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Original Research Paper

Dispersion of clusters of nanoscale silica particles using batch rotor-stators

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ABSTRACT

Nanoparticle powders added into a liquid medium form structures which are much larger than the primary particle size (aggregates and agglomerates)-typically of the order of 10's of microns. An important process step is therefore the deagglomeration of these clusters to achieve as fine a dispersion as possible. This paper reports the findings of a study on the dispersion of hydrophilic fumed silica nanoparticle clusters, Aerosil 200 V, in water using two batch rotor-stators: MICCRA D-9 and VMI. The MICCRA D-9 head consists of a set of teeth for the stator and another for the rotor, whereas the VMI has a stator with slots and a rotor which consists of a 4-bladed impeller attached to an outer set of teeth. The dispersion process, studied at different power input values and over a range of concentrations (1, 5, 10 wt.%), was monitored through the evolution of PSD. Erosion was found to be the dominant breakage mechanism irrespective of operating conditions or rotor-stator type. The smallest attainable size was also found to be independent of the power input or the design of the rotor-stator. Break up kinetics increased upon the increase of power input, and this also depended on the rotor-stator design. With MICCRA D-9 which has smaller openings on both the stator and rotor, the break up rate was faster. Increasing the particle concentration decreased break up kinetics. It could also be shown that operating at high concentrations can still be beneficial as the break up rate is higher when assessed on the basis of specific power input per mass of solids.

1. Introduction

The incorporation of nanoparticles in the formulation of a wide range of products varying from pharmaceuticals, coatings, paints, lubricants to nutraceuticals, textiles has seen a rapid increase over the past two decades resulting in not only the improvement of conventional products but also allowing products to be brought to market with properties and performance that could not be achieved previously. Dry nanoparticle powders exist in a hierarchy of structures which may include primary particles but most often agglomerates and aggregates. Whilst agglomerates can be broken up to much finer structures, aggregates cannot be disintegrated further due to the strong bonds holding them together. Therefore, once the particles are incorporated into the liquid, the main objective of the process is to deagglomerate the large structures to generate as fine a dispersion as possible.

Provided that the stresses present in a processing environment are sufficiently high to overcome the inter-particle bonds that hold the agglomerates together, break up can occur through different mechanisms as schematically shown in Fig. 1 [8]. Erosion and shattering give bi-modal Particle Size Distributions (PSD). As small fragments are eroded from the surface of larger agglomerates, the size distribution of coarse material (agglomerates) shifts to the left (Fig. 2). When a large agglomerate shatters into the smallest attainable fragments (often aggregates and where possible primary particles), the volume fraction of the coarse material decreases in time and that of fines increases as shown in Figs. 1 and 2. In the case of rupture, which occurs through the gradual fragmentation of large agglomerates into smaller ones, the PSD moves to the left, also becoming narrower (Figs. 1 and 2).

The process is typically carried out with power intensive devices. At high Reynolds numbers, turbulent stresses acting on agglomerates result in breakage. Agglomerates of a size, L_i, greater than the Kolmogoroff length scale, λ_k, smaller than macroscale of turbulence, l, i.e. $l \gg L_i \gg \lambda_k$, are broken up by inertial stresses:

$$\tau \propto \rho \varepsilon^{2/3} L_i^{2/3} \quad (1)$$

ε being the local energy dissipation rate per unit mass of liquid. Agglomerates which are smaller than the Kolmogoroff length scale,

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Nomenclature

C	impeller off-bottom clearance (m)	*Greek*	
D	rotor diameter (m)	ε	local energy dissipation rate per unit mass of liquid ($m^2\,s^{-3}$)
d_{32}	Sauter mean diameter	σ	diameter of surface atom (m)
H	Liquid height in tank (m)	λ_k	Kolmogorov microscale (m)
l	macroscale of turbulence (m)	μ	dynamic viscosity ($kg\,m^{-1}\,s^{-1}$)
L_i	agglomerate size (m)	ν	kinematic viscosity ($m^2\,s^{-1}$)
P	power input (W)	ρ	density ($kg\,m^{-3}$)
T	tank diameter (m)	τ	stresses acting on particle clusters (Pa)

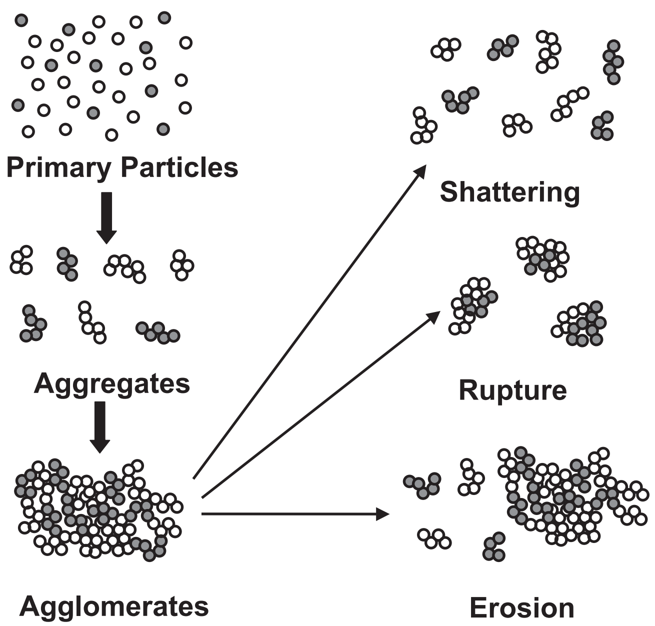

Fig. 1. Schematic representation of breakup of nanoparticle clusters through erosion, rupture and shattering [8].

$L_i < \lambda_k$, broken up by viscous subrange eddies as proposed by Baldyga et al. [2]:

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

ν being the kinematic viscosity of the liquid. Different process devices used for the purpose include the stirred bead mill [17,7,15], ultrasonic dispersers [2,3,13] in-line rotor-stators [2,12,10], a batch rotor-stator [20] or high pressure devices [14,16,20]. Local energy dissipation rate in these devices, responsible for the breakup of agglomerates, can be orders of magnitude higher than the average energy dissipation rate. Some of the process devices, such as the ultrasonic disperser, are more commonly used during the formulation stage, whilst others, for example the in-line rotor-stator, are more suited for large scale operation. It is therefore not uncommon to use different devices at different scales of operation which makes process scale up a challenging task. A knowledge of the comparative performance of different process devices provides valuable input for the design and scale up of such processes.

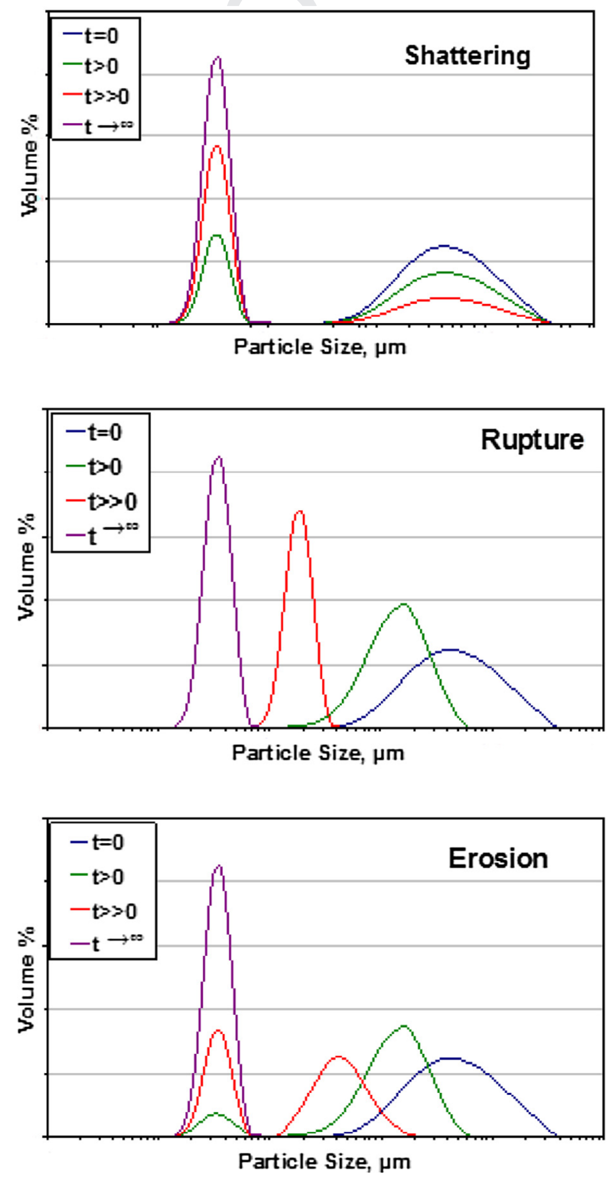

Fig. 2. PSDs resulting from different breakup modes [8].

This study was undertaken to assess the comparative performance of two batch rotor-stators as there is no published information on these designs for the breakup of nanoparticle clusters. The effects of varying power input on the smallest attainable size, mechanisms and kinetics of breakup were investigated. The test material used Aerosil 200 V, is available in large quantities on the market and widely used in numerous products to improve mechanical properties, for example to reinforce rubber, as rheology