Particle Dispersion in Liquid Formulation
Royal Society of Chemistry- Formulation Science and Technology Interest Group
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Dispersion of Fine Powders in Liquids

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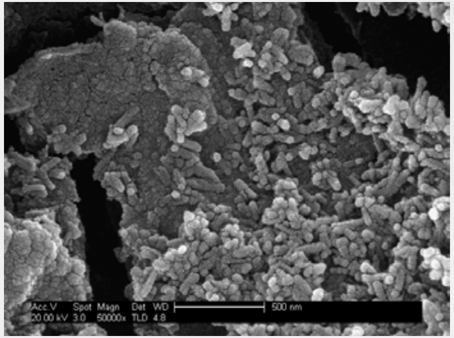
Nanocomposites

Nanoparticles\nanoclays dispersed in a liquid …

- chemically more reactive
- improved product properties (mechanical, thermal, optical)

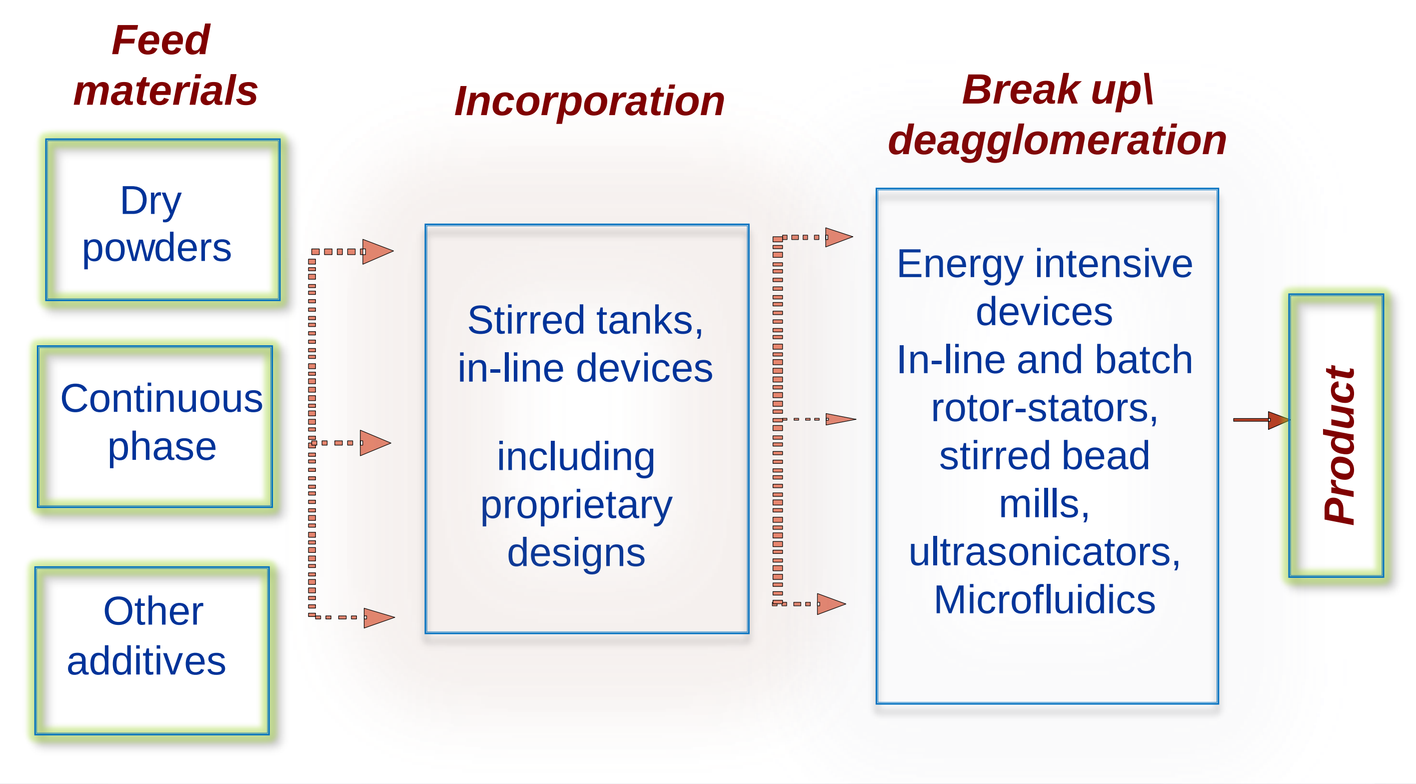
Already used in several products (health and personal care, coating, paints, fine chemicals,…)



… have been entering the market at a fast rate

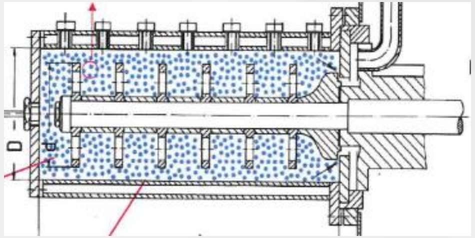
There is a need to manufacture nanoparticles in large quantities and incorporate them in the formulation of final products.

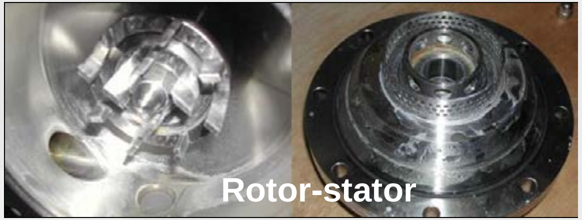
Dispersion of Fine Powders in Liquids



Selection of process devices

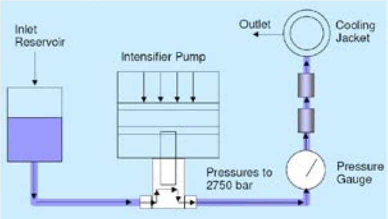
Stirred bead mill

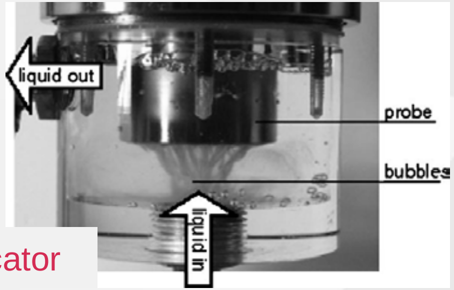




High pressure devices

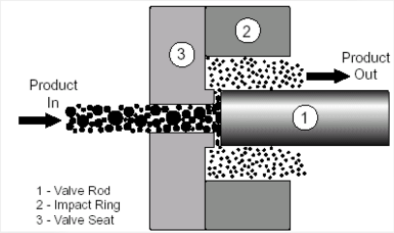
Microfluidics

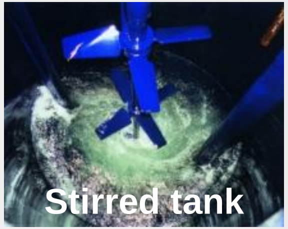




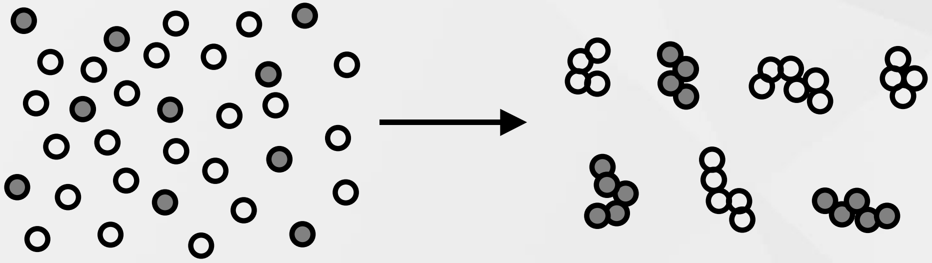
Ultrasonicator

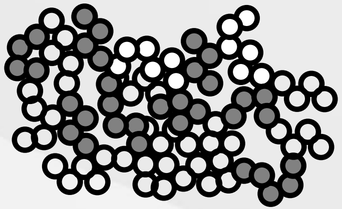
Valve homogeniser





Dispersion of Nanoparticle Clusters in Liquids





Primary Particles | **Aggregates** | **and** | **Agglomerates**

Typically: 5-50 nm | O(100) nm | O(10-100) μm

Nanoparticles → into a liquid

→ stronger clusters that tend to float

Break up occurs …

… through stresses acting on the agglomerates

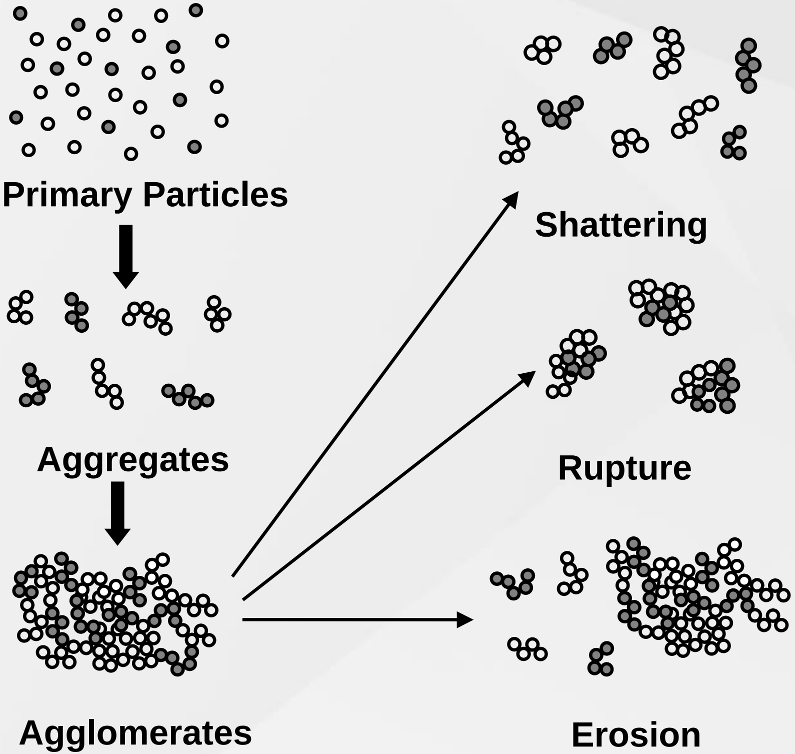
Hydrodynamic stresses:

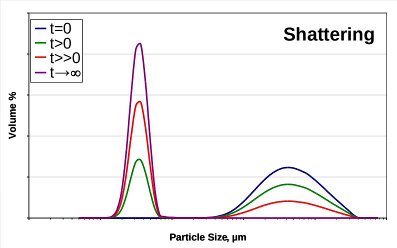
- Laminar flow: $$\tau = \mu\dot{\gamma}$$
- Turbulent flow: l>>L_i>>λ_k $$\tau \propto \rho\varepsilon^{2\backslash 3}L_i^{2\backslash 3}$$

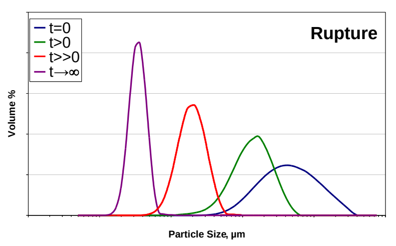
L_i<λ_k $$\tau \propto \mu \left(\frac{\varepsilon}{\nu}\right)^{1/2} = \rho\nu^{1/2}\varepsilon^{1/2}$$

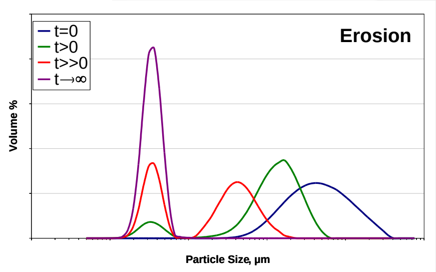
which are sufficiently high to overcome the tensile strength: $$\sigma_T = 1.1\frac{(1-\varepsilon_a)}{\varepsilon_a}\frac{F_{TOT}}{L_a^2}$$

Breakup of nanoparticle clusters



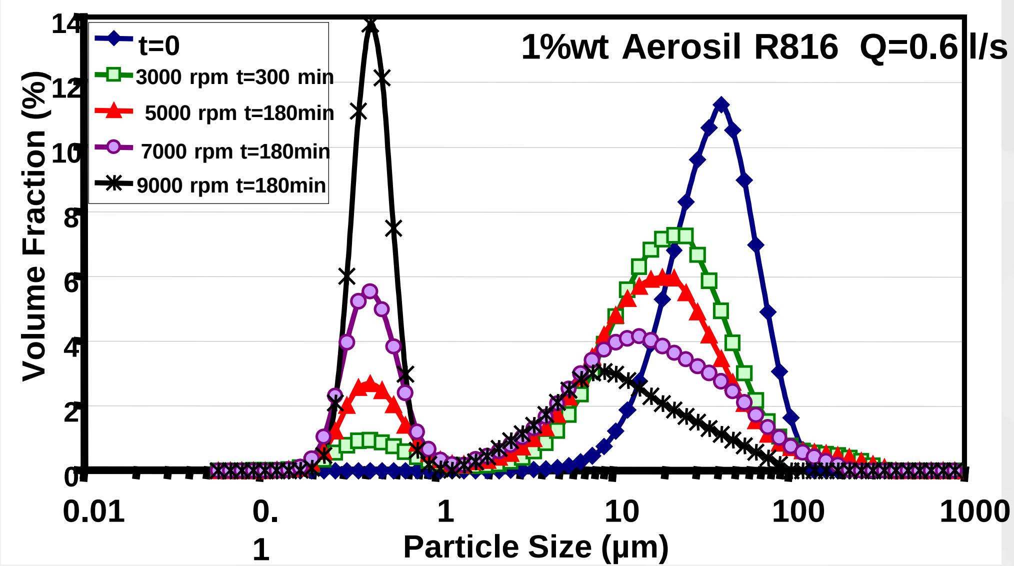


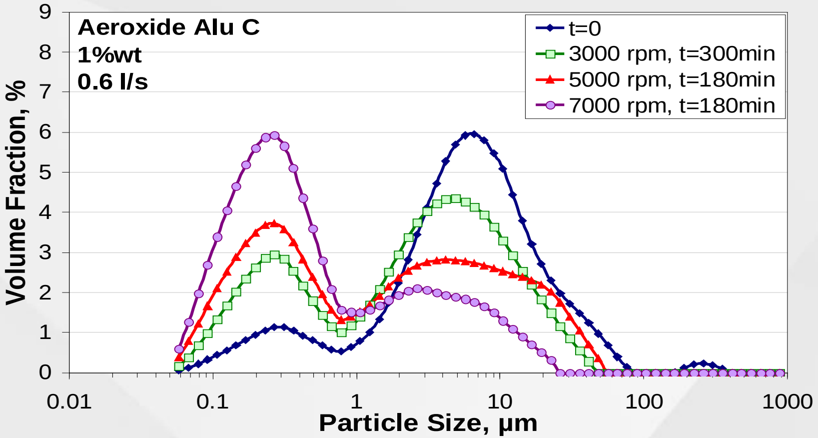




Özcan-Taşkın et al, 2009

Mechanism of breakup





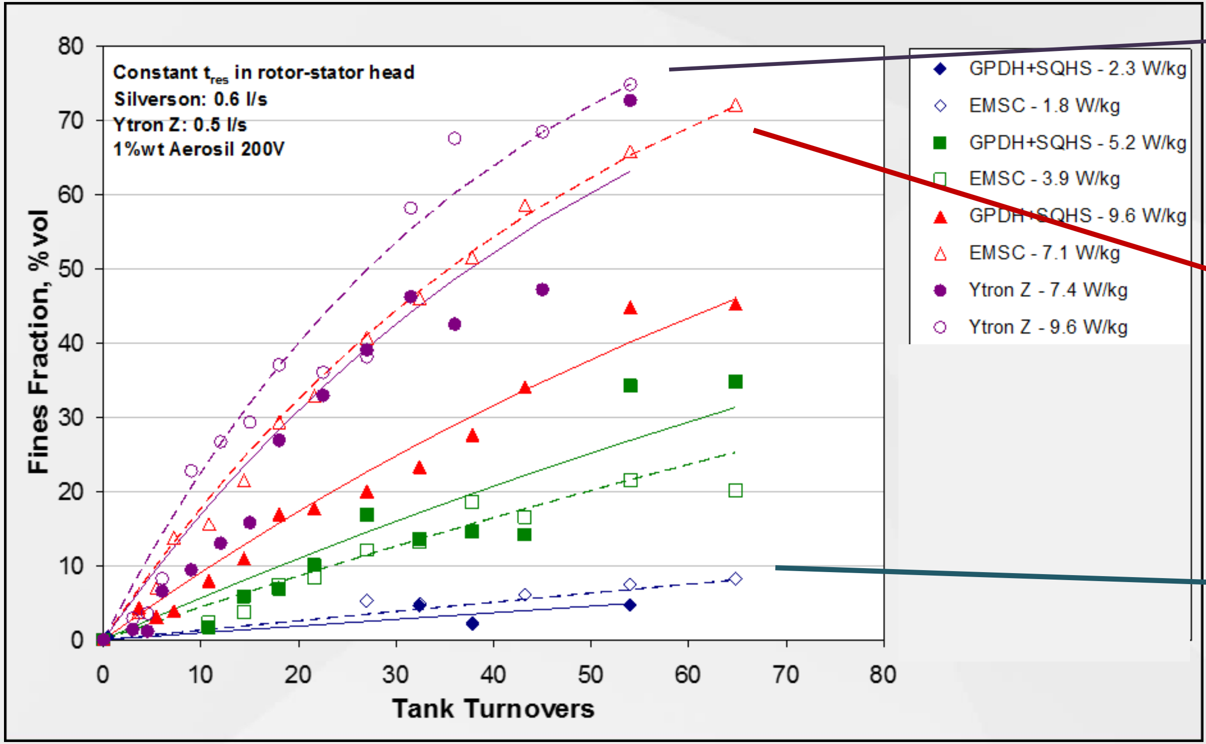
Mechanism of break up- primarily dictated by the material pair; regardless of the operating condition

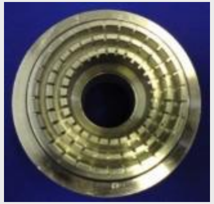
(Padron et al, 2008; Özcan-Taşkın and Padron, 2009)

Break up of nanoparticle clusters

- **Mechanisms of break up**
- **Kinetics of breakup**
- Dispersion fineness\ finest attainable size
- Dispersion rheology and homogeneity

Kinetics of breakup- equipment type





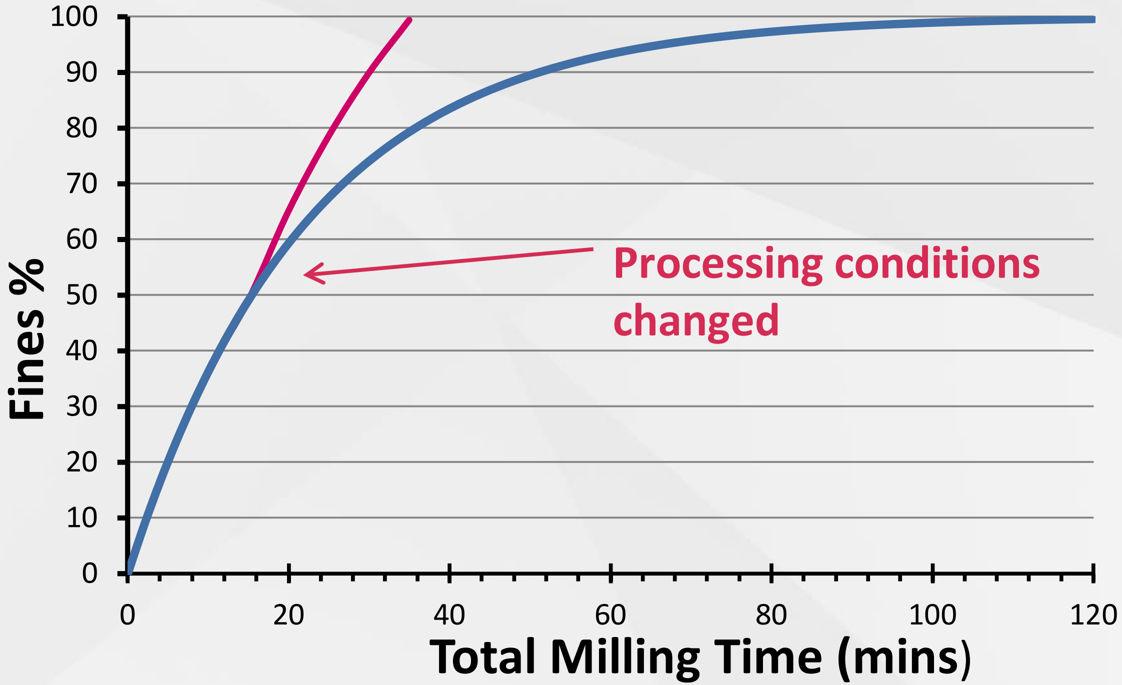
1 mm gaps between teeth

~1 mm holes

10 mm & 2.4 x 2.4 mm holes

(Özcan-Taşkın et al, 2016)

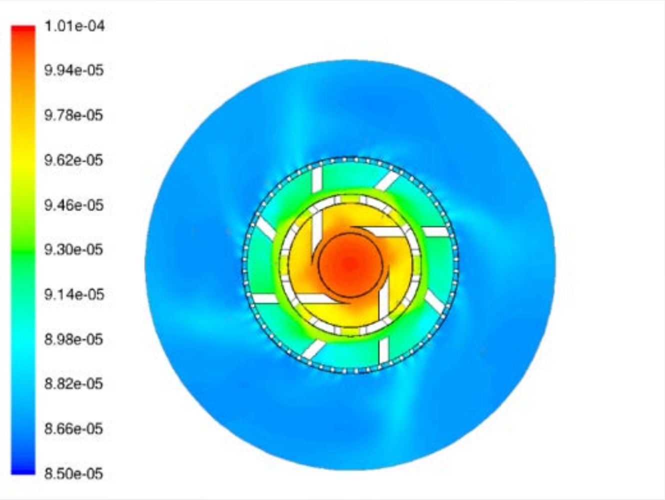
Breakup kinetics: Process optimisation – Stirred Bead Mill



Milling under given conditions required 120 minutes to reach 100% fines
2-stage process required 36 minutes to reach 100% fines.

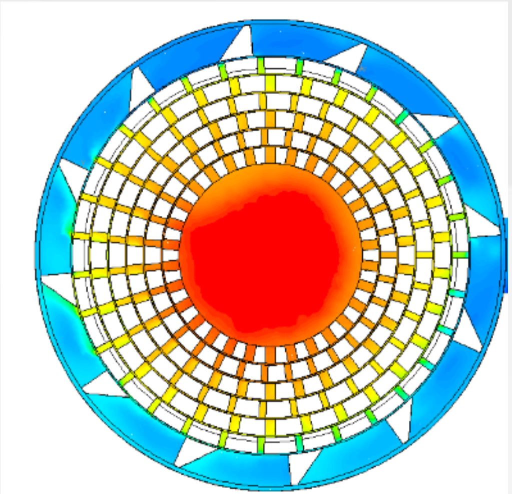
Example modelling results: in-line rotor-stator

GPDH-SQHS
N=9000 rpm,
Q=0.6 l/s, P=9.6 W/kg



d_{30}, μm

Ytron ZLab
N=7330 rpm,
Q=0.5 l/s, P=9.6 W/kg



(Özcan-Taşkın et al, 2016)

Break up of nanoparticle clusters

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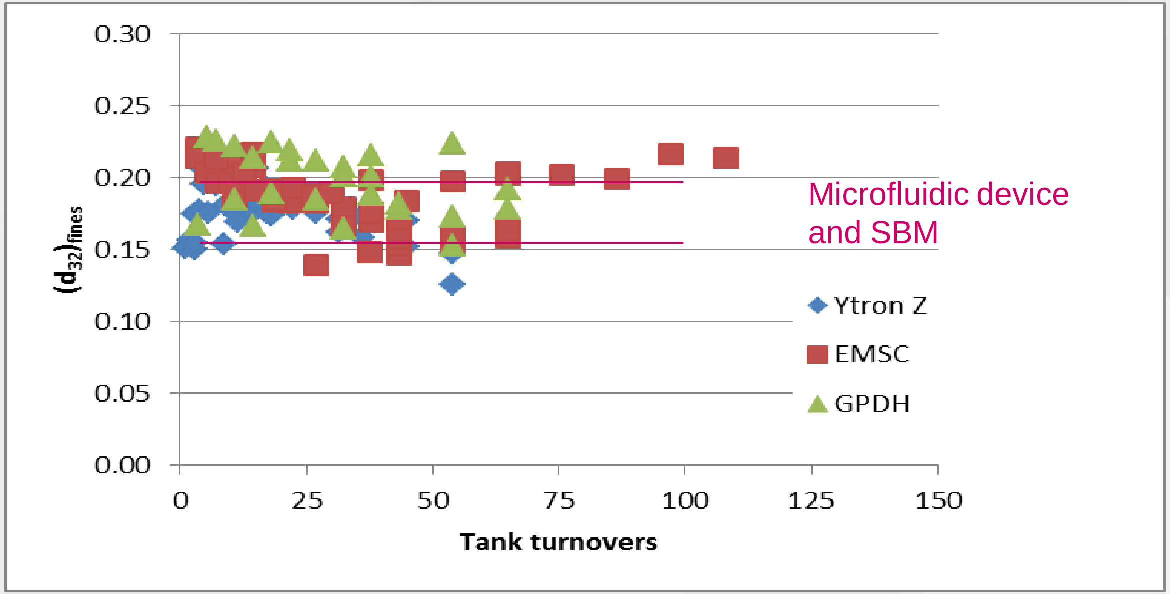
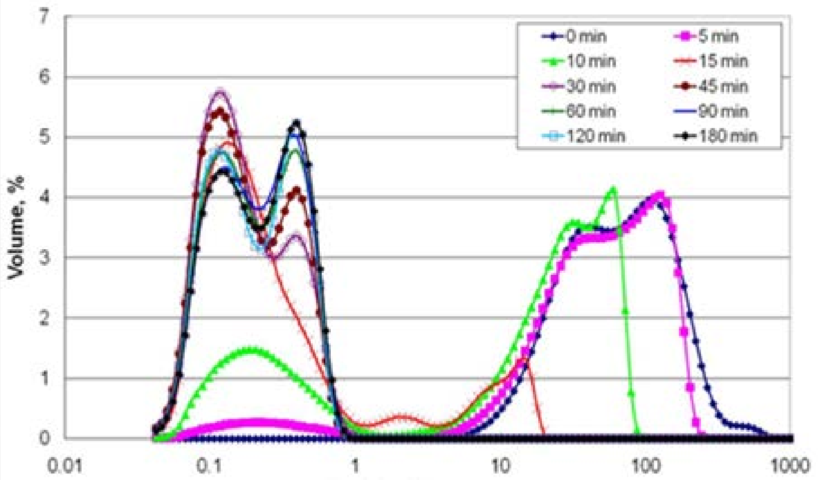
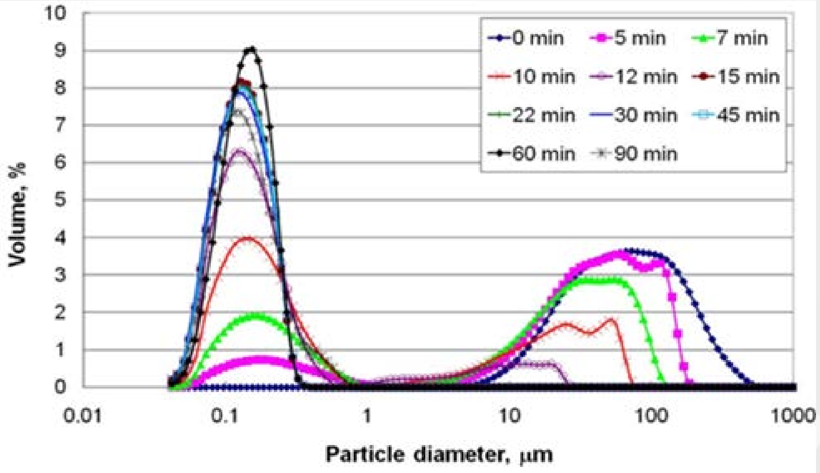
Smallest fragment\aggregate



For a given particle-liquid pair, smallest attainable size is independent of operating conditions, processing time or geometry. Breakup kinetics depends on all of these. *(Özcan-Taşkın et al, 2016)*

Damage ?





… would depend on the particle properties;
wear-and-tear of equipment may occur

Özcan-Taşkın et al, (2012)

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Break up of nanoparticle clusters

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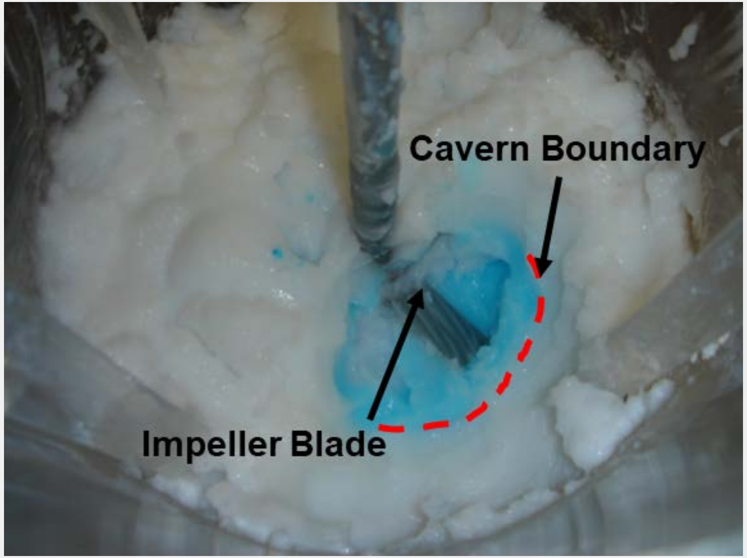
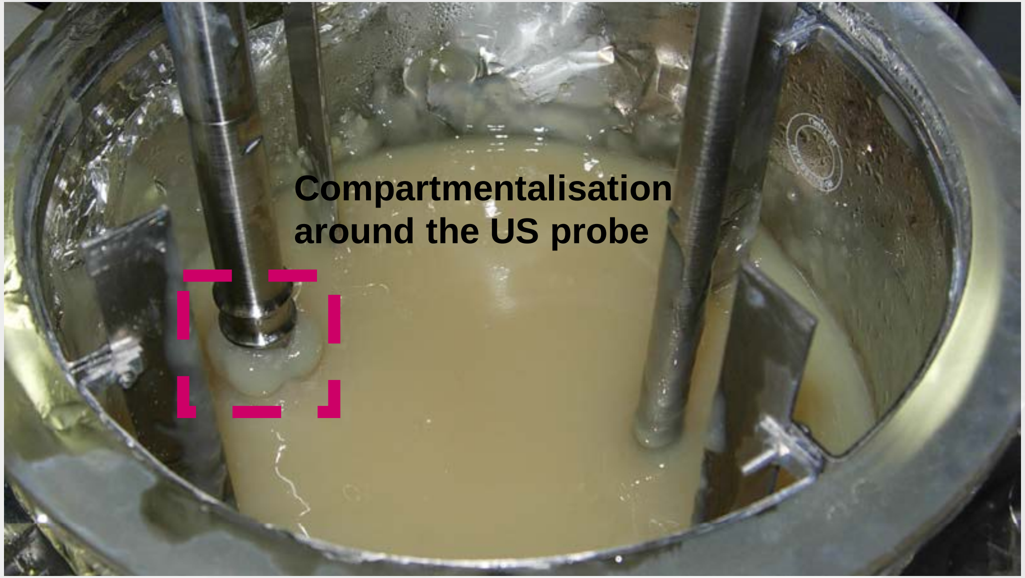
Dispersion rheology and …





… flow within/through the process device

Effect of Shear History on Rheology (15% Aerosil 200V)

Sample B: Milled and then left for 1 week

Sample A: Milled, left for 1 week then stirred

Concluding points

1. Mechanism of break up: erosion, rupture, shattering
2. Kinetics of break up
3. Dispersion characteristics: rheology, stability, smallest attainable size- aggregate or primary particle
4. Assess equipment performance: how fast, at what power input, … taking into account practical issues

for process design- selection of equipment type(s) & operating conditions and scale up

Concluding points

4. Assess equipment performance: how fast, at what power input, … taking into account practical issues

- is the performance affected by increasing viscosity and\or solids concentration?
- H&S of running a high intensity device
- is the equipment prone to frequent failure (due to wear and tear or blockage)?

for process design,

selection of equipment type(s) & operating conditions

scale up

Numerous collaborative projects

- PROFORM EC contract nb 505654-1
- AddNano EC Contract nb 229284
- DOMINO- Industrial consortium at BHR Group
- Loughborough University research projects

With contributions from many including

- Gustavo Padron
- Warren Eagles
- Dominik Kubicki
- Adi Utomo
- Ainee Cheah
- Tim Addison
- Emmanuela Gavi
- Carlos Fonte
- Neil Alderman
- Chris Rielly
- James Mitchell
- James Bacon

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University of Salerno	Willy Bachofen	Krafft
University of Maryland	Fuchs	Altana
Etchells Enterprises		

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