



# Effect of particle mixing on the hydrodynamics of fluidized bed of nanoparticles



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## ARTICLE INFO

### Article history:

Received 3 August 2016

Received in revised form 12 January 2017

Accepted 13 January 2017

Available online 15 January 2017

### Keywords:

Fluidization

Nanoparticles

Hydrodynamics

Particle mixing

Bed contraction

Deagglomeration

## ABSTRACT

In this study, we investigate the effectiveness of particle mixing technique to improve the fluidization hydrodynamics of nanoparticles that show strong agglomeration behavior during their dry processing. We choose small proportions of particles belonging to Geldart group A classification as additive particles because of their superior hydrodynamic behavior. This strategy not only suppresses the hysteresis phenomenon, but also helps to substantially reduce the size of agglomerates. Moreover, mixing of external micron-sized Geldart group A particles with resident nanoparticles of fluidized bed causes a variation in the volume-change of mixing in the range of –25% to –60%, indicating a significant contraction of the bed. Conventional models reported in the literature were unable to describe such a high degree of volume contraction. Results obtained in this study clearly establish the efficacy of particle mixing as an effective assisted fluidization technique in eliminating bed non-homogeneities and promoting the deagglomeration of nanoparticles.

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## 1. Introduction

An important challenge in the large-scale application and processing of nanoparticles is their agglomeration. While the primary size of nanoparticles ranges from 1–100 nm, their particle size analysis however often yields size distribution in the range of 1–200  $\mu\text{m}$ . This clearly implies the occurrence of large agglomerates, which are several thousand times larger than the actual size of nanoparticles. The phenomenon of agglomeration severely compromises the available surface area of nanoparticles, thereby affecting their efficacy in most applications. Therefore, in order to realize full potential of nanoparticles, it is important to develop strategies to promote their de-agglomeration.

For large-scale processing of fine and ultrafine particles, fluidized beds are gaining increasing attention because of low pressure drop, high heat and mass transfer rates and better contact between the fluid and solid phases [1–4]. Their hydrodynamics however critically depends upon the size and density of solid particles. Geldart [5] investigated this phenomenon in detail and classified the particles into four groups depending on their fluidization behavior. For example, group A particles exhibit smooth fluidization marked by a particulate expansion of the bed, whereas group B particles display bubbling behavior above the incipient fluidization. Meanwhile, fine particles with sizes <20  $\mu\text{m}$  (classified as Geldart group C) are often cohesive in nature, and are therefore difficult to fluidize. Such

particles show poor fluidization hydrodynamics and usually develop channels or plugs that lead to severe bed non-homogeneities, resulting in poor contact and low heat/mass transfer rates between phases [6–8].

In order to improve the fluidization hydrodynamics and promote de-agglomeration, conventional fluidization is often supplemented with additional input of energy to overcome interparticle forces that cause agglomeration. Such techniques include external or internal vibrations of the fluidized bed [9–11], use of ultrasonic comminution device [12], shear mixers [13], oscillating particles under magnetic field [14,15], and acoustic vibrations [16–19]. Instead of direct input of energy, other assisted fluidization techniques such as pulsated fluid flow [4, 20–24], use of tapered or tilted columns [25,26], and addition of inert particles have also been suggested [27–29]. Of particular interest is the particle-mixing strategy, which can be easily implemented without the expense of energy or without change in the fluidization setup. By choosing external particles with suitable physical properties, the interparticle force equilibrium of resident solid particles of the fluidized bed can be altered, thereby improving their fluidization hydrodynamics. For example, addition of inert particles conforming to Geldart group A classification, in small proportions of 5 vol% and 9 vol%, improved fluidization behavior of difficult-to-fluidize cohesive powders [27]. Even the addition of small fractions of external particles helped to enhance the bed homogeneity and improve the fluidization quality. The effect of particle mixing was also clearly evident on the power spectra of pressure fluctuations, which showed clear peaks compared with the complex and broadband spectra of the unassisted conventional fluidization.

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Later studies on particle mixing using additive sand to improve the fluidization of nano-powder yielded encouraging results [19], and pointed the need of detailed investigation of bed hydrodynamics. On the other hand, Song et al. [29] added relatively high proportions (30%–50%) of three different types of coarse particles (65–120  $\mu\text{m}$ ) to improve fluidization and suppress the elutriation of silica and titania nanoparticles. Best results were obtained by using smaller coarse particles with a size range of 65–80  $\mu\text{m}$ . They fitted the bed void fraction versus superficial velocity data with the Richardson–Zaki equation to obtain the exponent  $n$  and the apparent particle terminal velocity. The inter-agglomerate void fraction varied in the range of 0.65–0.8 in their fits while the overall void fraction of the bed, when computed from the bulk density data, could have been as high as 0.966. Both terminal velocity and exponent  $n$  increased with the increase in the proportion of external particles. Interestingly, external coarse particles that yielded best results in their experiments belonged to group A. Recently, Duan et al. [28] added various kinds of external particles (38–1000  $\mu\text{m}$ ) in binary mixtures of  $\text{SiO}_2$  and  $\text{ZnO}$  nanoparticles. Larger additive particles of  $\text{Al}_2\text{O}_3$  failed to yield any tangible improvement in the fluidization behavior, whereas the addition of smaller FCC particles was clearly effective. The external FCC particles used in these experiments belonged to Geldart group A, and  $\text{Al}_2\text{O}_3$  samples belonged to group B. Duan et al. processed their bed expansion data using the Richardson–Zaki correlation, and found that increasing the fraction of FCC particles helped to increase the correlation index  $n$  and led to a decrease in the minimum fluidization velocity. Note that the inter-agglomerate void fraction for static beds was assumed to be from 0.15 to 0.25 in their work, while the overall bed void fraction was 0.957 and 0.910 for the mono-component beds of  $\text{SiO}_2$  and  $\text{ZnO}$  nanoparticles, respectively.

In this study, we investigated the effectiveness of the particle mixing technique to improve the fluidization hydrodynamics of silica nanoparticles, which are widely used in the paint and pharmaceutical industries. Its dry particle size analysis yields the Sauter mean diameter as 20  $\mu\text{m}$  while its actual size is 12 nm [23,29]. As a result, when fluidized, these nanoparticles exhibit agglomerative bubbling fluidization that is characterized by bubbling fluidization of large agglomerates of wide size distribution. For the addition of external particles, we chose inert micron-sized sand particles from the Geldart group A classification in view of their superior particulate-type fluidization hydrodynamic behavior. To prevent the hydrodynamics of external particles from dominating that of resident nanoparticles, the proportion of external particles was deliberately kept low. Local bed dynamics was monitored using a sensitive fast-response pressure transducer, and bed expansion was recorded with a video camera. Unlike the previous study [19], where the effect of particle mixing only on the pressure transients was studied, we also carefully examined its influence on other aspects of the bed hydrodynamics including the expansion behavior. Although the mixing of dissimilar particles is known to be invariably associated with a volume change of mixing [30–32], this aspect is yet to be investigated in the context of fluidized bed of nanoparticles. This issue has attracted much attention in the literature particularly for the case of normal packing where the mono-component void fraction hardly exceeds 0.5. However, the bed of nanoparticles used in the present study is substantially different in view of its exceedingly high void fraction (>0.97). From the bed expansion data, we first determined the volume change of mixing; then, we compared our experimental data with well-established packing models to evaluate their efficacy. Since the main objective was to assess the efficacy of particle mixing to improve the fluidization behavior of nanoparticles, we also examined the hysteresis phenomenon by simultaneously conducting both fluidization and de-fluidization studies. Finally, to evaluate the effectiveness of the particle-mixing technique in promoting the de-agglomeration phenomenon, we used pressure-

drop data to compute the hydrodynamic diameter of agglomerates present in the fluidized bed.

## 2. Experimental

The schematic of the experimental set-up is shown in Fig. 1. A transparent 1.5 m-Perspex column of 70 mm internal diameter was used as the test section. Preceding the test section was a 500 mm calming section. A 12 mm-thick perforated plate with 4% fractional open area covered with a nylon mesh was used as a distributor. A fast-response bi-directional differential pressure transducer (Omega PX163-005BD5V) monitored the bed transients. The pressure taps were located at a distance of 110 mm and 230 mm above the distributor in the test section. The voltage signals from pressure transducer were recorded at a rate of 100 data/s using a data acquisition system (National Instruments USB-6289), which was controlled using LabView software on a laptop. Compressed air at ambient conditions was used as fluidizing gas; its flow was measured using two Gilmont flow meters of different ranges.

The nanoparticles used were hydrophilic fumed silica, Aerosil 200, which has a primary size of 12 nm and a surface area of 200  $\text{m}^2/\text{g}$ . Owing to strong interparticle forces, these particles tend to agglomerate. As a result, their dry particle size analysis shows a wide size distribution varying from 2 to 200  $\mu\text{m}$  and a Sauter mean diameter of 20  $\mu\text{m}$  [30]. The tapped bulk density of the Aerosil 200 nanopowder is very low (approximately 50  $\text{kg}/\text{m}^3$ ) as compared to its true solid density of 2200  $\text{kg}/\text{m}^3$ .

Our experimental strategy had two parts. In the first part, unassisted fluidization of nanoparticles was carried out using air superficial velocities of up to 171.5 mm/s, beyond which entrainment and subsequent elutriation of the particles from the bed were observed. To study the hysteresis phenomenon, we also conducted defluidization experiments by gradually decreasing the velocity. For the second part, we added inert sand particles with a size range of 38–75  $\mu\text{m}$  (Sauter mean diameter of 56.5  $\mu\text{m}$ ) and a particle density of 2664  $\text{kg}/\text{m}^3$  to the bed of nanoparticles. As shown in Fig. 2, the characteristics of the additive particle correspond to Geldart group A. The SEM picture of nanoparticles and additive sand particles are shown in Fig. 3. Three different proportions of external additive particles were used. In the first series of experiments, the fraction of the external particles was kept at 2.3 vol% (2.78 wt%) and doubled to 4.5 vol% (5.41 wt%) in the second series. Finally, 8.6 vol% (10.26 wt%) of external particles were used in the third series. Before the particle mixing experiments were started, both particles were loaded together in the column and the mixture was homogenized by the air flow. Once the bed homogeneity was clearly discernible, the air flow was stopped, and the bed was allowed to settle overnight for complete settling. Digital images of the fluidized bed with different amounts of external particles are shown in Fig. 4 (silica nanoparticles are white while the external particles of sand are red). Despite the differences in size and density of the two components, uniform dispersion of the additive particles occurred throughout the bed of nanoparticles.

## 3. Mathematical model

An important aspect of the present work is to investigate the effect of adding external particles on the expansion behavior of the fluidized bed. Owing to the dissimilar sizes of two components, the overall expansion of the bed may not be additive. Therefore, we compute the volume change of mixing for a two-component mixture as follows [31]:

$$\Delta V = V_M - [X_1 V_1 + X_2 V_2], \quad (1)$$

where  $X_i$  is the fluid-free volume fraction of the  $i^{\text{th}}$  component,  $V_M$  is the specific volume of the mixture, and  $V_i$  is the specific volume of the  $i^{\text{th}}$  component. The specific volume is essentially the volume of

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