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Breakup of nanoparticle clusters using Microfluidizer M110-P

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ABSTRACT

A commercial design, bench scale microfluidic processor, Microfluidics M110-P, was used to study the deagglomeration of clusters of nanosized silica particles. Breakup kinetics, mechanisms and the smallest attainable size were determined over a range of particle concentrations of up to 17% wt. in water and liquid viscosities of up to 0.09 Pa s at 1% wt. particle concentration. The device was found to be effective in achieving complete breakup of agglomerates into submicron size aggregates of around 150 nm over the range covered. A single pass was sufficient to achieve this at a low particle concentration and liquid viscosity. As the particle concentration or continuous phase viscosity was increased, either a higher number of passes or a higher power input (for the same number of passes) was required to obtain a dispersion with a size distribution in the submicron range.

Breakup took place through erosion resulting in a dispersion of a given mean diameter range regardless of the operating condition. This is in line with results obtained using rotor-stators. Breakup kinetics compared on the basis of energy density indicated that whilst Microfluidizer M110-P and an in-line rotor-stator equipped with the emulsor screen are of similar performance at a viscosity of 0.01 Pa s, fines volume fraction achieved with the Microfluidizer was much higher at a viscosity of 0.09 Pa s.

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1. Introduction

Microfluidic processors are employed for power intensive processes such as size reduction in emulsions (Lee and Norton, 2013; Persson et al., 2014; Bai and McClements, 2016), solid–liquid dispersions (Yurdakul et al., 2012), liposomes (Lajunen et al., 2014), cell rupture (Choi et al., 1997; Stupak et al., 2015) or as reactors for the synthesis of nanomaterials (Chomistek and Panagiotou, 2009; Panagiotou et al., 2009). These processes are common to a wide range of industries from pharmaceuticals, nutraceuticals, inks, coatings and bioindustries.

This study was performed with a commercial design microfluidic processor to assess its performance in deagglomerating clusters of nanosize silica particles. The last two decades have seen rapid

uptake of nanotechnology with the development of new products of improved performance or formulations with properties that are not possible to achieve otherwise. The development of new formulations brings the requirement of process design and scale up to enable the market introduction of these novel products. Large scale manufacture of nanoparticles is more commonly achieved through flame pyrolysis with the resulting powder consisting of agglomerates and aggregates of primary particles. A key step during the manufacture of an intermediate or final product in the form of a liquid based nanoparticle dispersion is the deagglomeration of these clusters of nanoparticles to achieve a fine dispersion. Deagglomeration can occur through erosion, rupture or shattering as shown in Fig. 1a, resulting in different evolutions of the Particle Size Distribution (PSD) during the course of processing as shown in Fig. 1b (Özcan-Taşkın et al., 2009).

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Nomenclature	
d	Interaction chamber channel diameter (m)
d_{32}	Sauter mean diameter (area-weighted average particle size) (μm)
E_v	Energy density (MJ m^{-3})
l	Macroscale of turbulence (m)
L_i	Agglomerate size (m)
m	Mass of dispersion (kg)
P	Power input (W)
p_i	Operating pressure (Pa)
Q_i	Impulse flow rate ($\text{m}^3 \text{s}^{-1}$)
Re	Reynolds number ($4 Q_i \rho / \mu \pi d$) (-)
t_i	Time of impulse (s)
V_i	Volume of impulse (m^3)
Greek	
ε	Local energy dissipation rate per unit mass of liquid ($\text{m}^2 \text{s}^{-3}$)
$\dot{\gamma}$	Shear or extensional rate (s^{-1})
λ_k	Kolmogorov microscale (m)
μ	Shear or extensional viscosity (Pa s)
ρ	Density (kg m^{-3})
τ	Shear or extensional stress (Pa)
ν	Kinematic viscosity ($\text{m}^2 \text{s}^{-1}$)

Commonly used impellers, such as turbines and hydrofoils, do not provide sufficiently high levels of power input to achieve deagglomeration into the sub-micron range (Xie et al., 2007). Therefore, power intensive process devices such as sawtooth impellers (Xie et al., 2007), batch (Xie et al., 2007; Kamaly et al., 2017) or in-line rotor-stators (Baldyga et al., 2008; Padron et al., 2008; Özcan-Taşkın et al., 2016), high pressure jets (Wengeler et al., 2006; Sauter and Schuchmann, 2007) are

employed. The hydrodynamic stresses in the flow field generated in such devices are sufficiently high to overcome the tensile strength of the agglomerates in order for breakup to occur. In the laminar regime, shear and/or extensional stresses (τ , Pa) cause breakup:

$$\tau = \mu \dot{\gamma} \quad (1)$$

where μ (Pa s) the shear or extensional viscosity and $\dot{\gamma}$ (s) is either the shear or extensional rate. In the turbulent regime, breakup occurs as a result of turbulent stresses acting on agglomerates. Agglomerates may be of a size L_i such that $l \gg L_i \gg \lambda_k$, l being macroscale of turbulence and λ_k , Kolmogorov microscale:

$$\lambda_k = \frac{\nu^{3/4}}{\varepsilon^{1/4}} \quad (2)$$

where ν ($\text{m}^2 \text{s}^{-1}$) is the kinematic viscosity and ε ($\text{m}^2 \text{s}^{-3}$) the local energy dissipation rate per unit mass of liquid for which the frequency of turbulence (f) is defined as follows:

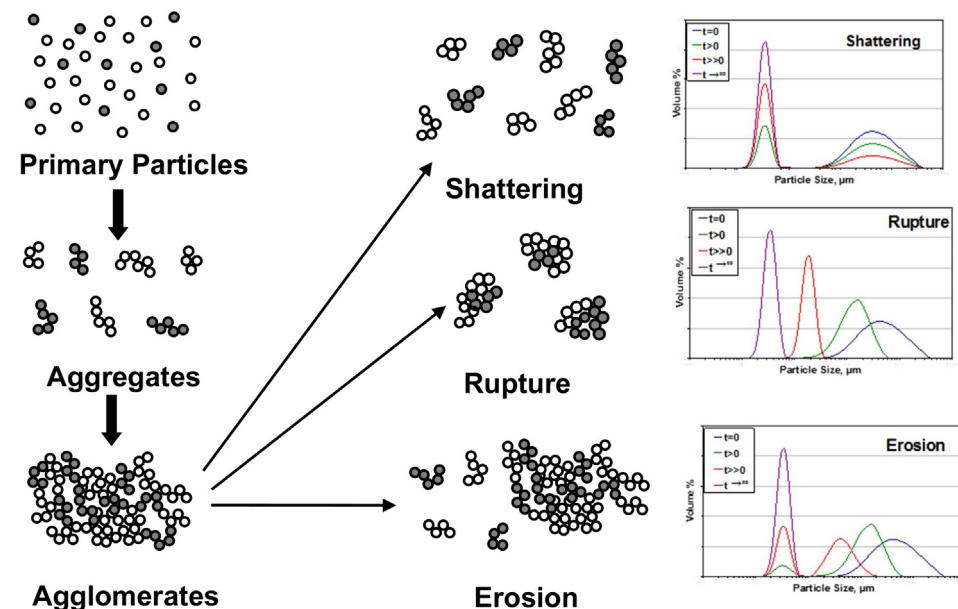
$$f = \frac{\varepsilon^{1/3}}{L_i^{2/3}} \quad (3)$$

These will be broken up through eddies in the inertial subrange of turbulence,

$$\tau \propto \rho \varepsilon^{2/3} L_i^{2/3} \quad (4)$$

where ρ (kg m^{-3}) is density. Agglomerates of a size $L_i < \lambda_k$ are broken up through viscous subrange eddies (Baldyga and Bourne, 1994):

$$\tau \propto \mu \left(\frac{\varepsilon}{\nu} \right)^{1/2} = \rho \nu^{1/2} \varepsilon^{1/2} \quad (5)$$



a Mechanisms of breakup of nanoparticle clusters

b Evolution of the PSD for different breakup mechanisms (Özcan-Taşkın et al., 2009)

Fig. 1 – (a) Mechanisms of breakup of nanoparticle clusters. (b) Evolution of the PSD for different breakup mechanisms (Özcan-Taşkın et al., 2009).

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