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De-agglomeration of Nanoparticles in a Jet Impactor-assisted Fluidized Bed

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Abstract

Nanoparticles in agglomerated state lose their outstanding properties; hence, it is essential to break them up prior to use and prevent the re-agglomeration. Even though there are several dry techniques to disperse nanopowders, none of them have been able to produce truly nanoscale aerosols so far. Here, we study de-agglomeration of dry silica nanopowder via a Jet Impactor-assisted Fluidized Bed (JIAFB). The particle size distribution of fragmented powders was characterized by in-line Scanning Mobility Particle Spectrometry (SMPS) and offline Transmission Electron Microscopy (TEM). In order to ascertain the jet length and that the kinetic energy of particles is sufficient for de-agglomeration, a CFD simulation was carried out. Both SMPS and TEM measurements imply that at certain fluidization velocity, increasing the jet velocity shifts the particle size distribution towards smaller diameters, and at higher velocities the mode value reduced from 113-130 to 55-60 nm. However, the geometric standard deviation or degree of polydispersity rises from 1.5 to 2.0 by increasing the jet velocity up to 197 m/s, as it will increase the total superficial velocity and consequently entrainment of larger particles from the bed. In addition, the TEM results indicate that the range of individual particle size in supplied nanopowder is wide; hence, increasing the geometric standard deviation can be an indicator of higher level of agglomerate dispersion.

Keywords

Fluidized bed, nanoparticles, de-agglomeration, impaction, particle size distribution, interparticle forces

1. Introduction

Nanoparticles, due to their high surface area-to-volume ratio and free atoms on their surfaces, have a tendency to assemble together as well as absorb a wide range of molecules such as water, oxygen, etc. In other words, these features will lead the particles to have high surface energy, become unstable and very cohesive. Therefore, individual nanoparticles, in order to reach to a lower energy state, attract each other

and form assemblages under the influence of some external and internal interparticle forces such as van der Waals, electrostatic, and capillary forces [1]. These friable and readily dispersed assemblages of particles are called agglomerates or "soft" agglomerates and can be formed during production, transportation or storage as a result of Brownian motion, collisions, and pressure arising from stacking. In this form, particles lose the extraordinary surface-driven properties they had as individual nanoparticles. In order to take advantage of their “nanoproperties”, it is necessary to break up the agglomerates and reduce their high surface energy, or "passivate" them, before use. The ability to produce bulk quantities of highly dispersed nanoparticles is a significant limitation of nanotechnology [2].

De-agglomeration of nanopowder can be performed in gas [3-11] or liquid phase [12-17], and a variety of theoretical and experimental studies have been conducted (Table 1). Various techniques have been developed to disperse nanoparticles in fluids, typically through use of mechanical and acoustic energy. Ultrasonication is a well-known technique to disperse nanoparticles homogeneously in suspensions using acoustic energy. Through acoustic cavitation and streaming, the formation, growth and implosion of bubbles occurs, resulting in the rupture of agglomerates. Time, power and irradiation modes (continuous or pulsed) are the key parameters affecting dispersion quality in an ultrasonic bath. Nguyen et al. [16] showed that there is an optimum power input in ultrasonication: past a certain point, a higher vibration amplitude will not improve dispersion quality, but will actually increase the re-agglomeration rate. High-speed revolution shearing, milling [14], and high-pressure homogenizers [13] are the main mechanical dispersion approaches. In milling, dispersed nanoparticles are introduced from the bottom of the mill in a slurry. Agglomerates are broken by passing through the stirrer, impinging the beads, and being stirred by rotating pins. In the upper part of the mill, the beads and slurry are separated by centrifugation and the dispersed-particle containing slurry is discharged. Similar to sonication, Inkyo et al [14] also indicated that there is an optimum time for milling, after which re-agglomeration occurs.

While liquid phase techniques are generally effective at dispersing nanoparticles, they do not directly address the issue of re-agglomeration. To stabilize the suspension, additives must be supplied to provided electrostatic, steric and electrosteric repulsion effects. Surfactants are commonly used for this purpose, resulting in electrostatic repulsion between surfactant-coated nanoparticles [18] and reducing particle agglomeration caused by attractive van der Waals forces [19]. However, surfactants face several limitations, not the least of which is their thermal instability: surfactants can desorb from nanomaterials at relatively low temperatures (65-70 °C) [20] , cancelling out their repulsive properties.

Gas phase methods have several advantages over liquid-phase approaches, such as the absence of solvent waste, the simplification of downstream separation, the feasibility of continuous processing, and the versatility with respect to particle material and size and structure [21]. The de-agglomeration of

nanoparticles down to their constituent primary particles in the gas phase can be achieved by applying an external force larger than the interparticle forces. There are several methods to de-agglomerate nanoparticle clusters in the gas phase ranging from high energy (e.g. rapid expansion of supercritical suspensions [3] or low pressure single stage impactors [4]) to low energy (e.g. fluidized bed [5]). However, so far, the low energy methods have not been able to produce sub-100 nm particle sizes, and the high energy methods have significant scale-up issues because of their operating conditions. Indeed, the rapid expansion of supercritical suspension systems requires high pressures (1.9–7.9 MPa). Nurkiewicz et al. [6] presented a nanoparticle aerosol generator consisting of a vibrating fluidized bed with a baffle, a vibrating Venturi disperser and a cyclone separator. Although they discussed the de-agglomeration of nanopowders and preventing re-agglomeration (strictly through dilution), the investigation was mostly focused on generating nanoparticle aerosols at constant particle concentration over time to perform inhalation studies, limiting scale-up potential. In order to break the agglomerates in a controllable and scalable manner, further investigation is necessary, namely on interparticle forces and attaining the required de-agglomeration energy in a fluidized bed configuration.

In this work, we break the large fractal-shaped agglomerates of silica nanoparticles to smaller clusters continuously through use of a jet impactor-assisted fluidized bed (JIAFB). The force required to destroy the agglomerates is controlled by the gas jet velocity in the impaction zone. Calculating the impaction velocity determines the kinetic energy of particles upon impaction, making it possible to measure the theoretical fragmentation degree of nanoparticles. As the agglomeration is a reversible phenomenon, in order to produce stable particles, reducing interparticle attraction and preventing the nanoparticles from re-agglomeration are inevitable. Therefore, following agglomerate destruction, the JIAFB includes a surface functionalization post-treatment, based on photo-initiated chemical vapor deposition (PICVD) [22], to ensure particle stability and prevent re-agglomeration (not reported here).

2. Experimental

2.1 Materials

Silica nanopowder manufactured by TEKNA™, via thermal plasma synthesis, were used for all fluidization experiments. Primary particles have an average diameter of 20 nm. The powder's specific surface area is 200 m^2/g, with a solid density of 2200 kg/m^3, and bulk density of 35 kg/m^3. As nanopowders are strong absorber of humidity, the particles were dried at 140 °C and -70 kPa vacuum before any fluidization experiments. Argon was used as fluidizing gas and air was used as jet and diluter gas in all experiments.

2.2 Experimental Set-Up

The JIAFB consists of a jet and an impaction plate which are placed inside the bed, as well as an air-driven venturi pump installed on the outlet of the column (Fig. 1). The JIAFB uses a high-speed jet to accelerate the fluidized agglomerates onto the impaction plate. The venturi pump provides vacuum in order to entrain out broken particles, as well as minimize re-agglomeration of dispersed particles by diluting the outlet flow and send it to the particle sizer (dilution ratio of 1:25). The particle size and mass concentration produced by the JIAFB was measured utilizing an in-line Scanning Mobility Particle Spectrometer (SMPS), operated in aerosol mode. A calibrated rotameter was used to set the inlet flow rate to the particle sizer. A cylindrical quartz tube was used as the fluidized bed with an internal diameter of 8 mm and a height of 60 cm. After passing through a porous glass wool distributor, argon gas is introduced to the quartz tube. The superficial gas velocity was adjusted using a mass flow controller. A 250-micron jet was installed vertically, upwards, by passing through the distributor. The compressed air flow was supplied for the high speed jet after passing through a mass flow controller. An impaction plate welded to a stainless steel rod was introduced to the fluidized bed from the top. The jet-to-plate distance was set to 5 mm. For Transmission Electron Microscopy (TEM) sampling, four sites were selected on the lateral surface of the rod to attach the TEM grids.

In order to study the effect, if any, of the fluidized bed system on the particle size distribution, the setup was configured in a "bypass" mode (Fig 1.b). In this case, the supplied silica nanopowder container is connected directly to the venturi pump. The connection to the SMPS remained the same, and the same air flow rate used during normal experiments was supplied to the venturi to generate vacuum.

2.3 Measurement Methods

A SMPS (TSI Inc., Shoreview, MN., USA) was used to measure the real time number distribution of the particles in the outlet stream of fluidized bed. TEM was performed on a JEOL JEM-2100F operated at 200 kV in bright-field imaging to confirm the effect of impaction on dispersion. The particles were collected on TEM grids (Electron Microscopy Sciences, CF-400-Cu, carbon film on 400 square mesh copper grid), directly from the aerosol phase.

3. Results and discussions

In order to ascertain that the jet length and applied kinetic energy are sufficient for de-agglomeration, a single-phase computational fluid dynamics (CFD) simulation in the vicinity of the jet and plate was performed for all jet velocities using the ANSYS Fluent 6.3 CFD software. The two-dimensional axisymmetric case was solved isothermally at constant room temperature and the conservation equations for momentum were solved for a computational domain. The geometry was defined as an 8 mm by 50 cm

rectangle. A 250 μm line was defined as the tip of the jet and a rectangular impaction plate (5 mm by 1.5 mm) was place 5 mm from the jet. The geometries of the computational domains were generated and meshed by Gambit 2.4.6, leading to a multi-zone grid structure in which cell sizes ranged from 8×10^{-5} m near the impaction plate to 2×10^{-3} m near the outlet. Downstream from the impaction plate, mesh sizes were stretched away with the length of the cells adjacent to the impactor section. The geometries were meshed with quadrilateral meshes, that, after grid adaption to y-plus to meet the mesh requirement of the near-wall modeling of turbulence, gave a total number of cells of around 8×10^{4}. Air was selected as a compressible fluid flow medium. The air jet velocity changes from 33 to 197 m/s, while the co-flow velocity outside of the jet is 0.2 m/s. The Reynolds numbers for the jet, based on the outlet velocity and the inner diameter of the jet, lay approximately in the range of 3 to 8×10^{3}, indicating that the jet is fully turbulent. Hence, k-ε turbulent physics were used for the case of stationary studies. The turbulence intensity was assumed to be 2% for the subsonic jet. The boundary conditions were the fluid inlets for the fluidizing gas (0.2 m/s – $U/U_{mf} = 6.2$) and jet velocity (ranging from 0 to 197 m/s) at x=0 as well as fluid outlets at x=50. The other boundaries, including wall column and impaction plate, were considered as wall. Also, as the velocity field close to the wall (impaction plate) is crucial for turbulence modeling, enhanced wall treatment was selected as the wall function according to the y-plus value. To apply the enhanced wall treatment, a fine mesh that can resolve the viscosity-affected near-wall region was defined. A y-plus value less than 5 is considered acceptable, as long as it is well inside the viscous sublayer (FLUENT 6.3 User's Guide). For the worst flow condition (highest jet velocity), y-plus was checked after the solution had converged. When y-plus did not fulfill the requirement, grid adaptation was applied and the solution was processed again. As an example, the resulting velocity field in an axial symmetry view at the maximum jet velocity of 197 m/s is shown in Fig 2. The CFD simulation does not account for the motion of particles, but it serves as a gross approximation to determine the maximum kinetic energy of per unit mass of particles based on the gas velocity field, calculated as half of the near-wall velocity squared (Table 2). This near-wall velocity term implicitly accounts for turbulent kinetic energy through an artificial viscous term (calculated through the RANS k-ε turbulent physics model). At a jet velocity of 197 m/s, the gas velocity at the impactor is on the order of 100 m/s. Froeschke et al. [10] showed that the energy required to break up large fractals (with fractal dimension of 1.7-2.7) of air born metal oxide nanoparticles with primary particle sizes on the order of 6 to 95 nm lay in the range of 1 to 10×10^{3} m^2/s^2. For instance, in their experiments, titania agglomerates could be almost completely fragmented at a kinetic energy per unit mass of particles corresponding to 8×10^{3} m^2/s^2. Comparing the amount of kinetic energy provided for fragmentation in the present study with their numbers demonstrates that impaction velocities greater than 48 m/s lay within the acceptable range to provide the kinetic energy to break-up the agglomerates into smaller clusters. The agglomerates can further be fragmented to their constituent

nanoparticles, provided that the gas jet velocity is higher than 99 m/s. Particle size measurements confirm this by showing a significant drop of the mode size at both fluidization velocities (Table 3). The jet penetration length was calculated according to correlations proposed by Guo [23] and Hirsan [24] for upward jets in a fluidized bed (Table 2). The jet length penetration values indicate that the jet of air, even at the lowest velocity, can still penetrate through the bed and touch the plate.

In this system, two principal parameters affect the choice of an appropriate fluidization velocity: the impactor system level and the particle entrainment rate. The superficial gas velocity must be high enough that the bed can reach the impactor system level (i.e. higher than the jet outlet), but not too high that it leads to a high entrainment of particles beyond the impactor plate. In this particular configuration, for $U/U_{mf} < 5.5$, the jet cannot capture particles and bring them into the jet-impaction system. Conversely, for $U/U_{mf} > 6.2$, particle entrainment becomes high, thereby increasing the particle concentration in the aerosol outlet stream beyond the specifications of the SMPS. Therefore, the fluidization velocity range selected for this work was between $U/U_{mf} = 5.5$ and 6.2.

Fig. 3 shows the particle size distributions of the silica nanoparticles at constant fluidization velocity ($U/U_{mf} = 5.5$) and different jet velocities, running from 0 to 197 m/s. All data are number-weighted and show the electrical mobility diameter of particles. The geometric mean diameters differ from the median diameters by 0-12% and follow the same trend with increasing jet velocity. The graphs show that under the same fluidization conditions, by increasing the impaction velocity, the particle size distribution shifts toward smaller sizes for jet velocities greater than 99 m/s, reaching their smallest values (below 100 nm) at a jet velocity of 197 m/s. However, the geometric standard deviation (GSD), as a dispersity criterion of particle size distribution, increases at the same time (Table 3). This can happen owing to the fact that increasing the local jet velocity increases the total superficial velocity, which in turn leads to entrainment of larger particles. Furthermore, due to the wide range of individual nanoparticle sizes in the supplied nanopowder, increasing the GSD can actually be an indicator of higher level of agglomerate dispersion. On the other hand, as seen in Fig. 3, by increasing the jet velocity the overall particle concentration increases. Indeed, increasing the superficial velocity in bed increases the particle loss from the bed.

In the bypass configuration, the particle size distribution of the nanopowders does not change considerably compared to the control case of (jet velocity = 0 m/s), confirming that de-agglomeration occurs mainly as a result of impaction inside the bed, not through collisions in the fluidized bed.

Finally, TEM images and geometric particle size distribution obtained from them confirm the effect of impaction on dispersion of dry agglomerates (Fig. 4). The non-impacted sample was collected on TEM grids by installing the TEM grid on the lateral surface of the rod close to the bed. This allows fluidized

particles to sit on TEM grids only by diffusion mechanism. Dendritic structures with high fractal-like dimensions are observed, typical of silica nanoparticle agglomerates in the gas phase. Particles collected on a TEM grid attached to the impaction plate, following treatment with a jet velocity of 99 m/s (Fig. 4, right) are dispersed into small clusters. ImageJ [25] was used to analyze the TEM micrographs and extract particle size distributions, implementing watershed separation after the threshold was set to neutralize any shielding effects. Particle sphericity close to unity facilitated calculations of the particle diameter from the surface area. To plot the histogram, image artefacts showing diameters smaller than the primary particle size (20 nm) were eliminated. A large number of small primary particles ranging from 20 to 550 nm were identified after impaction, three times greater than the number of discrete particles before impaction. After impaction, almost 80% of particles were smaller than 100 nm, with the remainder composed of larger individual particles and some small clusters. On the other hand, before impaction, the size of agglomerates reached 900 nm. It should be noted that TEM sampling was conducted directly at the impactor level – it therefore illustrates the trend of de-agglomeration, but the size distribution is different from that observed downstream at the SMPS. In addition, TEM results revealed the supplied nanopowder have a wide primary particle size distribution in the range of 20 to 500 nm. This affects the fluidization behavior, the agglomeration/de-agglomeration rate of powders, and accounts for some of the increased GSD.

4. Conclusion

This experimental work forms the basis of our efforts towards a controllable technique for nanoparticle dispersion in the gas phase. Here, we showed that utilizing a high speed jet and impaction plate in a fluidized bed can increase the de-agglomeration rate of nanoparticles and produce particles with a smaller mode size. The results of CFD simulation reveal that the kinetic energy of particles upon impaction is high enough to break up the agglomerates, which is confirmed by SMPS monitoring. TEM results, also, confirm the effect of impaction on fragmentation of large agglomerates to smaller clusters and individual nanoparticles. As confirmed by a bypass configuration study, the de-agglomeration rate is strictly controlled by jet velocity, which governs the impaction mechanism. However, the effect of re-agglomeration inside the fluidized bed after impaction remains unknown and will be the focus of on-going work. Furthermore, to overcome reagglomeration of nanoparticles, their surface energy needs to be reduced by surface functionalization which will be the focus of upcoming work.

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