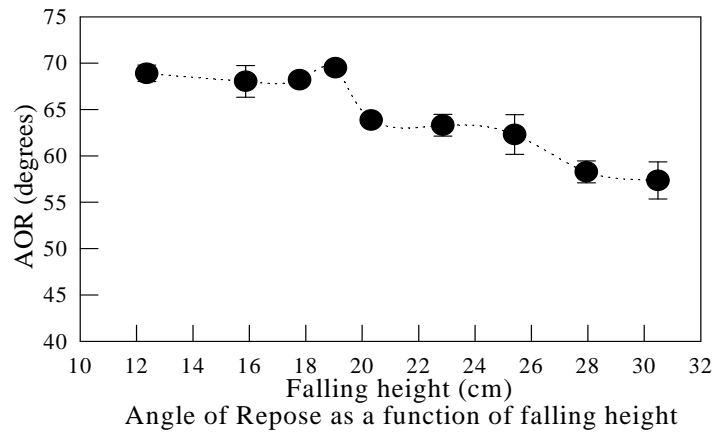
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Material Studied: Cornstarch
AOR as a function of height of fall



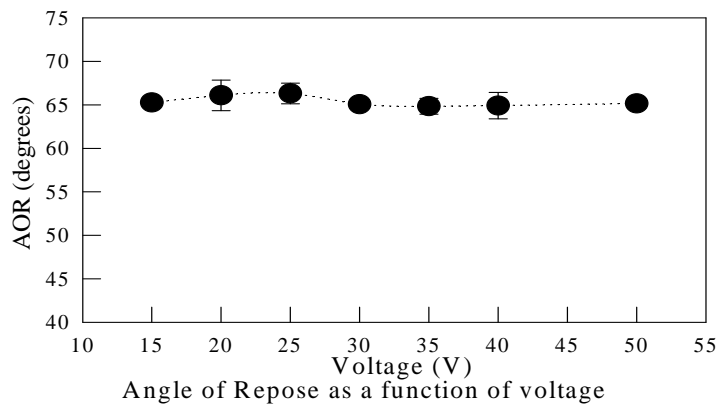
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AOR as a function of applied voltage (related to magnetic field strength)



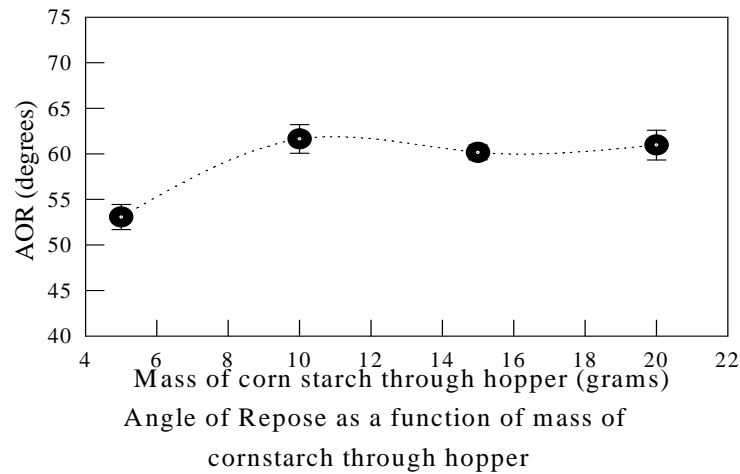
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AOR as a function of the powder mass



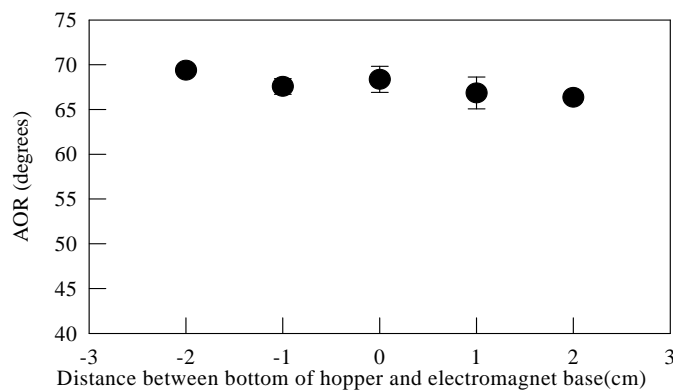
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AOR as a function of hopper/funnel position within the magnetic field



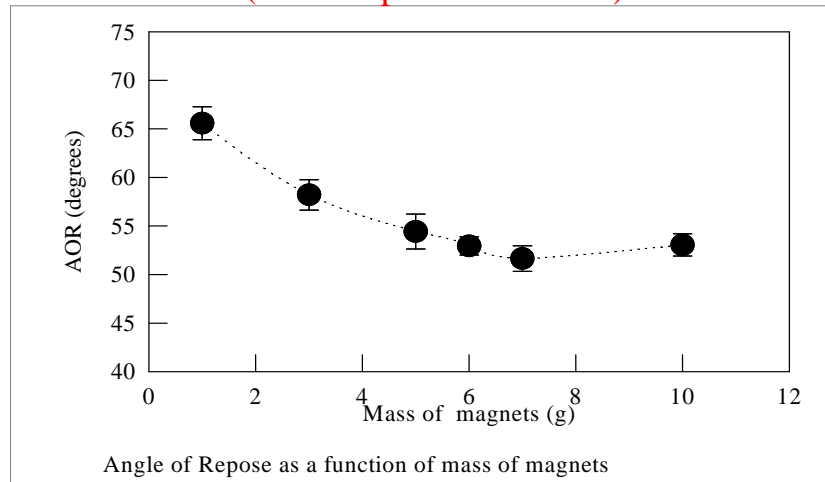
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AOR versus mass of magnets (mass of powder is fixed)



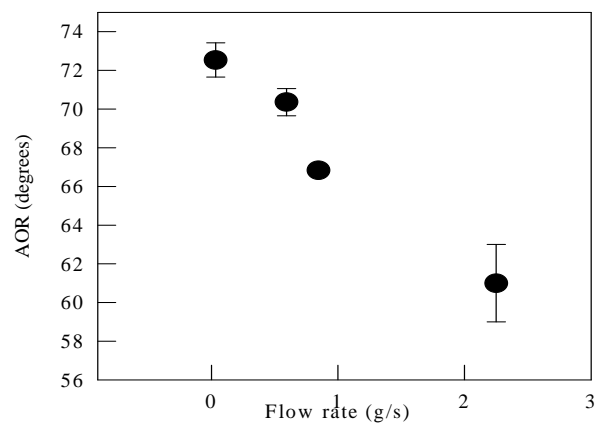
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AOR versus flow rate



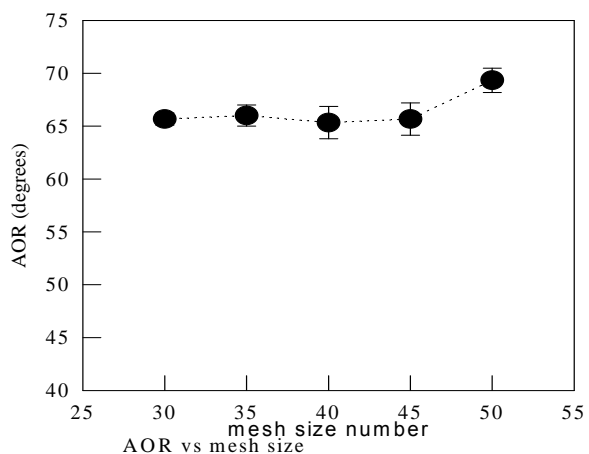
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AOR versus outlet mesh size



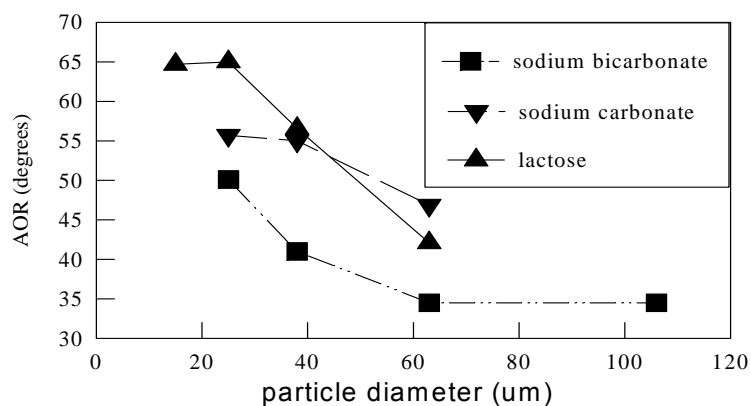
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AOR for different powders as a function of particle size



Angle of repose versus particle size



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Comparison between MAPF and a Mechanically Vibrated Hopper

Mass flow rate = 2.6 g/s		
Height of hopper above surface = 6 ins. (15.24 cm)		
<i>Mass of Cornstarch</i>	<i>AOR MAPF (degrees)</i>	<i>AOR Vibrated Hopper (degrees)</i>
5 grams	58	56
10 grams	62	65

Values obtained for experiment comparing AOR for vibrated hopper and MAPF hopper.



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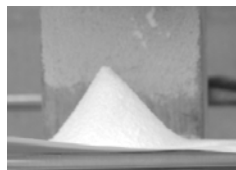
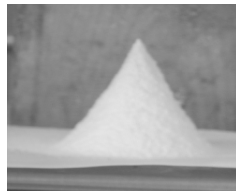


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Comparison of piles obtained using MAPF and Vibration

Piles using MAPF



Piles using Hosakawa Micron Tester



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Summary

- ❑ An **AOR measurement device** based on Magnetically Assisted Powder Flow System allows for “erasing the memory of the previous state”
- ❑ It provides **clean heaps with sharp boundaries**, thus gives precise values for AOR, eliminating false peaks
- ❑ It is **easy to operate** thereby reducing errors due to inexperienced operators
- ❑ High degree of **reproducibility of results** is obtained
- ❑ Tests require very **little time**



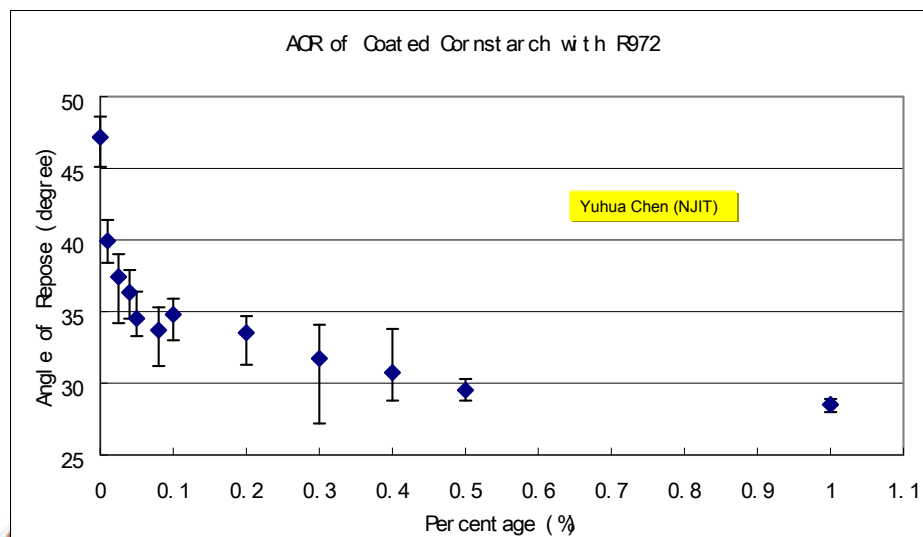
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Angle of repose (AOR) to indicate flow improvements from nano-additives



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Carr Indices (Carr, 1965)

- Two indices: “flowability” and “floodability”
- Flowability is a measure of a powder’s ability to flow freely.
- Floodability is a measure of a powder’s tendency to fluidize in air and produce liquid-like flow.
- Measured using the Hosokawa Powder Tester



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Hosokawa Powder Tester

The Hosokawa powder tester is a multi-test apparatus, that measures:

- Cohesion Index: Defined as the relative amounts of solid remaining in three sieves of 74, 149 and 250 μm meshsize after sieving for a definite time.
- Angle of Spatula: Obtained taken upwards a plate immersed in the powder (i.e the angle of repose at the plate).



- Angle of Fall
- Angle of Repose
- Compressibility (i.e. Carr Index)
- Aerated Bulk Density and Packed Bulk Density
- Uniformity: characterizes the width of the particle size distribution

Flowability is determined from a combination of all results.

According to Schwedes, it can only be used as a very rough classification of bulk solids behavior.

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Carr Indices

- Carr's flowability index is an empirically derived score that combines measurements of a powder's:
 - angle of repose
 - compressibility
 - angle of spatula, and
 - either cohesion or coefficient of uniformity
- $0 \leq \text{Flowability} \leq 100$ with 100 indicating excellent flow qualities



Carr Indices...

- Compressibility is the relative change in bulk density of a powder that is “tapped” and “aerated”:

$$\text{compressibility} = \frac{\rho_T - \rho_A}{\rho_T}$$

where ρ_A and ρ_T are the aerated and tapped bulk densities (density = mass of the powder/total volume occupied by the powder as the bulk or bed)

- The aerated bulk density, ρ_A , is the bulk density of the powder sifted into a containing vessel
 - in practice ρ_A is the most loosely packed bulk density rather than an aerated density



Tests based on density

Tests that measure the density are based on the fact that in cohesive powders, the attractive forces between particles can stabilise arrangements of particles with solid fractions smaller than those found in non-cohesive powders.

Because of this, if a cohesive powder is gently poured in a container, its solid fraction (defined as the ratio of the volume of the powder or solid and the total volume occupied by the powder bed) ϕ_{loose} would be on the lower end of the range attainable for that powder.

If the container is tapped, the acceleration on the taps causes a consolidation stress on the powder that rearranges the particles into a larger solid fraction ϕ_{tapped} .

The result of the test is usually given in terms of the tapped solid fraction ϕ_{tapped} , the ratio $\phi_{\text{tapped}}/\phi_{\text{loose}}$ (Housner ratio) or $(\phi_{\text{tapped}} - \phi_{\text{loose}})/\phi_{\text{tapped}} \times 100$ (Carr index).

Typically, the higher $\phi_{\text{tapped}}/\phi_{\text{loose}}$ and the lower ϕ_{tapped} the poorer is the flowability of the powder



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Tapped density- Caution

- The tapped bulk density, ρ_T , is the bulk density obtained while subjecting the powder to a prescribed series of taps.
 - The tapped bulk density has been shown to increase with the number of taps (Yu and Hall, 1994). For example:

$$\rho_n = \rho_\infty - (\rho_\infty - \rho_0) \exp\left(-n/T\right)$$

where ρ_n is the bulk density after n taps, ρ_∞ is the asymptotic bulk density, ρ_0 is the initial bulk density, and T is a time constant

- The tapped bulk density is also a function of the tapping style (Abdullah and Geldart, 1999).



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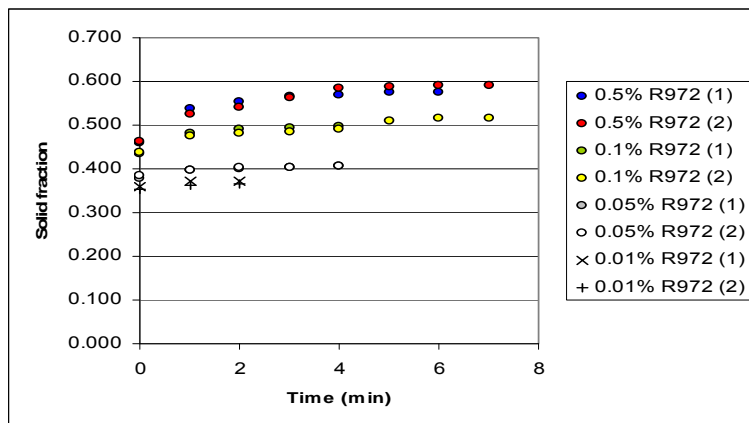


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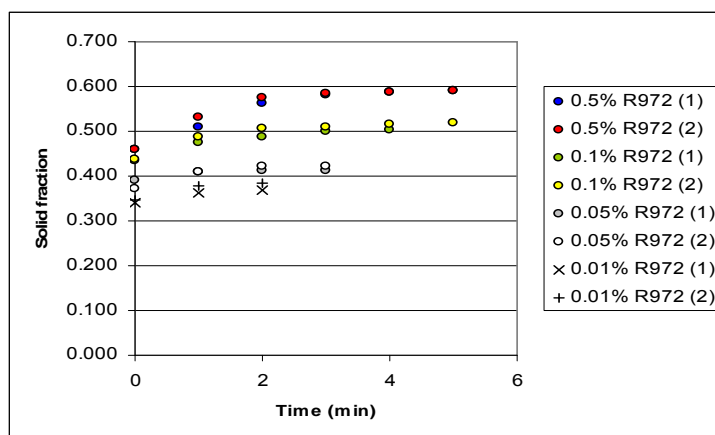
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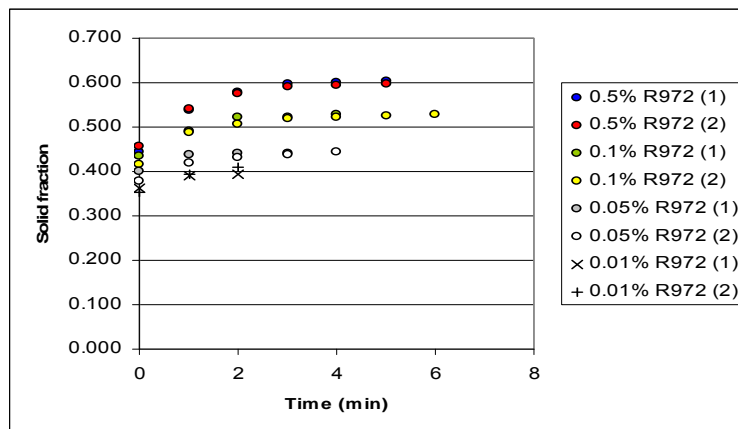
Amplitude=0.025mm



Amplitude=0.05mm



Amplitude=0.075mm



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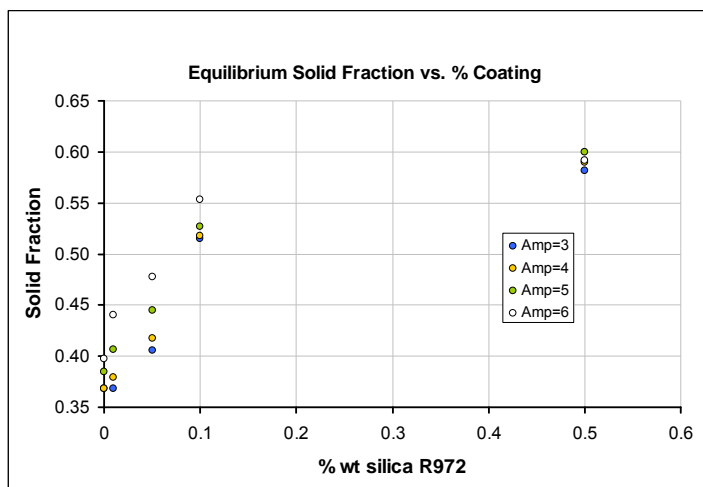


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Flow Improvement Correlates Well with Packing Density (Vibrated Packing)



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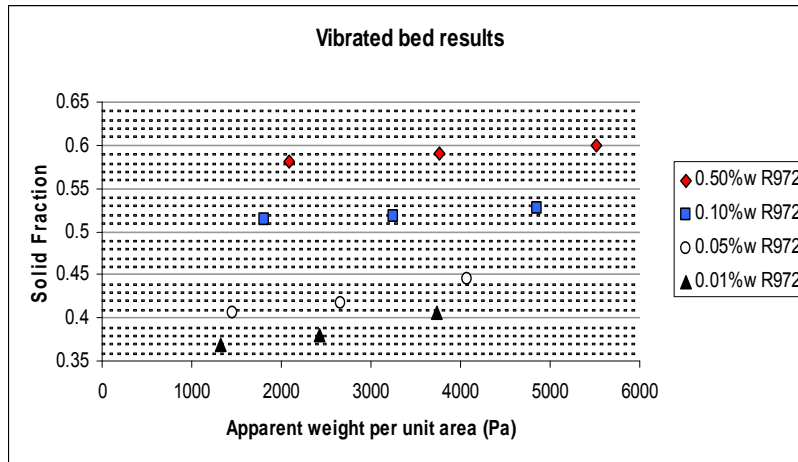


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Principal Contributor: Beach

Another Representation of the Vibrated Bed Packing Results



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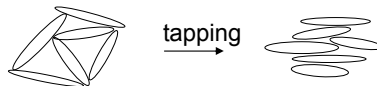
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Principal Contributor: Beach

Carr Indices...

- Powders with significant frictional and cohesive interactions (which tend to decrease flowability) will have a larger difference in their tapped and aerated bulk densities.



- Increasing compressibility generally implies decreasing flowability (table from Hiestand, 1991):

Subjective Flowability	Compressibility [%]
excellent	5 – 10
good	11 – 15
fair	16 – 20
passable	21 – 25
poor	26 – 31
very poor	32 – 37
exceeding poor	38 – 45



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Principal Contributor: Wassgren

Carr Indices...

- Carr quantified a powder's cohesion by measuring the mass of powder retained on three vibrating, stacked screens.
 - The screen mesh sizes are a function of the powder's working bulk density ($\rho_W = \rho_A + (\rho_T - \rho_A) \cdot \text{compressibility}$):
 - $160 < \rho_W < 400 \text{ kg/m}^3$: 40, 60, 100 mesh
 - $400 < \rho_W < 960 \text{ kg/m}^3$: 60, 100, 200 mesh
 - $960 < \rho_W < 1440 \text{ kg/m}^3$: 100, 200, 325 mesh
 - Start with 2 g of -200 mesh material on the top screen.
 - The vibration duration increases with decreasing ρ_W .
 - The cohesion of a powder is given by:

$$\text{cohesion} \equiv 5n_1 + 3n_2 + n_3 \quad \text{where} \quad \begin{aligned} n_1 &= \text{floor}((\text{mass on top screen})/0.1 \text{ g}) \\ n_2 &= \text{floor}((\text{mass on middle screen})/0.1 \text{ g}) \\ n_3 &= \text{floor}((\text{mass on bottom screen})/0.1 \text{ g}) \end{aligned}$$



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Decreasing cohesion implies increasing flowability.

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Principal Contributor: Wassgren

Carr Indices...

- The uniformity coefficient is used in place of cohesion for larger sized (*i.e.* granular or coarse) materials.
- Uniformity is defined as the ratio of the width of sieve opening that passes 60% of the material (by mass) to the width of sieve opening that passes 10% of the material.

$$\text{uniformity} \equiv \frac{d_{60}}{d_{10}}$$

- Increasing uniformity implies increasing flowability.



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Carr Indices...

- The flowability index is determined from an empirical formula based on the behavior of >2,800 dry materials (Carr, 1965).

Flowability and Performance	Angle of Repose		Compressibility		Angle of Spatula		Uniformity Coeff.*		Cohesion**	
	Deg.	Points	%	Points	Deg.	Points	Units	Points	%	Points
Excellent, 90 - 100 pts	25	25	5	25	25	25	1	25		
Aid not needed	26 - 29	24	6 - 9	23	26 - 30	24	2 - 4	23		
Will not arch	30	22.5	10	22.5	31	22.5	5	22.5		
Good, 80 - 89 pts	31	22	11	22	32	22	6	22		
Aid not needed	32 - 34	21	12 - 14	21	33 - 37	21	7	21		
Will not arch	35	20	15	20	38	20	8	20		
Fair, 70 - 79 pts	36	19.5	16	19.5	39	19.5	9	19		
Aid not needed (but vibrate if necessary)	37 - 39	18	17 - 19	18	40 - 44	18	10 - 11	18		
	40	17.5	20	17.5	45	17.5	12	17.5		
Passable, 60 - 69 pts	41	17	21	17	46	17	13	17		
Borderline. Material may hang up	42 - 44	16	22 - 24	16	47 - 59	16	15 - 16	16		
	45	15	25	15	60	15	17	15	< 6	15
Poor, 40 - 59 pts	46	14.5	26	14.5	61	14.5	18	14.5	6 - 9	14.5
Must agitate, Vibrate	47 - 54	12	27 - 30	12	62 - 74	12	19 - 21	12	10 - 29	12
	55	10	31	10	75	10	22	10	30	10
Very poor, 20 - 39 pts	56	9.5	32	9.5	76	9.5	23	9.5	31	9.5
Agitate more positively	57 - 64	7	33 - 36	7	77 - 89	7	24 - 26	7	32 - 54	7
	65	5	37	5	90	5	27	5	55	5
Very, very poor, 0 - 19 pts	66	4.5	38	4.5	91	4.5	28	4.5	56	4.5
Special agit., hopper or eng'g	67 - 89	2	39 - 45	2	92 - 99	2	29 - 35	2	51 - 79	2
	90	0	> 45	0	> 99	0	> 36	0	> 79	0

* Used with granular and powdered granular materials.

** Used with powders or where an effective cohesion can be measured.

Principal Contributor: Wassgren

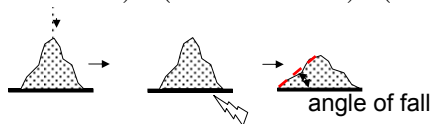
Carr Indices...

- Floodability is an empirically derived score that combines measurements of a powder's:
 - flowability
 - angle of fall
 - angle of difference
 - dispersibility
- $0 \leq \text{floodability} \leq 100$ with 100 indicating the material is very floodable

Carr Indices...

- Angle of difference is the difference between the angle of repose and the angle of fall.

$$(\text{angle of difference}) \equiv (\text{angle of repose}) - (\text{angle of fall})$$



- A floodable material typically has a small angle of fall since air trapped within the heap of powder causes the material to fluidize when the base is impacted.
- The larger the angle of difference, the more likely the material will be floodable.



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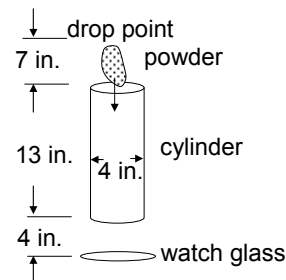
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Principal Contributor: Wassgren

Carr Indices...

- Dispersibility is a measure of the scattering and dusting characteristics of a powder.
 - The more dispersible a material is, the more floodable it is.
- Dispersibility is measured by dropping a 10 g sample of material en masse through a 4 in. diameter, 13 in. long cylinder from a height of 24 in. above a watch glass (which in turn is located 4 in. from the bottom of the cylinder). The material remaining on the watch glass is weighed and the dispersibility is given by:



$$\text{dispersibility} \equiv 10 * [10 - (\text{mass remaining, in grams})]$$



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Principal Contributor: Wassgren

Carr Indices...

- The floodability index is determined from an empirical formula (Carr, 1965).

Floodability and Performance	Flowability		Angle of Fall		Angle of Difference		Dispersibility	
	Units	Points	Deg.	Points	Deg.	Points	%	Points
Very floodable, 80 - 100 pts	60+	25	10	25	30+	25	50+	25
Positive rotary seal	59 - 56	24	11 - 19	24	29 - 28	24	49 - 44	24
will be necessary	55	22.5	20	22.5	27	22.5	43	22.5
	54	22	21	22	26	22	42	22
	53 - 50	21	22 - 24	21	25	21	41 - 36	21
	49	20	25	20	24	20	35	20
Floodable, 60 - 79 pts.	48	19.5	26	19.5	23	19.5	34	19.5
Rotary seal will	47 - 45	18	27 - 29	18	22 - 20	18	33 - 29	18
be necessary	44	17.5	30	17.5	19	17.5	28	17.5
	43	17	31	17	18	17	27	17
	42 - 40	16	32 - 39	16	17 - 16	16	26 - 21	16
	39	15	40	15	15	15	20	15
Inclined to flood, 40 - 59 pts	38	14.5	41	14.5	14	14.5	19	14.5
Rotary seal is desirable	37 - 34	12	42 - 49	12	13 - 11	12	18 - 11	12
	33	10	50	10	10	10	10	10
Could flood, 25 - 39 pts	32	9.5	51	9.5	9	9.5	9	9.5
Rotary seal probably needed	31 - 29	8	52 - 56	8	8	8	8	8
depending on drop, velocity	28	6.25	57	6.25	7	6.25	7	6.25
Won't flood, 0 - 24 pts	27	6	58	6	6	6	6	6
Rotary seal will	26 - 23	3	59 - 64	3	5 - 1	3	5 - 1	3
not be needed	< 23	0	> 64	0	0	0	0	0



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Carr Indices...

- Standards related to measuring Carr Indices:
 - ASTM D6393-66: Standard Test Method for Bulk Solids Characterization by Carr Indices
 - ASTM D6683-01: Standard Test Method for Measuring Bulk Density Values of Powders and Other Bulk Solids
 - USP 24 / NF 19: <616> Bulk and Tapped Density



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Hausner Ratio (Hausner, 1967)

- Perhaps the most commonly used quantities to determine flowability
- Defined as the ratio of the tapped bulk density to the aerated bulk density:

$$\text{Hausner ratio} \equiv \frac{\rho_T}{\rho_A}$$

- Directly related to the compressibility:

$$\text{HR} \equiv \frac{\rho_T}{\rho_A} = \frac{1}{1 - \frac{\rho_T - \rho_A}{\rho_T}} = \frac{1}{1 - (\text{compressibility})}$$



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Hausner Ratio...

- | Subjective Flowability | Compressibility [%] | Hausner Ratio |
|------------------------|---------------------|---------------|
| excellent | 5 – 10 | 1.05 – 1.11 |
| good | 11 – 15 | 1.12 – 1.18 |
| fair | 16 – 20 | 1.19 – 1.25 |
| passable | 21 – 25 | 1.27 – 1.33 |
| poor | 26 – 31 | 1.35 – 1.45 |
| very poor | 32 – 37 | 1.47 – 1.59 |
| exceedingly poor | 38 – 45 | 1.61 – 1.82 |

(Table adapted from Hiestand, 1991.)
- HR can also be used to distinguish between Geldart Group A (aeratable, easy-to-fluidize, HR < 1.25) and Group C (cohesive, difficult-to-fluidize, HR > 1.4) powders. Powders with 1.25 < HR < 1.4 are Group AC (transitional) powders (Geldart *et al.*, 1984).



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Principal Contributor: Wassgren

Hausner Ratio...

- The Hausner ratio (*i.e.* compressibility) has been known to give erroneous flowability results:
 - If the cohesion strength is greater than the tapping strength, the initial packing arrangement may not change \Rightarrow HR small indicating good flowability when in fact the flowability is poor (Li *et al.*, 2004; Lavoie *et al.*, 2002). Example: MCC 103
 - If the cohesion is significant, the initial packing may already have a large initial bulk density so tapping won't decrease it significantly \Rightarrow HR small indicating good flowability when in fact the flowability is poor (Li *et al.*, 2004).
 - Non-cohesive, angular particles may pack loosely initially but small perturbations result in significant re-packing \Rightarrow HR large indicating poor flowability when in fact the flowability is good (Li *et al.*, 2004). Example: pre-gelatinized starch
 - Other erroneous results: stearic acid (HR indicates poor flowability when in fact the flowability is very poor) (Li *et al.*, 2004)



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Principal Contributor: Wassgren

Hausner Ratio...

Material	Hausner Ratio	Flowability
Glass beads	1.04 (Lavoie <i>et al.</i> , 2002)	excellent
Sucrose	1.10 (Lavoie <i>et al.</i> , 2002)	excellent
Povidone	1.13 (Li <i>et al.</i> , 2004)	good
Sodium chloride	1.15 (Lavoie <i>et al.</i> , 2002)	good
MCC 103	1.19 (Lavoie <i>et al.</i> , 2002)	fair
Lactose 100	1.20 (Lavoie <i>et al.</i> , 2002)	fair
MCC 105	1.22 (Lavoie <i>et al.</i> , 2002)	passable
Lactose 300	1.23 (Lavoie <i>et al.</i> , 2002)	passable
Maltodextrin	1.24 (Lavoie <i>et al.</i> , 2002)	passable
Avicel PH-102	1.26 (Schussele and Bauer-Brandl, 2003)	poor
Avicel PH-101	1.28 – 1.29 (Sinka <i>et al.</i> , 2004)	poor
Avicel PH-102	1.29 (Sinka <i>et al.</i> , 2004)	poor
Starch 1500	1.29 (Schussele and Bauer-Brandl, 2003)	poor
Paracetamol	1.39 (Li <i>et al.</i> , 2004)	exceedingly poor
Magnesium stearate	1.39 (Li <i>et al.</i> , 2004)	exceedingly poor
Lactose 200	1.41 (Lavoie <i>et al.</i> , 2002)	exceedingly poor



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Principal Contributor: Wassgren

Hausner ratio/Compressed Bulk Density

•Thalberg et al. Powder Technology 146 (2004) 206–213

Apparatus: GeoPyc 1360 from Micromeritics

Materials used:

Ordered mixtures of a carrier (Pharmatose 325M, $d_p \approx 50 \mu\text{m}$), intermediated sized particles (Pharmatose 450M, $d_p \approx 20 \mu\text{m}$) and micronized lactose ($d_p \approx 2 \mu\text{m}$)

Composition of ordered mixtures

Denomination	Pharmatose 325M, % w/w	Pharmatose 450M, % w/w	Micronized lactose, % w/w
[100/0/0] (Pure carrier)	100	0	0
[95/5/0]	95	5	0
[95/2.5/2.5]	95	2.5	2.5
[90/7.5/2.5]	90	7.5	2.5
[95/0/5]	95	0	5
[90/5/5]	90	5	5
[90/2.5/7.5]	90	2.5	7.5
[85/7.5/7.5]	85	7.5	7.5
[80/10/10]	80	10	10

Conclusions:

- ✓ Poured bulk density decreases with increased amounts of lactose.
- ✗ If density is measured after a compression of 35 KPa, the values are larger than the tapped density. Hausner Ratio is calculated as compressed bulk density (CBD) over poured density.
- ✓ The Hausner Ratio discriminates well between the investigated mixtures, increasing with and increase of micronized particles
- ✗ The Hausner Ratio levels out at about 1.5 for the most cohesive powders
- ✓ Comparing the measured density with the theoretical density of the mixture gives insight into the arrangements of the particles



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Hausner Ratio

•Mullarney et al. International Journal of Pharmaceutics 257 (2003) 227–236

Apparatus: VanKel tapping device fitted with a 100 ml glass measuring cylinder

Materials used:

Material	Mean volume diameter (μm)	10th % size (μm)	50th % size (μm)	90th % size (μm)
Sucrose	632	424	629	851
Acesulfame potassium	338	61	294	683
Aspartame	12	1	6	26
Saccharin sodium	20	2	11	49

Conclusions:

- ✓ Solid fraction measurements can discriminate the effect of the size and shape of the particles: large rounded shaped particles can be tapped to a much higher solid fraction than the small irregularly shaped particles
- ✓ Results are comparable to those obtained with the Aeroflow, although the rank order for both methods is not the same



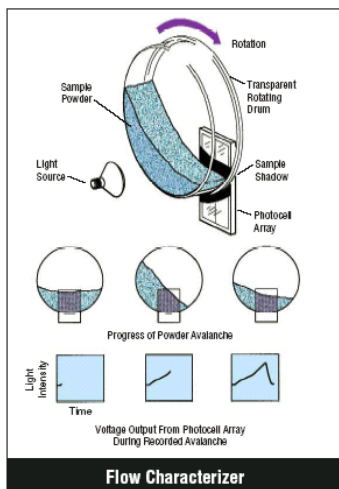
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Avalanche Flowability Index



- Powder sample placed inside a slowly rotating drum
 - Avalanches detected photoelectrically
 - Measures mean avalanche time and scatter in avalanche time
 - Typical drum diameter = 150 mm and rotation speed = $\frac{1}{3} - \frac{1}{2}$ rpm (typically just test at one speed)
 - Operator independent \Rightarrow more repeatable
 - Powder is in a dynamic, rather than static, state



Figure from the TSI Aero-Flo Powder Flowability Analyzer Model 3250 Specifications Sheet.

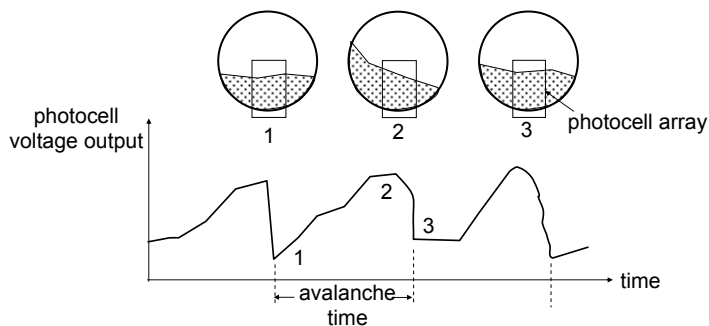


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Avalanche Flowability Index...



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Avalanche Flowability Index...

- Shorter, more reproducible avalanche times \Rightarrow better flowability
- Smaller scatter \Rightarrow smaller cohesion
- Lavoie *et al.* (2002) propose testing at multiple drum speeds and define the following indices:

$$\text{Flowability Index} \equiv \frac{1}{n} \sum_{i=1}^n \sigma_i$$

$$\text{Cohesion Index} \equiv \frac{1}{n} \sum_{i=1}^n m_i$$

where n is the number of different speeds tested, σ_i is the standard deviation of the time between avalanches at speed test i , and m_i is the mean time between avalanches at speed test i

- The time between avalanches is proportional to the drum speed (Lavoie *et al.*, 2002) so the proposed indices are artificially weighted toward the slower speed values. The indices would be more useful if the time between avalanches were normalized by the drum rotation period before calculating standard deviations and means.



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Principal Contributor: Wassgren

Avalanche Flowability Index...

- Issues (Thalberg *et al.*, 2004):
 - Need to use a rough boundary surface (e.g. sand paper or mesh) to prevent slipping at the drum walls
 - Need to minimize electrostatic forces at the observation windows to prevent erroneous light obscuration due to sticking particles
 - Gives errors if agglomerates form since the time between agglomerate avalanches rather than particle avalanches
 - Not recommended for cohesive powders.



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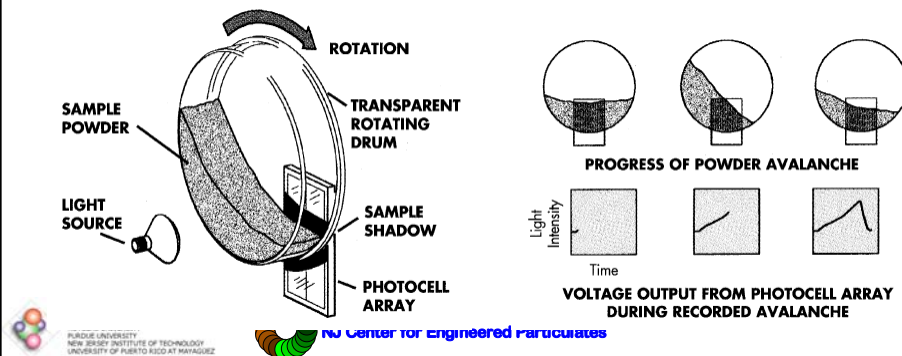
Principal Contributor: Wassgren

Tests based on avalanches

The tests based on avalanches characterise the flowability of a powder by measuring the time interval between avalanches in a rotating drum.

The shorter the time between avalanches, the better the flowability of the powder

Example: Aeroflow®, TSI Instruments



Review on results using the Aeroflow (I)

•Hancock et al. *Journal of Pharmaceutical and Biomedical Analysis* 35 (2004) 979–990

Materials used:

	Particle morphology	True density (g ml ⁻¹)	Median particle size (μm)	10th percentile size (μm)	90th percentile size (μm)
Ascorbic acid	Equant crystals	1.66	249	33	479
Citric acid	Equant crystals	1.60	388	196	585
Croscarmellose sodium	Elongated twisted fibres	1.55	42 ^a	18 ^a	114 ^a
Hydroxypropyl cellulose	Fragmented fibres	1.21	85	12	247
Lactose anhydrous	Equant crystals	1.50	136	12	324

^a Particle size parameters for this material are only approximate because of the elongated nature of the particles.

Conclusions:

- ✓ For cohesive powders, the numbers of avalanches per time was less than that of less cohesive powders.
- ✓ Positive correlation with the results of a benchmark method (the simplified shear cell)
- ✗ However, the average time between avalanches and the width of the distributions of the avalanche times do not arrange the materials in the same order from more cohesive to less cohesive

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Principal Contributor: Sanchez-Quintanilla

Review on results using the Aeroflow (II)

•Lee et al. *AAPS PharmSciTech*, 2000; 1 (3) article 21

Materials used:

Microcrystalline cellulose. $d_v = 70.51 \mu\text{m}$ (Avicel PH 101®, $d_v = 70.51 \mu\text{m}$,
Avicel PH 102®, $d_v = 115.03 \mu\text{m}$
Lactose monohydrate crystals $d_v = 144.21 \mu\text{m}$
Lactose monohydrate "Fast-Flo" $d_v = 99.29 \mu\text{m}$
Pregelatinized maize starch $d_v = 77.84 \mu\text{m}$
Calcium phosphate, dibasic anhydrous $d_v = 14.72 \mu\text{m}$

Conclusions:

✓ The powders exhibited four behaviors: rolling (Flow category 1), slumping (FC2), slipping (FC3), cataracting (FC4). As the value of the flow category increased, the powder flow became worse.

✗ Determination of flow properties cannot be based solely on strange attractor plots, mean time to avalanche or scatter. A combination of visual observation of the type of motion with the numerical values appears more accurate.

✓ The flow behaviour determined with the aeroflow and with the critical orifice diameter method and Carr compressibility tend to show the same



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Principal Contributor: Sanchez-Quintanilla

Review on results using the Aeroflow (III)

•Thalberg et al. *Powder Technology* 146 (2004) 206–213

Materials used:

Ordered mixtures of a carrier
(Pharmatose 325M, $d_p \approx 50 \mu\text{m}$),
intermediated sized particles
(Pharmatose 450M, $d_p \approx 20 \mu\text{m}$)
and micronized lactose ($d_p \approx 2 \mu\text{m}$)

Composition of ordered mixtures

Denomination	Pharmatose 325M, % w/w	Pharmatose 450M, % w/w	Micronized lactose, % w/w
[100/0/0] (Pure carrier)	100	0	0
[95/5/0]	95	5	0
[95/2.5/2.5]	95	2.5	2.5
[90/7.5/2.5]	90	7.5	2.5
[95/0/5]	95	0	5
[90/5/5]	90	5	5
[90/2.5/7.5]	90	2.5	7.5
[85/7.5/7.5]	85	7.5	7.5
[80/10/10]	80	10	10

Conclusions:

✓ Short and reproducible times between avalanches indicate a good flowability, while long and/or irregular times indicate poor flowability

✗ Care must be taken to avoid sliding of powder and powder adhering to the glass walls, which give false avalanches.

✗ The AeroFlow is suitable for ordered mixtures with 5% micronized lactose or less, but cannot discriminate between the more cohesive powders.

✗ Cohesive powders may display short times between avalanches due to aggregate formation and thereby wrongly be assessed as having good flow.



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Principal Contributor: Sanchez-Quintanilla

Review on results using the Aeroflow (IV)

•Lindberg et al, Drug Development and Industrial Pharmacy, Vol. 30, No. 7, pp. 785–791, 2004

Materials used:

Batches containing approximately 15% of active pharmaceutical ingredient (API), fillers, binders, and lubricant (2%)

The author does not give details about the components

Conclusions:

- ✓ An index of the bulk flow of the material is provided by the time between avalanches.
- ✓ The scatter of the data provides an index of the cohesivity, which is related to the irregularity of the flow.
- ✓ Short and reproducible times between avalanches, i.e., low mean time and scatter, indicate better flow properties.
- ✓ The rank order correlation was similar with all the tested techniques (Hausner ratio, uniaxial tester, powder rheometer, Jenike tester) and reflected the behavior during processing of the powder mixtures



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Principal Contributor: Sanchez-Quintanilla

Rheometers

Rheometers characterize the powder flowability by measuring the force, torque or powder needed to maintain the movement of a impeller in the powder

Example: Freeman FT4 Powder Rheometer®

- The FT4 forces a twisted blade along a helical path through a powder sample, causing a flow inside the powder.



- Samples are prepared for testing by a conditioning process in which the blade causes gentle displacement of the powder to establish a reproducible packing density.

- In a test, the blade digs into the sample compressing the powder. The axial and rotational forces acting on the blade are measured continuously to derive the work done, or energy consumed, in displacing the powder.

- The basic flowability energy (BFE) is defined as the energy required to complete a standard test and it is regarded as a measure of the rheological properties of the powder.



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Principal Contributor: Sanchez-Quintanilla

(Freeman) Powder Rheometer

- FT4 Powder Rheometer
- Rotating blade moved helically in a cylinder containing powder
- Measure energy required to drive the blade
 - “The basic flowability energy [BFE] is therefore the energy required to displace a constant volume [of] conditioned powder at a given flow pattern and flow rate.”
(<http://www.freemantech.co.uk/>)
- Other related measures:
 - stability index: the factor by which the BFE changes during repeated testing
 - flow rate index: The factor by which the energy requirement is changed when the flow rate is reduced by a factor of 10.
 - compaction index: The factor by which the BFE is increased when the powder is consolidated.



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aeration ratio: the factor by which the BFE is reduced by aeration.

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Principal Contributor: Wassgren

Review on results on Rheometers

•Lindberg et al, Drug Development and Industrial Pharmacy, Vol. 30, No. 7, pp. 785–791, 2004

Apparatus: Freeman FT4 Powder Rheometer

Materials used:

Batches containing approximately 15% of active pharmaceutical ingredient (API), fillers, binders, and lubricant (2%)

The author does not give details about the components

Conclusions:

✗ There was a noticeable change in the rheological behavior if the test was repeated several times, although results are reproducible using fresh samples

✗ For some samples results were rate independent while for others not

✓ The basic flowability index changes with the density of the sample when it is compacted or aerated, reflecting the change in rheological properties with the consolidation acting on the sample

✓ Rank order of the materials according to flowability in agreement with other techniques



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Principal Contributor: Sanchez-Quintanilla

Review on results on Rheometers

•Navaneethan et al. AAPS PharmSciTech 2005; 6 (3)

Apparatus: ManUmit Powder Rheometer combined with Texture Technologies TA.XT2i Texture Analyzer

Materials used:

Metronidazole, colloidal bismuth citrate, and tetracycline hydrochloride, as active ingredients

Excipient: microcrystalline cellulose Binder: PVP and croscarmellose sodium

Lubricant: magnesium stearate at different concentrations

Conclusions:

- ✗ For powders with large amounts of entrapped air or coarse characteristics, the correlation between basic flowability index and flowability may fail
- ✓ Apart from the basic flowability index, the presence of peaks in the torque acting on the blade and its frequency is an indication of powder flowability
- ✓ The test can discriminate the effect of different amounts of lubrication and give an optimum concentration of lubricant



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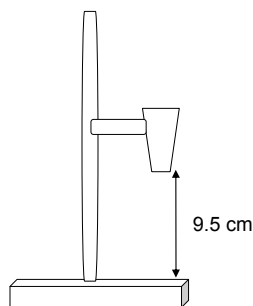
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Principal Contributor: Sanchez-Quintanilla

Hall Flow Meter

The Hall Flow meter measures the flowability of a powder measuring the rate of discharge Q of the powder from funnels with different orifice size



Typically, the higher the rate of discharge Q the better the flowability of the powder



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Principal Contributor: Sanchez-Quintanilla

Flow Through an Orifice...

- Similar, but more simplified, than the Jenike minimum outlet diameter analysis.
- Does not represent powder behavior under dynamic conditions (Lavoie *et al.*, 2002)
- A flowing powder could become non-flowing when forced through small openings (Lavoie *et al.*, 2002)

Material	Minimum Diameter for Flow [mm]	
Sucrose	< 4	(Lavoie <i>et al.</i> , 2002)
Sodium chloride	5	(Lavoie <i>et al.</i> , 2002)
Lactose 100	17	(Lavoie <i>et al.</i> , 2002)
Lactose 325	19	(Lavoie <i>et al.</i> , 2002)
Lactose 200	29	(Lavoie <i>et al.</i> , 2002)
MCC 105	> 34	(Lavoie <i>et al.</i> , 2002)



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Principal Contributor: Wassgren

Funnel Flow

- Schüssele *et al.* International Journal of Pharmaceutics 257 (2003) 301–304

Apparatus: Sotax Powder Flow Tester FT 300 and European Pharmacopoeia's funnel based flowability test

Materials used:

Tablettose™ 80, Tablettose™ 100, Fujicalin™, Emcompress™ Dihydrat, Avicel™ PH-102, Starch 1500™,

Conclusions:

- ✗ No significant difference found between free flowing materials
- ✓ Vibrating the funnel can be used to induce flow in the most cohesive materials, that otherwise can not be tested
- ✗ Due to widely different bulk densities of powders, the expression of flowability in terms of time per mass may in some cases not match the macroscopic flow qualities. It is proposed that "volume-flowability" is a better description



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Principal Contributor: Sanchez-Quintanilla

Shear Cells

- Used to measure a powder's yield strength as a function of the pressure used to compact the powder.
- Usually performed as a quasi-static test, *i.e.* at incipient yield
- Measurements can be used in design, not simply for comparisons
- Typically used in the design of hoppers and bins



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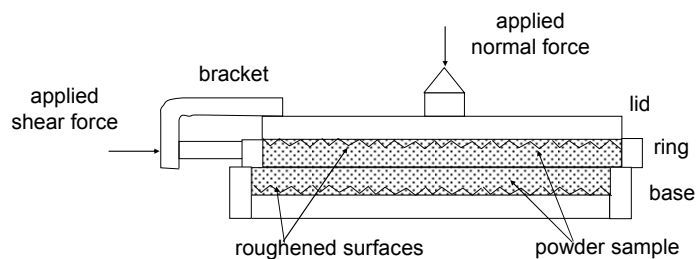
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Shear Cells...

- An example: The Jenike Shear Cell



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Shear Cells...

- Procedure:
 - Base and ring assemblies are filled and the lid is placed on the particulate material. A consolidating load, resulting in a stress of σ_c , is applied to the lid in order to compress the material. The material now has a bulk density, ρ_b .
 - The consolidating lid is removed and a new normal load is applied. The material is now sheared by applying a shear force to the lid/ring assembly. The material may expand or contract depending on the applied normal load. The normal load under which the material volume does not change (referred to as the end-point load) and the corresponding shear stress are noted.
 - The shear cell is emptied and a new sample is prepared following the procedure outlined in the first step using the same consolidating load. Now a new normal load, which is less than the end-point load, is applied to the lid and the shear stress required to just shear the material is noted. This procedure is repeated several times for different applied normal loads; all of which are smaller than the consolidating load.



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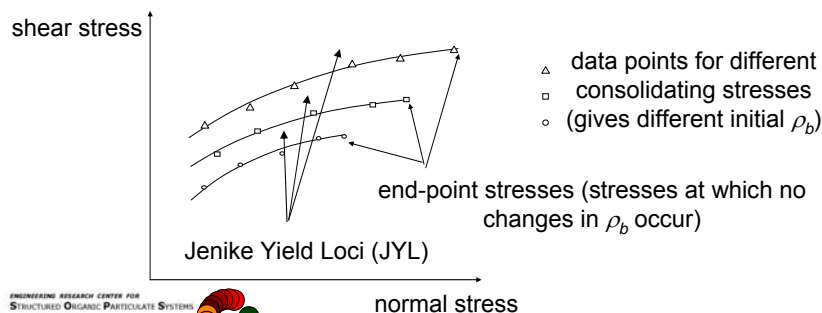
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Principal Contributor: Wassgren

Steps are repeated for different consolidating loads.

Shear Cells...

- Jenike Yield Loci (JYL) are the values of the shear stress required to initiate movement in the material as a function of the applied normal stress for different consolidating stresses.



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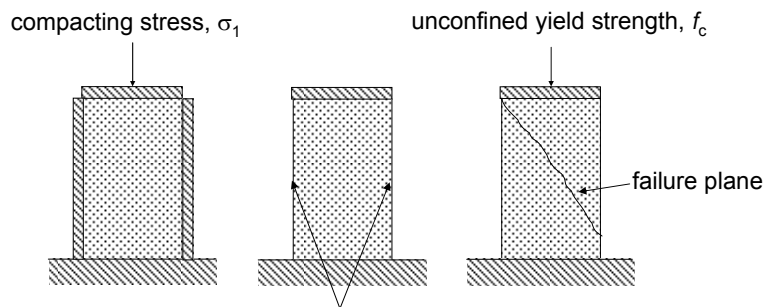
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Shear Cells...

- The unconfined yield strength, f_c , is the maximum stress that a powder's free surface can withstand before failing (i.e. flowing).



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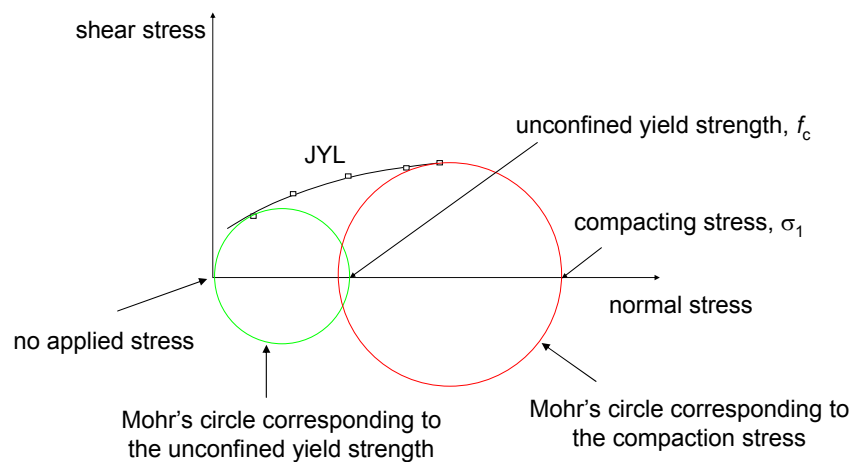


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Shear Cells...



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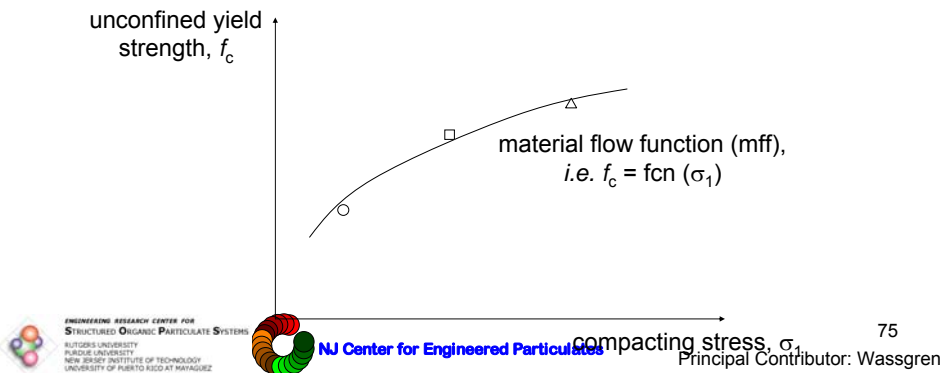
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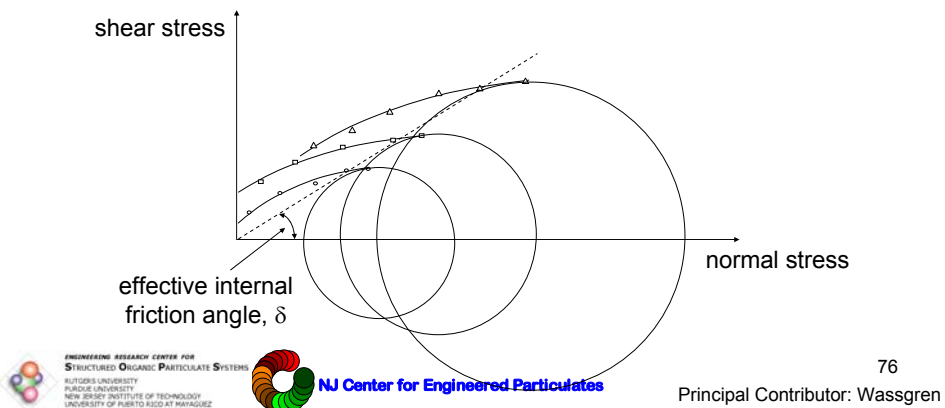
Shear Cells...

- A material flow function (mff) is the relationship between a powder's unconfined yield strength, f_c , and the compacting stress, σ_1 .



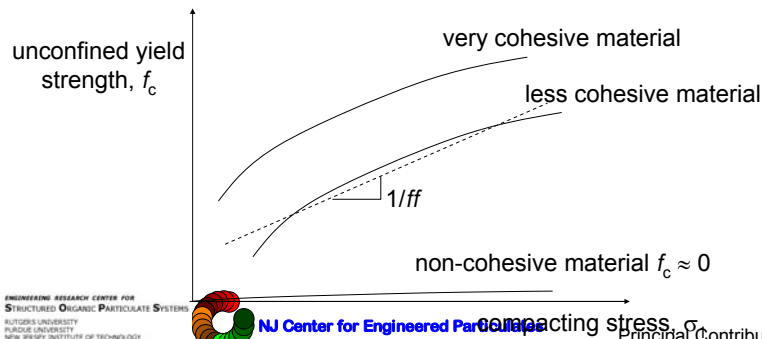
Shear Cells...

- The effective internal friction angle, δ , is the angle of the line that is tangent to the end-point load Mohr's circles



Shear Cells...

- The flow function, ff , is the inverse of the slope of the mff : $ff = \sigma_1/f_c$
 - $ff < 1$ hardened
 - $1 < ff < 2$ very cohesive
 - $2 < ff < 4$ cohesive
 - $4 < ff < 10$ easy flowing
 - $10 < ff$ free flowing



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Principal Contributor: Wassgren

Review on results from Shear Testers (I)

- Ramachandruni et al. Journal of Pharmaceutical Sciences, vol. 90, no. 5, 2001

Apparatus: custom made annular shear cell.

Materials used:

Powder density ($\text{g} \cdot \text{cm}^{-3}$)

Measured magnitudes:

Cohesion, angle of internal friction ϕ , effective angle of internal friction δ , flow factor FF and shear index n .

Conclusions:

- ✗ In some cases it was found that the ranking of powders was different when different indices like δ , FF, and n were used.
- ✗ The ranking of powders using shear analysis was different from other flow methods (Carr index, funnel flow).
- ✗ For this shear cell, instrument and process parameters are of significant importance and need to be standardized

Sample	Mean volume diameter (μm)	Particle shape	Powder density ($\text{g} \cdot \text{cm}^{-3}$)		
			True	Bulk	Tap
Avicel PH-101	52	rod shaped	1.65	0.31	0.45
Avicel PH-102	96	round	1.66	0.30	0.42
Avicel PH-200	209	agglomerates	1.64	0.35	0.46
		round			
Anh. Lactose	124	irregular	1.66	0.50	0.75
Emcompress	141	irregular	2.37	0.91	1.09
Pearlitol SD 200	193	round	1.50	0.44	0.53
Super-Tab	76	round	1.56	0.55	0.77
Anh. Theophylline	84	irregular	1.42	0.44	0.75
Theophylline $\cdot \text{H}_2\text{O}$	—	needle shaped	1.44	0.35	0.54

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Principal Contributor: Sanchez-Quintanilla

Review on results from shear testers (II)

•Lindberg et al, Drug Development and Industrial Pharmacy, Vol. 30, No. 7, pp. 785–791, 2004

Materials used:

Batches containing approximately 15% of active pharmaceutical ingredient (API), fillers, binders, and lubricant (2%)

The author does not give details about the components

Apparatus: Jenike shear cell and uniaxial compression tester.

Typical stresses: 3.8 KPa for Jenike tester and 80 KPa for the uniaxial compression tester

Conclusions:

✓ The results of the uniaxial tester are useful for measuring tableting characteristics

✗ The results of the Jenike tester do not give much information about tableting characteristics

✓ Rank order correlation for both testers similar than the obtained using the FT4 Powder Rheometer, the Aeroflow and the Houssner



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Review on results from Shear Testers (III)

•Gabaude et al. Journal of Materials Science **36** (2001) 1763 – 1773

Apparatus: Johansson indizicer and uniaxial press Lloyd LR30K

Materials used:

Three direct compression excipients: Avicel PH 102, Starch 1500, and Pharmatose DCL 21

Three drug substances: SRX1, SRX2 CP (CP: Coarse Particles) and SRX2 FP (FP: Fine Particles), SRX1 and SRX2 being two different polymorphs of the same drug substance.

Measured magnitudes: Packing coefficient, flow function

Conclusions:

✓ Using the flow function the apparatus discriminates between materials with poor flow properties

✗ On the contrary, using the flow functionthe apparatus does not discriminate between materials with free flowing properties

✗ The packing coefficient combines several material properties, such as particle shape, particle size distribution, interaction between particles, electrostaticity, as well as flow properties to which it can be linked.



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Principal Contributor: Sanchez-Quintanilla

Review on results from Shear Testers (IV)

- Thalberg et al. Powder Technology 146 (2004) 206–213

Apparatus: custom made uniaxial tester

Materials used:

Ordered mixtures of a carrier (Pharmatose 325M, $d_p \approx 50 \mu\text{m}$), intermediated sized particles (Pharmatose 450M, $d_p \approx 20 \mu\text{m}$) and micronized lactose ($d_p \approx 2 \mu\text{m}$)

Composition of ordered mixtures

Denomination	Pharmatose 325M, % w/w	Pharmatose 450M, % w/w	Micronized lactose, % w/w
[100/0/0] (Pure carrier)	100	0	0
[95/5/0]	95	5	0
[95/2.5/2.5]	95	2.5	2.5
[90/7.5/2.5]	90	7.5	2.5
[95/0/5]	95	0	5
[90/5/5]	90	5	5
[90/2.5/7.5]	90	2.5	7.5
[85/7.5/7.5]	85	7.5	7.5
[80/10/10]	80	10	10

Measured magnitudes: yield strength

Conclusions:

✗ The tester cannot discriminate between ordered mixtures with good flowability.

✓ On the other hand, the tester seems suitable for assessment of more cohesive powders.



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Principal Contributor: Sanchez-Quintanilla

Correlation Between Flowability Indices

- Lindberg *et al.* (2004) found that rank order correlation was similar between the Hausner ratio, avalanching, powder rheometry, uniaxial compression, and Jenike shear cell
- Thalberg *et al.* (2004) found that there was a linear correlation between the Hausner ratio and angle of repose (for materials that weren't too cohesive); uniaxial compression only suited for more cohesive materials



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Principal Contributor: Wassgren

Homework

1. Which flow regime(s) is(are) addressed by each of the following testers: AOR, Housner ratio, Avalanche tester, Freeman tester
2. Housner ratio (HR) may be defined in terms of either bulk density or solid fraction. What are these two equations? Can you show if they are the same or not? What are the limiting values of the HR?
3. Reading assignment: Thalberg et al., *Powder Technology* 146 (2004) 206–213; provide a concise summary of the lessons learned and conclusions made.



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Factors Affecting Flowability

- Particle size
 - affects ratio of inter-particle attractive and inertial forces
- Particle size distribution
 - more uniform size distribution \Rightarrow lower bulk density \Rightarrow easier to shear
- Particle shape
 - mechanical interlocking of particles
 - more spherical \Rightarrow better flow
- Particle surface roughness
 - affects the significance of attractive forces
 - smooth surfaces \Rightarrow surfaces can get closer \Rightarrow stronger van der Waals forces



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rough surfaces \Rightarrow greater surface energy \Rightarrow stronger electrostatic forces

Principal Contributor: Wassgren

Factors Affecting Flowability

- Interstitial air
 - larger permeability \Rightarrow air can easily infiltrate gaps when bulk expands under shear
 - fluidized powder easily flows
- Moisture
 - low moisture \Rightarrow increases electrostatic forces
 - high moisture \Rightarrow increases capillary forces

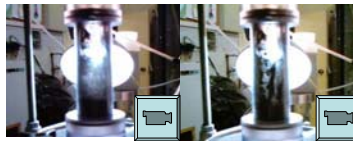
The Sevilla Powder Tester (SPT)

• Any reliable device to measure powder properties should be able to **erase powder memory**. Otherwise results are history-dependent

• This can be achieved by driving the powder into a highly expanded **fluidization state**

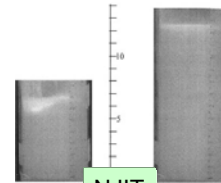
Preconditioning (fluidization) of fine cohesive powders

- Fine cohesive powders are difficult to fluidize (**Geldart C**)
- Classical **Geldart C** classification does not refer to an intrinsic property of the powder
- Pre-conditioning process to achieve uniform fluidization **are available or have to be developed**. They need to be applied before any measurement
- Examples: Coupling gas flow with: **vibration, magnetic assistance, acoustic excitation and/or centrifugal force** (Pfeffer, Nam, Dave, Liu, Quevedo, Yu, Zhao:US2006086834 patent)



•For xerographic toners, coated cornstarch, silica nano-particles...**strong shaking** is enough.

•Note that **shallow beds** are employed



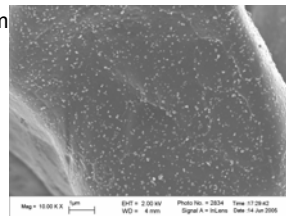
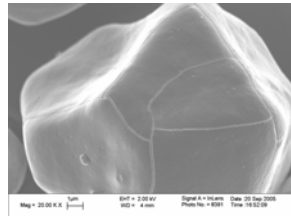
For micron-sized particles memory is erased when inter-particle contacts are broken to the level of individual particles

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Fluidization of Dry-Coated Fine Powders

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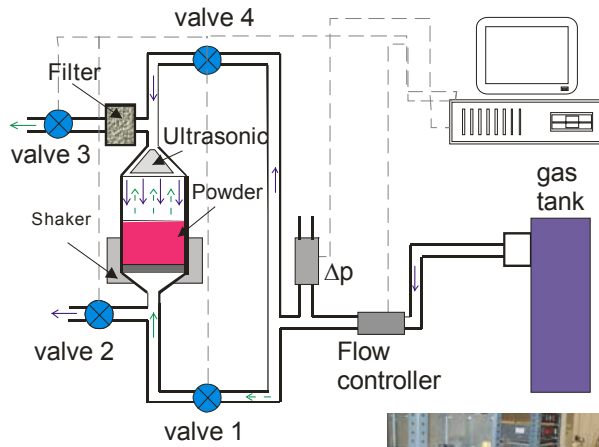
Raw Cornstarch
Size: 15 micron

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0.1% R972 Coated Cornstarch
Size: 15 micron

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The Sevilla Powder Tester (SPT)



Developed from the work of A.T. Perez, A. Ramos, JM Valverde, **MAS Quintanilla**, A Castellanos (U. Seville), MA Morgan, F Genovesse, PK Watson (Xerox Co.).



Gas velocity:

$$v_g$$

Gas Pressure

Drop:

(load or unload)

$$\sigma_c = W \pm \Delta p$$

$$\sigma_t$$

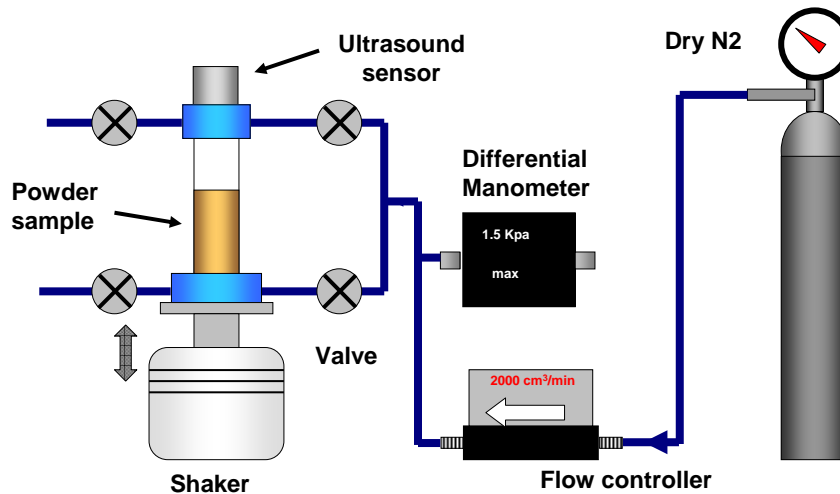
Ultrasonic:

Bed height H ,
packing fraction
 χ , settling velocity
 v_s

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Principal Contributor: Valverde

Powder tester set-up



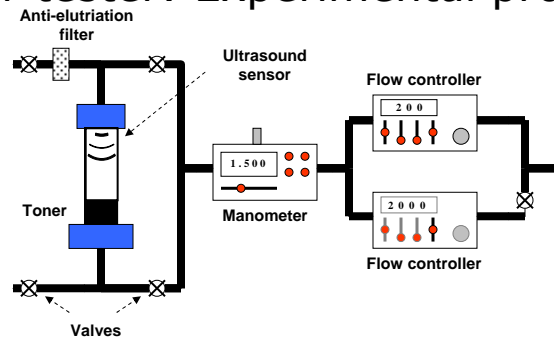
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Powder tester: Experimental procedure



- 1 Material is fluidized to be initialized
- 2 The gas flow is reduced and the powder collapses
- 3 The powder is pressed or decompressed by the gas
- 4 The ultrasonic sensor measures the porosity
- 5 The gas flow breaks the powder bed to measure its tensile stress



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Powder Tester: Measured variables

- **Consolidation stress.** Calculated from the mass m , the filter area A and the pressure drop Δp as:

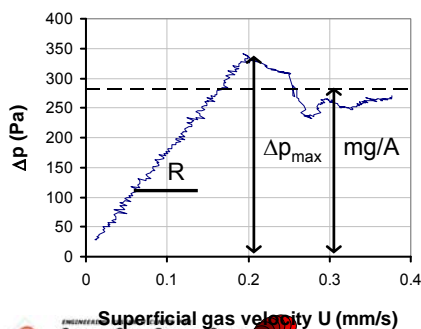
$$\sigma_c = \frac{mg}{A} \pm \Delta p$$

The sign of Δp depends on whether the gas flow consolidates the powder or holds part of its weight

- **Solid fraction.** Calculated from the mass m , the particle density ρ , the filter area A and the height H measured by the ultrasound sensor as:

Pressure drop during the rupture process

$$\phi = \frac{m}{\rho HA}$$



- **Tensile strength.** The difference between the maximum value of the pressure drop and the weight per unit area during the rupture process

$$\sigma_c = \Delta p_{\max} - \frac{mg}{A}$$



- **Powder resistance to the gas flow.** The slope of the linear relationship between pressure drop Δp and superficial gas velocity U

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Principal Contributor: Valverde



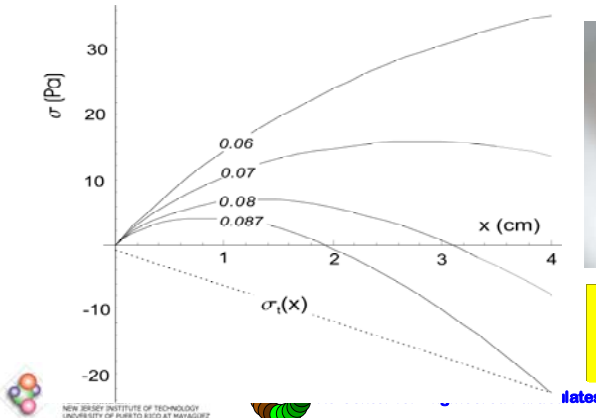
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Breaking takes place at the bottom

$$\left. \begin{aligned} \frac{d\sigma}{dx} &= \rho_p \phi g - \frac{dp}{dx}, \\ \frac{dp}{dx} &= \frac{E\mu}{d_p^2} \frac{\phi^2}{(1-\phi)^3} v_g, \end{aligned} \right\} \begin{aligned} \sigma(x) &= \rho_p \phi_0 g x \left(1 - \frac{10\phi_0}{(1-\phi_0)^3} \frac{v_g}{v_p} \right) \quad (E=180) \\ \sigma(x) &= \int_0^x \rho_p \phi(\xi) g \left(1 - \frac{10\phi(\xi)}{[1-\phi(\xi)]^3} \frac{v_g}{v_p} \right) d\xi \quad v_p = \frac{1}{18} \frac{\rho_p g d_p^2}{\mu} \end{aligned}$$

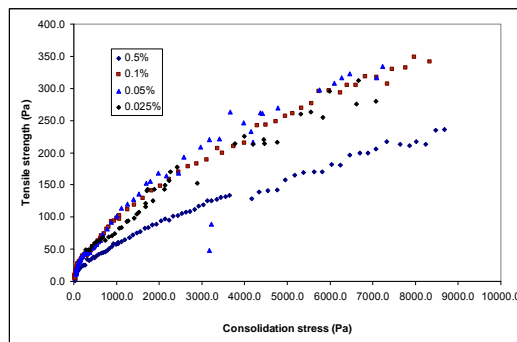


We systematically observe a layer of powder that remains adhered to the gas distributor

Principal Contributor: Valverde

The tensile stress from SPT

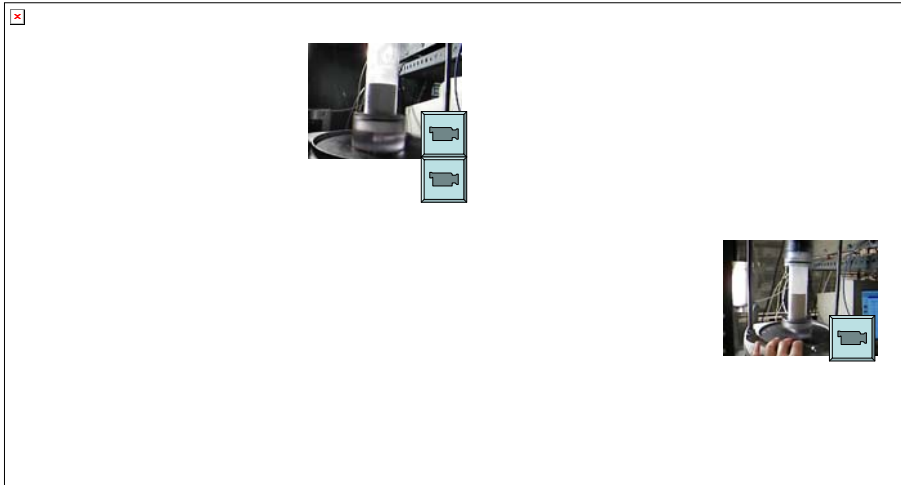
The cohesivity of a powder is characterised by the plot of its tensile strength σ_t as a function of the consolidation stress σ_c



Additionally, the tester provides the solid fraction of the powder versus the consolidation stress in uniaxial compression

The smaller the tensile strength for a given consolidation, the better the powder will flow in the plastic regime

Over-weight consolidation and under-weight consolidation by gas flow



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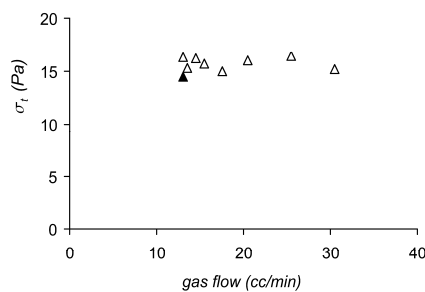


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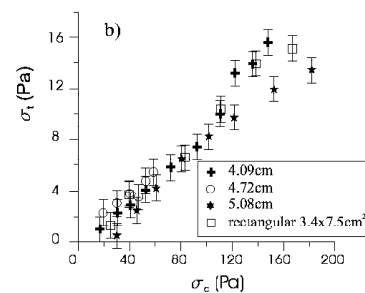
Principal Contributor: Valverde

Checks



Tensile yield stress measured by increasing quasi-statically the gas flow (solid triangle) and by imposing instantaneous values (void triangles) of the gas flow represented in the horizontal axis.

Increase of 1 cc/min in 3 sec is good enough for quasi-static condition



Tensile yield stress versus consolidation stress. Data obtained for toner with 0.4%wt of additive obtained with different bed diameters (4.09 cm, 4.72 cm, 5.08 cm, 8.0 cm) and with a rectangular bed.

Wall effects are negligible if bed height is kept low

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Principal Contributor: Valverde

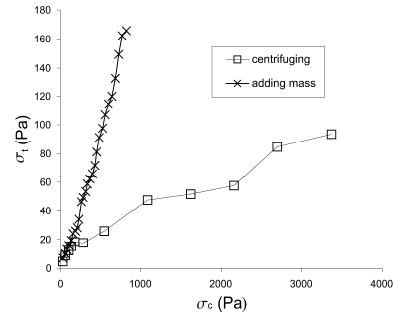
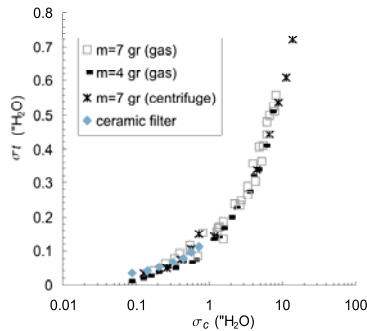


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Checks



Tensile yield stress as a function of the consolidation stress for an experimental xerographic toner with 0.2%wt concentration by weight of additive. The data from different tests using consolidation by gas (two tests with samples of different masses) and centrifuging in a bed with a metallic gas distributor are plotted jointly with data obtained in a bed with a ceramic gas distributor where consolidation was increased by adding new mass to the sample.

Using different techniques of consolidation. One of them consists of adding mass to the sample contained in a cylindrical bed (diameter $D=5.08$ cm). The other way of consolidation consists of centrifuging the bed. With the technique of adding mass, wall effects are not negligible for around 200 Pa, corresponding to bed heights typically larger than the bed diameter.

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The Center for Integrated Fabrication

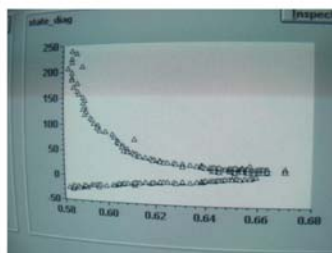
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Minimum accuracy is needed



Gas velocity accuracy 0.01mm/s
Gas pressure drop accuracy 2Pa

Bed height accuracy 0.1mm



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Essential requirements:

- Pre-conditioning procedure (assisted fluidization) to **erase powder memory**
- **Shallow beds** to avoid wall effects
- **Accuracy** of the measuring devices



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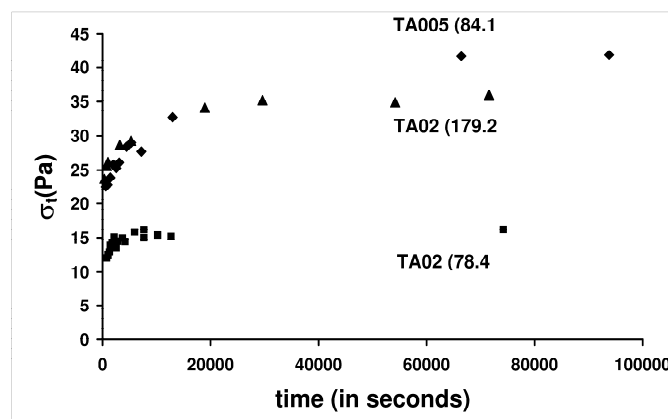


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Viscosity (consolidation time) effects on interparticle contacts



Tensile stress as a function of the **time during which the powder is consolidated**. Examples are shown for TA toner particles with different surface Aerosil coverage and subjected to different load forces (indicated in parenthesis)



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Effect of surface additive

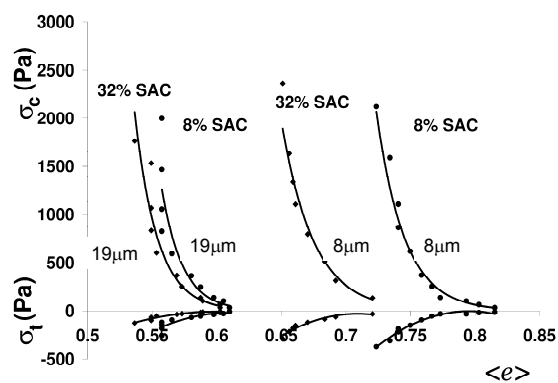


Powders that flow well pack well

101

Principal Contributor: Valverde

Effect of surface additive and particle size



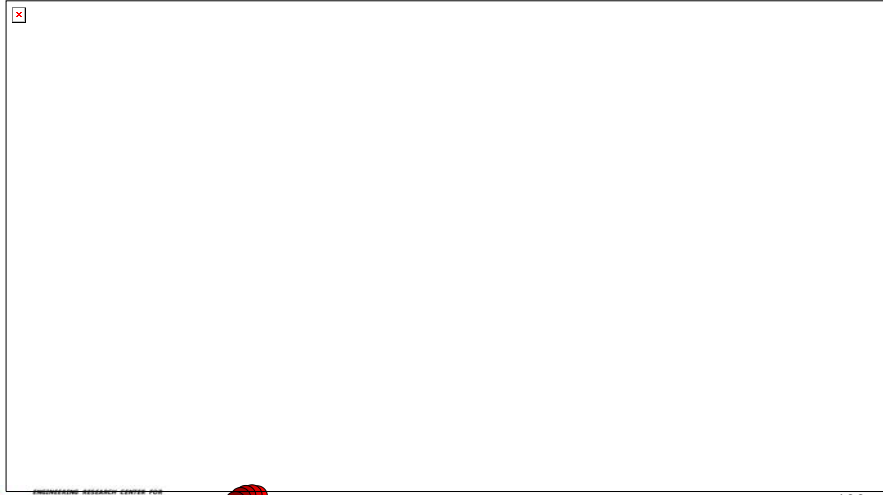
In the old days, the xerographic industry did not have powder flow related problems...



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Effect of gel content in host particle



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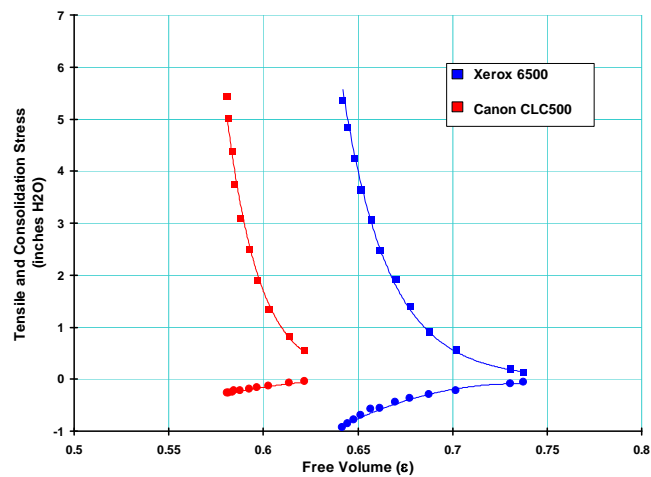


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Sevilla Powder Tester measurements of Xerox 6500 and Canon CLC500 Toners



Example of use in industry. Powder flowability may be inferred from the state diagram.



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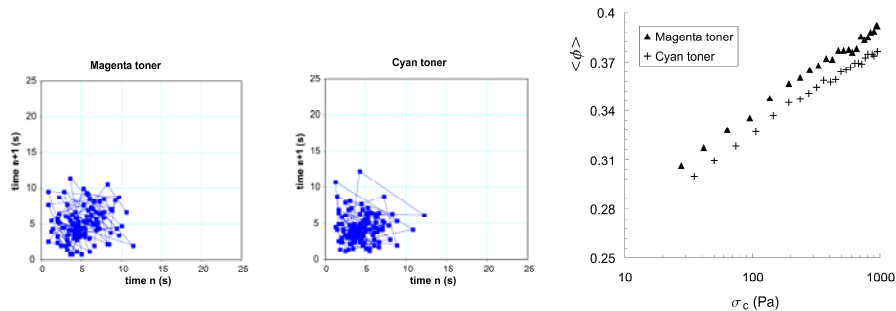


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Principal Contributor: Valverde

SPT versus Aeroflowmeter

Two samples of different color (magenta and cyan) of the commercial xerographic toner Canon CLC700.



The points of time T_n at which an avalanche occurs are represented against the points of time T_{n+1} of the next avalanche

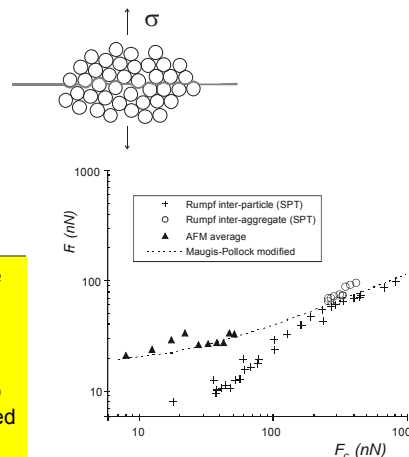
Average particle volume fraction as a function of the consolidation stress

The SPT as a research tool on contact forces

$$\sigma \approx \frac{\xi F}{\pi d_p^2} \phi \quad (\text{Rumpf eq.})$$

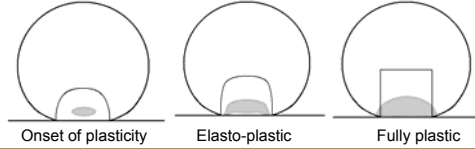
$$\xi \approx \frac{\pi}{2} (1 - \phi)^{-\frac{3}{2}} \quad (\text{coordination no.})$$

- First derived by Rumpf for a monodisperse packing of hard spheres, wherein the distribution of stresses is isotropic and homogeneous
- The estimated forces are well correlated to forces between individual particles measured by AFM, although for low consolidation stress particle agglomeration must be considered



Contact forces directly measured with the AFM, predicted by the modified Maugis-Pollock equation ($w = 0.07 \text{ J/m}^2$, $\nu = 1/3$, $E = 6 \text{ GPa}$, and $H = 0.3 \text{ GPa}$), estimated by means of the Rumpf equation and forces estimated by means of the modified Rumpf equation. The powder tested has 10% of surface additive coverage and $12.7 \mu\text{m}$ particle size.

Theory on contact forces



Taking into account the Hertz solution for elastic solids the critical load for the initiation of plastic yield within the bulk is $P_Y \sim \pi^2 d_a^2 Y^3 (6E)^2$ where d_a is the asperity size, E is the Young modulus and Y is the yield strength.

Attractive forces contribute to an effective load P_0 on the contact and, in the absence of external loading, may originate by their own plastic deformation if $P_0 > P_Y$.

For zero external load the equivalent Hertzian load due to attractive forces is $P_0 \sim \pi w d_a$ (Derjaguin et al. 1975; Johnson et al. 1971), where w is the adhesion energy.

Thus we estimate that attractive forces would induce a plastic deformation ($P_0 > P_Y$) if $d_a < \sim 6 w E / (\pi^2 Y^3)$.

For polymer-polymer contacts (typically $w \sim 0.07 \text{ J/m}^2$, $E \sim 6 \text{ GPa}$, $Y \sim 0.1 \text{ GPa}$) we get $d_a < \sim 1 \mu\text{m}$ for the critical asperity size below which plastic deformation is present. This value is clearly larger than our typical asperity size. Thus the elastic limit is well exceeded due to the presence of attractive forces alone.

In the regime where the whole area of contact deforms plastically (fully plastic regime), the modified Maugis-Pollock equation gives an estimate of the interparticle adhesion force F_t as a function of the interparticle load force F_c :

$$F_t = \frac{3\sqrt{\pi}}{4} \frac{wE}{(1-\nu^2)H^{3/2}} \sqrt{F_c + \frac{\pi}{2} w d_a}$$

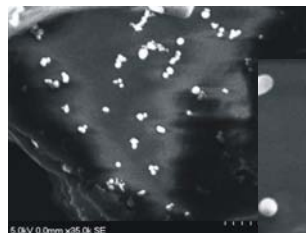
H is the contact hardness

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Effect of surface additives, size hardness, etc on interparticle forces can be estimated



100% SAC of 8nm silica nanoparticles on host 7 microns toner particle



20% SAC of 40nm silica nanoparticles on host 7 microns toner particle

Hitachi S-5200 ultra-high resolution SEM (CITIUS, University of Seville)



- The size of silica nanoparticle agglomerates determines asperity size
- The %SAC controls contact hardness



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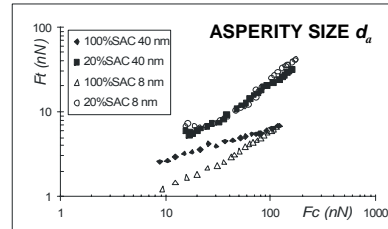
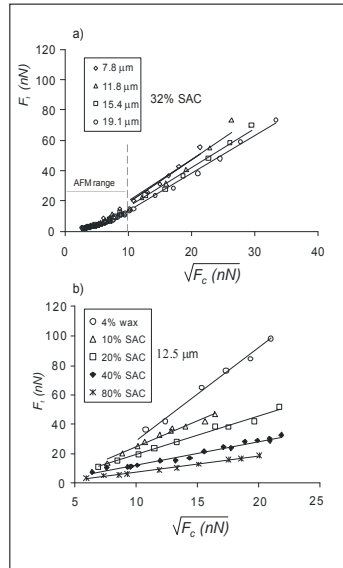
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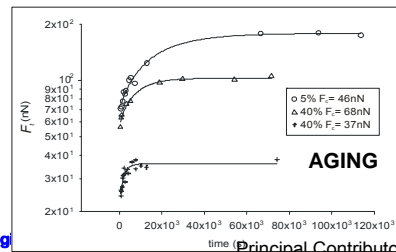
Estimated Interparticle Forces

$$\sigma \approx \frac{\xi F}{\pi d_p^2} \phi \quad (\text{Rumpf eq.})$$

$$\xi \approx \frac{\pi}{2} (1 - \phi)^{\frac{3}{2}} \quad (\text{coordination no.})$$



$$F_t \propto \frac{w E}{H^{3/2}} \sqrt{F_c + \pi w d_a} \quad (\text{plastic contact})$$



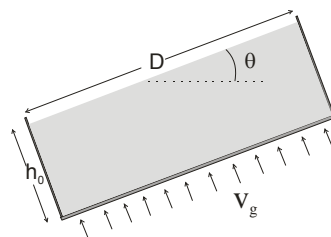
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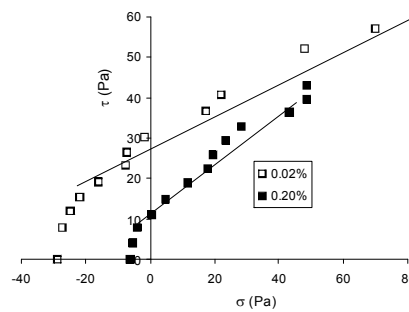
The SPT to measure the yield locus



$$\tau \approx \rho g h_0 \sin(\theta)$$

$$\sigma \approx \rho g h_0 \cos(\theta) - \Delta P(v_g)$$

Coulomb yield locus
at low consolidations



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Summary- SPT

- Advantages of the Sevilla Powder Tester are i) results are **operator-insensitive** since measurements are automatically taken, and ii) fluidization provides a convenient method to have the sample in a **reproducible initial state that lacks memory of previous processes**.
- Every step in the process is determined by gas flow, and by means of a set of valves and flow controllers, the entire process runs by a computer. The measurements involving gas flow velocity, pressure difference, and bed height are accessed by the same computer, and from these sets of measurements, the values of **consolidation stress, average particle volume fraction, and uniaxial tensile yield stress** are automatically calculated.
- An **upward/downward-directed gas flow** is used for **deconsolidating/consolidating** the powder, and an **ultrasonic device** measures the bed height giving an average value of the particle volume fraction.
- The usefulness of the SPT is not just restricted to flowability diagnosis. Our experimental work shows that the tester is a **powerful instrument of research in powder technology**.



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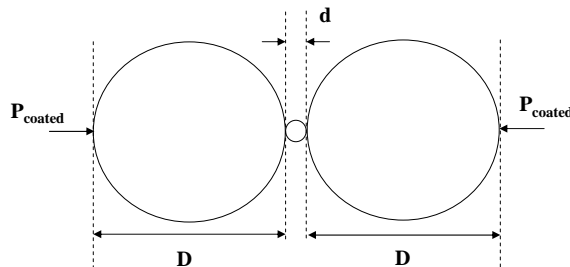


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Cohesion Reduction by Nano Coating: Theoretical Considerations



$$P_{uncoated} \cong \frac{A}{12} \frac{D}{2} \frac{1}{h_o^2}$$

If two particles are not coated, then van der Waal's attraction force between them is given by the above equation

$$P_{coated} \cong \frac{A}{12} \frac{dD}{d+D} \frac{1}{h_o^2}$$

The equation above gives the contact force between two coated particles having one guest particle between the two.

Here A is the Hamaker constant for these materials, and h_o is the atomic scale separation between the two contacting surfaces

Hence the ratio between the two cohesive forces (coated and uncoated particles) is as below, assuming d is much smaller than D :

$$\frac{P_{coated}}{P_{uncoated}} = 2 \frac{d}{D} \cong \frac{d}{D}$$



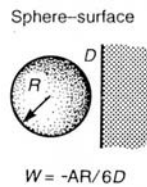
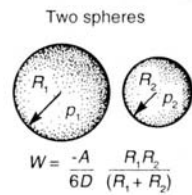
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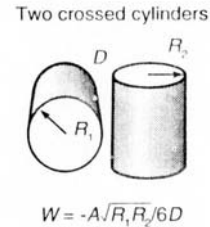
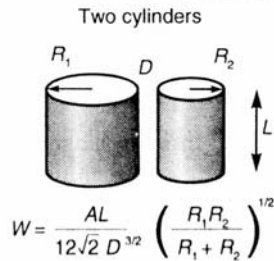
Reminder: Van der Waal's Interactions between Various Geometries



Interaction energies are given for various geometries.

A = Hamaker constant

D = Separation between the bodies, $1.65 - 4 \text{ \AA}$



Force between two spheres can be given by:

$$F(D) = -\frac{A}{6D^2} \frac{R_1 R_2}{(R_1 + R_2)} \quad F(D) = -\frac{AR}{12D^2}$$

For two equal spheres of radius, R

From: Israelachvili, "Intermolecular and Surface Forces", 2nd edition, 1992, p. 113

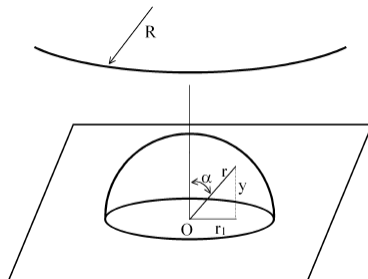
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Modeling of Adhesion Force of Group C Powders

• Rumpf Model

$$F_{ad} = \frac{A}{12z_0^2} \left[\frac{dD}{d+D} + \frac{D}{(1+d/2z_0)^2} \right]$$

A Hamaker constant
 D Particle diameter
 d Diameter of Asperity
 z_0 Distance between two contact particles



- Asperity is considered as a hemisphere centered on the contact surface
- Adhesion force comprises two parts- "contact" and "noncontact" force
- Does not take into account the effect of deformation and surface asperity distribution

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Rumpf, H. "Particle Technology", Chapman & Hall, London/New York, 1990

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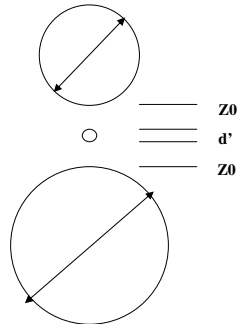
Principal Contributor: Chen

Modeling of Adhesion Force of Cohesive Powders

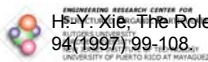
• Sandwich Model

$$F^{0+} = F^{0'} + F^{0''} = \frac{AD}{12(z_0 + d')^2} + \frac{A(D_1' + D_2')}{12z_0^2}$$

A Hamaker constant
 d' Diameter of small particle
 z_0 Distance between two contact particles
 D, D_1', D_2' The mean diameter of contact particles



- Adhesion force consists of two terms
- If the asperity is less than 10nm, the surface can be treated as smooth
- If the asperity is more than 0.1μm the asperity-particle attraction is dominant
- Does not consider the deformation and surface asperity distribution



H.-Y. Xie, The Role of Interparticle Forces in the Fluidization of Fine Particles, Powder Technology 115 94(1997) 99-108.
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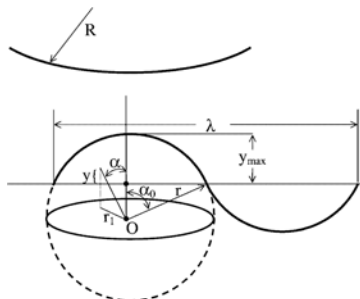
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Modeling of Adhesion Force of Group C Powders

• Rabinovich Model

$$F_{ad} = \frac{AD}{12z_0^2} \left[\frac{1}{1 + \frac{16Dk_1rms}{\lambda^2}} + \frac{1}{\left(1 + \frac{k_1rms}{z_0}\right)^2} \right]$$

$k_1 = 1.817$
 A Hamaker constant
 λ Peak-to-peak Distance
 D The diameter of contact particles
 rms Root-Mean-Square Roughness
 z_0 Distance between two contact surface



- Adhesion force has two components
- Asperity is treated as a hemisphere centered below the contact surface
- The rms roughness is applied instead of the radius of asperity
- The model is valid for small asperity and large peak-peak distance ($\lambda / rms \geq 14.5$)



Y. Rabinovich, et al.: Adhesion Between Nanoscale Rough Surface I. Role of Asperity Geometry, Journal of Colloid and Interface Science 222:10-16(2000).



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Modeling of Adhesion Force of Group C Powders

• Mei's Model

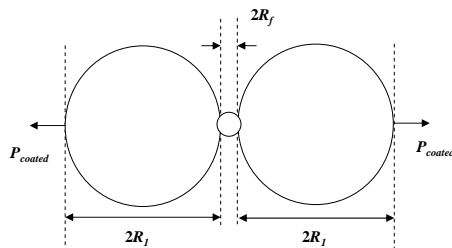
$$\frac{P_{coated}}{P_{uncoated}} = \frac{2R_f}{R_1}$$

R_f Radius of guest particle

R_1 Radius of host particle

P_{coated} Adhesion force between coated particles

$P_{uncoated}$ Adhesion force between uncoated particles



- Adhesion force model is based on surface energy and has only one part
- The adhesion force is reduced dramatically by dry particle coating
- Does not consider the guest particle distribution



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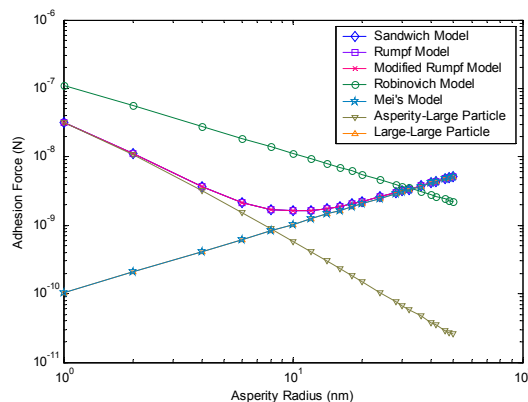


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Modeling of Adhesion Force of Group C Powders

• Comparison of the Current Models



Rabinovich model provides the largest adhesion force, Mei's model provides the smallest

Rumpf, sandwich and modified rumpf model have the same results

These plots indicate that there exists a certain asperity size to where the adhesion force takes a minimum value (this will give us the direction on choosing guest particles when we use dry coating to reduce adhesion force)

Comparison of Current Adhesion Force Models of Rough Particles



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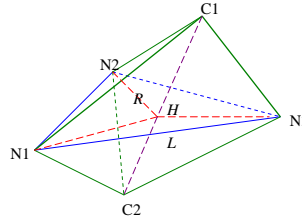
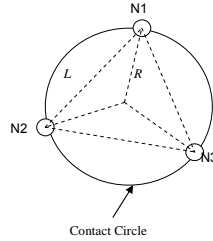
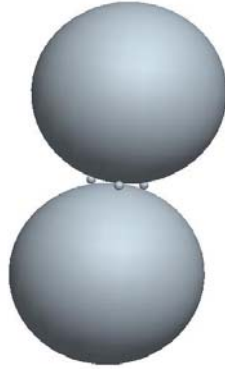


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Modeling of Adhesion Force of Group C Powders



N1, N2, N3 are the centers of guest particles respectively

C1 and C2 are the centers of two host particles

R is the radius of contact circle formed by three guest particles

L is the lateral length of equilateral triangle with three guest particles on vertexes

H is the distance between centers of host particles

$$F_{ad} = \frac{A}{12z_0^2} \frac{3dD}{d+D} + \frac{A}{12H_0^2} \frac{DD}{D+D} \Rightarrow F_{ad} = \frac{Ad}{4z_0^2} + \frac{A}{24 \left(\sqrt{\left(1 + \frac{d}{D}\right)^2 - \frac{1.21}{SAC} \left(\frac{d}{D}\right)^2} - 1 \right)^2} D$$



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Modeling of Adhesion Force of Group C Powders

The distance between two host particles H_0 is calculated by

$$H_0 = H - (2z_0 + D) \approx H - D$$

The distance between center of two host particles H is calculated by

$$R^2 = \left(\frac{D+d}{2}\right)^2 - \left(\frac{H}{2}\right)^2 \Rightarrow R^2 \approx \left(\frac{D+d}{2}\right)^2 - \left(\frac{D+H_0}{2}\right)^2$$

Radius of contact circle R is derived from the area of contact triangle S

$$S = \frac{3\sqrt{3}}{4} R^2$$

Assuming that guest particles are evenly and individually distributed on host particle surface, S could be deduced from the number of guest particles by

$$N = \frac{\pi D^2}{2S} = \frac{2\sqrt{3}\pi D^2}{9R^2}$$

The number of guest particles N can be derived from the Surface Area Coverage (SAC) or weight ratio of guest particles respectively by

$$SAC = \frac{N \times \pi d^2}{4\pi \left(\frac{d+D}{2}\right)^2} \times 100\% = \frac{N \times d^2}{4(d+D)^2} \times 100\% \approx \frac{N \times d^2}{4D^2} \times 100\% \quad \text{or} \quad Wt\% = \frac{(Nd^3\rho_d)}{(D^3\rho_D) + (Nd^3\rho_d)} \times 100\%$$

Finally, the H_0 is

$$H_0 = \sqrt{\left(\frac{D+d}{2}\right)^2 - \frac{1.21d^2}{SAC}} - D$$



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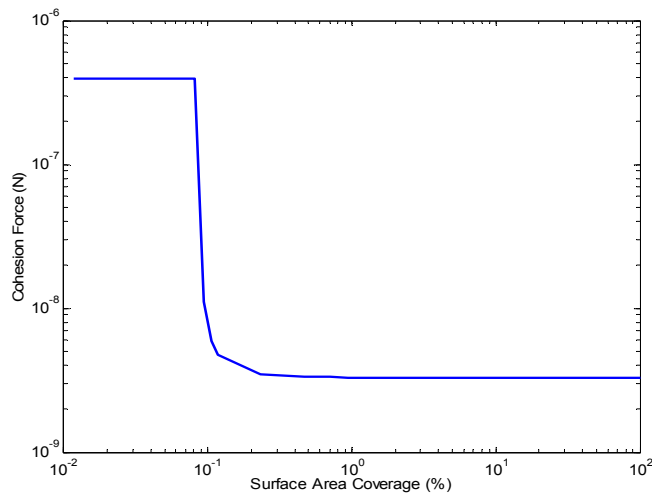


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Principal Contributor: Chen

Cohesive Force for the Coated Particles as a Function of SAC



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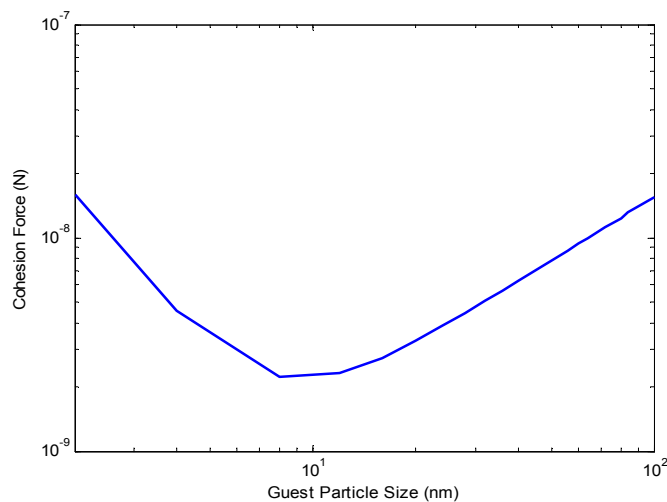


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Cohesive Force for the Coated Particles as a Function of Guest Particle Size



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Powder Testing of Functionalized Powders

- **Functionalization**

- Modifying properties of powder

- Dry coating

- MAIC (Magnetically Assisted Impaction Coating)

- Hybridizer

- **Characterize powders both before and after modification**

- Sevilla Powder Tester

- Hosokawa Powder Tester

- Carr Indices

- Vibrated Packing density



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The following materials were used:

- Host particles:

Cornstarch, diameter $D \sim 15 \mu\text{m}$, density

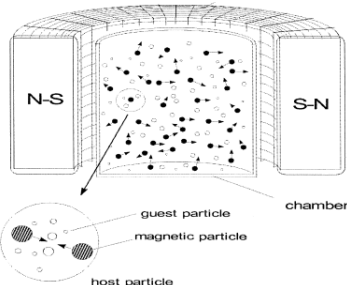
$\rho_h = 1.55 \text{ g/cm}^3$

Acetaminophen (APAP), diameter $D \sim 50 \mu\text{m}$, $\rho_h = 1.29 \text{ g/cm}^3$

Tolmetin Sodium dihydrate, diameter $D \sim 120 \mu\text{m}$

- Guest particles:

Silica R972, diameter $d = 16 \text{ nm}$, density $\rho_g = 2.55 \text{ g/cm}^3$, hydrophobic



Powders used in the experiment are processed in Magnetically Assisted Impact Coater (MAIC)

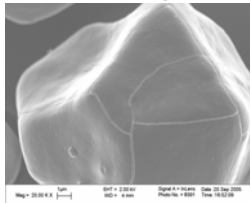


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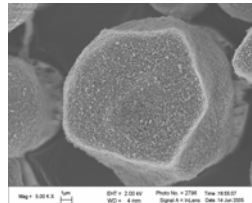


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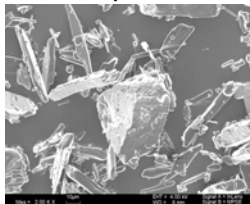
Bare cornstarch particles



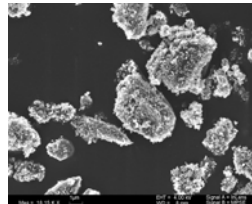
Cornstarch+ silica R972



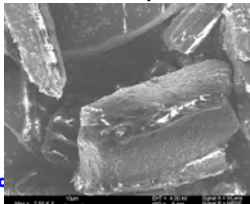
Bare APAP particles



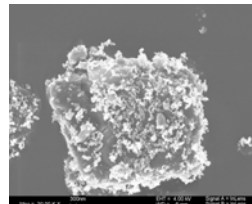
APAP+ silica R972



Bare Tolmetin particles



Tolmetin + silica R972

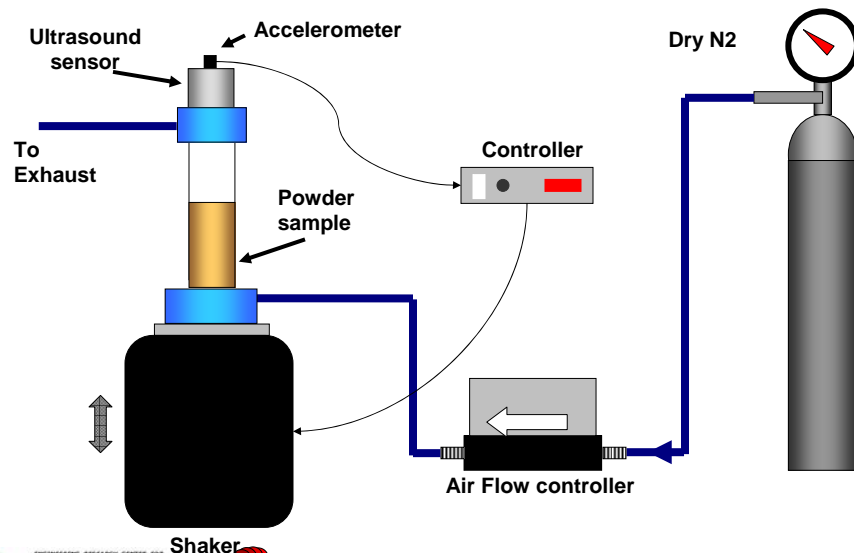


Characterization: Packed Density

Motivation: While SPT provides very useful data on flowability such as the tensile strength and packed density as a function of consolidation stress, it cannot work well with powders that cannot be fluidized well. Hence a new approach is required for measuring packed density.

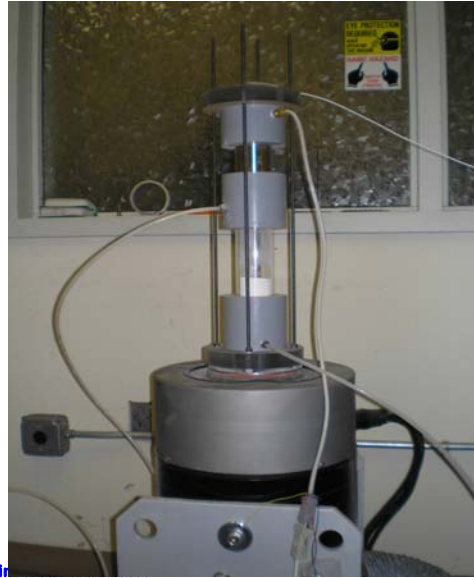
- Useful as a test of flowability improvement
- Under the same consolidation stress, a more flowable powder will have a higher solid fraction upon packing
 - Under the same consolidation stress, a less flowable powder will have a lower solid fraction than another
- **Testing has been done with cornstarch and varying amounts of additive processed in the MAIC under standard conditions**

Packed Density Apparatus



Packed Density Apparatus

- Material fluidized to initialize and clear history of the powder
- Material is given time to randomly settle
- Frequency is set to 60 Hz
- Acceleration velocity ranged from 0.5 to 4g in 0.5g increments
- Time cumulatively increased by a minute for each test as the acceleration velocity is increased
- Ultrasound measures the height of the powder and is translated into solid fraction based on the weight of the powder



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Packed Density Controller Program

Totally automated setup with continuous testing

Labworks
automated
software to
control the
vibration
setup



WinSpan
program
for ultra
sonic
height
sensor



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Packed Density Results

- As additive amount increases, solid fraction increases for each consolidation stress
- With 0.5% R972+ Cornstarch, upon vibration, solid fraction nears that of random close packing of a sphere
 - Coated cornstarch, approximately 16 micron in size begins to behave similarly to a 100 micron round glass sphere.
- However, 2% and 5% R972+ Cornstarch do not follow the trend
 - These percentages are over the percentage additive needed for 100% surface area coverage using the MAIC
 - Excess silica might be interfering with the packing.



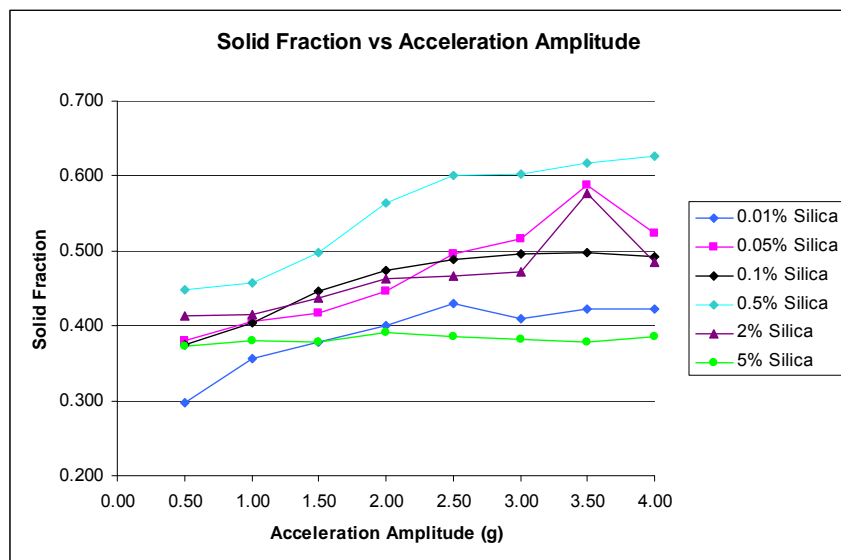
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Packed Density Results (Cornstarch)



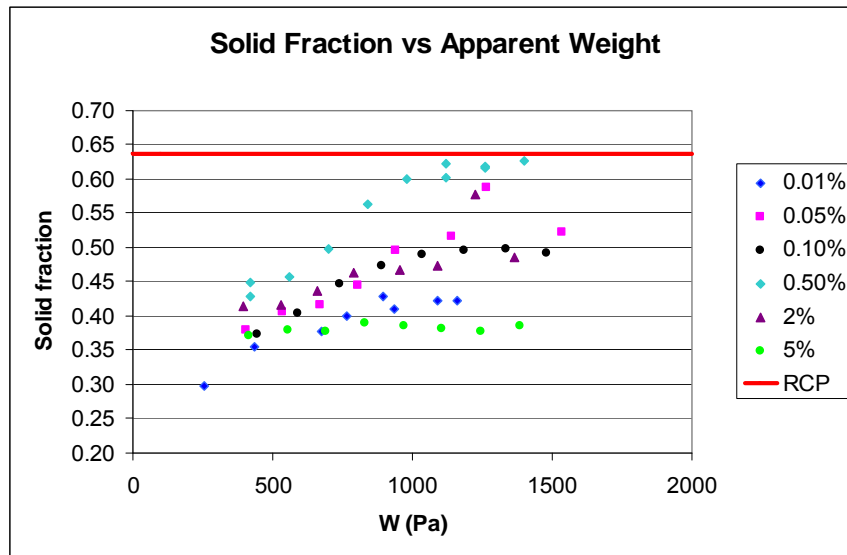
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Packed Density Results (Cornstarch)



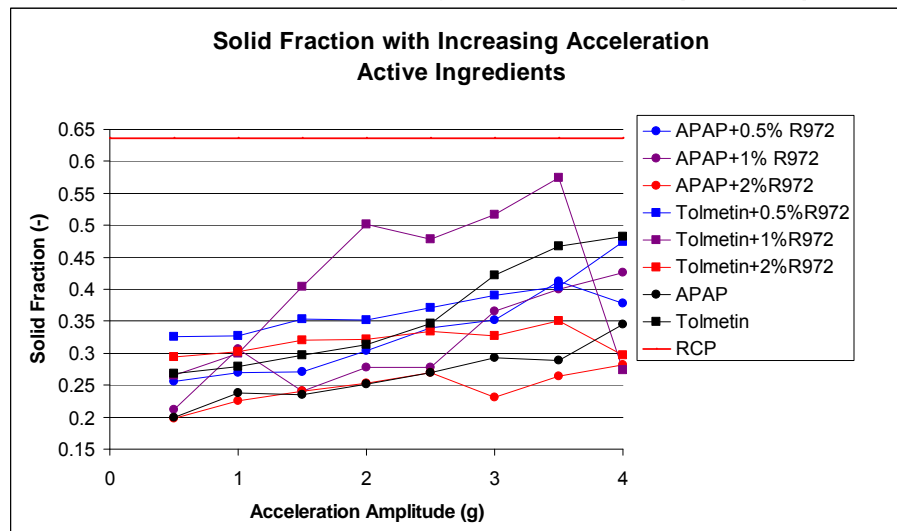
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Packed Density Results (APIs)



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Homework

1. Which flow regime(s) is(are) addressed by the Sevilla Powder Tester? What part of the Yield Surface does the SPT measurements provide?
2. Derive the following equation for estimate of cohesion reduction due to nano-coating.

$$\frac{P_{coated}}{P_{uncoated}} = 2 \frac{d}{D} \cong \frac{d}{D}$$

3. Reading assignment: Valverde JM et al: The tensile strength of cohesive powders and its relationship to consolidation, free volume and cohesivity, *Powder Tech.* 97 (1998) 237-245. (a) Explain Fig 9. (b) What can we learn about the capability of the SPT as a flowability measurement device from the results such as those of Fig 12?

