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

**Feed materials**
- Dry powders
- Continuous phase
- Other additives

**Incorporation**
- Stirred tanks, in-line devices including proprietary designs

**Break up\deagglomeration**
- Energy intensive devices
- In-line and batch rotor-stators, stirred bead mills, ultrasonicators, Microfluidics

**Product**

Loughborough University

#InspiringWinners since 1909
Selection of process devices

Stirred bead mill

High pressure devices

Microfluidics

Valve homogeniser

Ultrasonicator

Stirred tank
Dispersion of Nanoparticle Clusters in Liquids

Typically: 5-50 nm

O(100) nm

O(10-100) μm

Nanoparticles into a liquid

stronger clusters that tend to float

Primary Particles → Aggregates → Agglomerates
Break up occurs …

… through stresses acting on the agglomerates

Hydrodynamic stresses:

- Laminar flow: \( \tau = \mu \dot{\gamma} \)

- Turbulent flow: \( l \gg L_i \gg \lambda_k \)

\[ \tau \propto \rho \varepsilon^2 \frac{L^2}{L_i^3} \]

\[ \tau \propto \mu \left( \frac{\varepsilon}{V} \right)^{1/2} = \rho V^{1/2} \varepsilon^{1/2} \]

which are sufficiently high to overcome the tensile strength:

\[ \sigma_T = 1.1 \left( \frac{1 - e_a}{e_a} \right) \frac{F_{TOT}}{L_a^2} \]
Breakup of nanoparticle clusters

- Primary Particles
- Aggregates
- Agglomerates
- Shattering
- Rupture
- Erosion

Özcan-Taşkın et al, 2009
Mechanism of breakup- primarily dictated by the material pair; regardless of the operating condition.

(Padron et al, 2008; Özcan-Taşkin 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-Taşkin et al, 2016)
Milling under given conditions required 120 minutes to reach 100% fines. A 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

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

\[ d_{30}, \mu m \]

(Özcan-Taşkin et al, 2016)
Break up of nanoparticle clusters

- Mechanisms of break up
- Kinetics of breakup
- Dispersion fineness\ finest attainable size
- Dispersion rheology and homogeneity
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)
... would depend on the particle properties; wear-and-tear of equipment may occur

ÖZCAN-TAŞKİN ET AL., (2012)
Break up of nanoparticle clusters

- Mechanisms of break up
- Kinetics of breakup
- Dispersion fineness\ finest attainable size
- Dispersion rheology and homogeneity
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
Thanks are also due to many including:

Warsaw University of Technology, Bayer, Michelin
Karlsruhe University, Unilever, Total
Poznan University of Technology, Huntsman Polyurethanes
University of Birmingham, Solvay, P&G
Politecnico di Torino, GSK, Thomas Swan
Ecole Centrale de Lyon, Teva Pharma, Ytron Quadro
University of Salerno, Willy Bachofen, Krafft
University of Maryland, Fuchs, Altana
Etchells Enterprises

for challenging questions and fruitful discussions in the area