Fluidization Dynamics of Hydrophobic Nanosilica with Velocity Step Changes

Ebrahim H. Al-Ghurabi, Mohammad Asif, Nadavala Siva Kumar and Sher Afghan Khan

1 Department of Chemical Engineering, King Saud University, P.O. Box 800, Riyadh 11421, Saudi Arabia; ealghurabi@ksu.edu.sa (E.H.A.-G.); snadavala@ksu.edu.sa (N.S.K.)
2 Department of Mechanical Engineering, Faculty of Engineering, IIUM Malaysia, Kuala Lumpur 50728, Malaysia; sakhan@iium.edu.my
* Correspondence: masif@ksu.edu.sa; Tel.: +966-56-981-7045

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Abstract: Nanosilica is widely used in various applications, with its market expected to grow over USD 5 billion by 2025. The fluidized bed technology, owing to its intimate contact and efficient mixing of phases, is ideally suited for the large scale processing of powders. However, the bulk processing and dispersion of ultrafine nanosilica using the fluidized bed technology are critically affected by the interparticle forces, such that the hydrophilic nanosilica shows agglomerate bubbling fluidization (ABF), while the hydrophobic nanosilica undergoes agglomerate particulate fluidization (APF). This study carried out a detailed investigation into the fluidization hydrodynamic of the hydrophobic nanosilica by monitoring the region-wise dynamics of the fluidized bed subjected to a regular step change of fixed duration in the gas velocity. The gas flow was controlled using a mass controller operated with an analog output signal from a data acquisition system. The analog input data were acquired at the sampling rate of 100 Hz and analyzed in both time and temporal frequency domains. The effect of velocity transients on the bed dynamics was quickly mitigated and appeared as lower frequency events, especially in regions away from the distributor. Despite the apparent particulate nature of the fluidization, strong hysteresis was observed in both pressure drop and bed expansion. Moreover, the fully fluidized bed’s pressure drop was less than 75% of the theoretical value even though the bed appeared to free from non-homogeneities. Key fluidization parameters, e.g., minimum fluidization velocity ($U_{mf}$) and the agglomerate size, were evaluated, which can be readily used in the large scale processing of nanosilica powders using fluidized bed technology.

Keywords: fluidization dynamics; hydrophobic nanosilica; hysteresis; hydrodynamics; a velocity step change

1. Introduction

Intimate fluid-solid contact and good interphase mixing are essential to ensure the efficiency of processes that require the interaction of the solid and fluid phases. While small particles inevitably provide high interfacial area, thereby enhancing the surface-based rate processes, their effectiveness could however be compromised due to strong interparticle forces. The role of the interparticle forces has long been recognized in the context of the fluidization of powders [1]. Fine powders, composed of small particles (<20 µm), are mostly cohesive due to interparticle forces, which make their fluidization challenging as well as unpredictable [2,3]. Strong hysteresis effect and severe bed non-homogeneities are observed in their fluidization behavior. Interestingly, nanopowders, despite being composed of ultrafine particles, mainly exhibit two distinct kinds of fluidization behavior, viz.
agglomerate bubbling fluidization (ABF) and agglomerate particulate fluidization (APF) [4,5]. As the name suggests, the constituent nanoparticles of ultrafine powders exist as large agglomerates, which are sometimes as much as a thousandfold larger than the primary size of the individual nanoparticle. Therefore, the fluidization hydrodynamics of ultrafine powders, whether bubbling or particulate, are characterized by the fluidization behavior of their constituent agglomerates.

Nano-silica is extensively used in various applications, e.g., concrete, agriculture, rubber, plastics, electronics, cosmetics, gypsum, battery, pharmaceuticals, and food [6–9]. The estimated global market of nanosilica in 2015 was 3.3 million tons, which is projected to reach over USD 5 billion by 2025 [10]. Depending upon the application requirement, silica nanoparticles’ surface treatment can significantly alter the interparticle force equilibrium, thereby affecting their mutual interaction. For example, hydrophilic nanosilica shows ABF, while its hydrophobic counterpart mostly undergoes APF, even if their primary dimension and density are comparable [5]. The bubbling in ABF limits the bed’s expansion and leads to a size-based stratification of constituent agglomerates along the bed height [11,12].

On the contrary, APF shows a smooth and homogeneous expansion of the bed [13]. The height of the expanded bed can reach several times the height of the original bed. The agglomerates of the hydrophobic nanosilica are morphologically soft and fluffy. The $U_{mf}$ in APF could sometimes be as much as an order of magnitude lower than the one in ABF. As a result, the fluidization hydrodynamics of the ABF have attracted a lot of attention. Efforts were mainly oriented towards understanding ABF hydrodynamics and using assisted fluidization techniques to improve their fluidization behavior [14–20]. On the other hand, any rigorous study on the hydrodynamics of the APF appears to be still lacking the literature.

Therefore, a detailed investigation of the hydrophobic nanosilica undergoing APF has been carried out to thoroughly characterize its hydrodynamics. The information obtained from this study can be readily used during the processing of such nanopowders in large-scale industrial applications, where critical process conditions, such as velocity, could change during the operation. In this study, the nanosilica bed was subjected to velocity step changes (of fixed duration), while the transients in various bed regions were monitored using sensitive pressure transducers located at different heights in the fluidized bed. Analog voltage signals were acquired using an AD converter (18-bit) of a data acquisition system (DAQ). We used the sampling frequency of 100 Hz to ensure accurate monitoring of different events occurring in the fluidized bed. The velocity was first gradually increased during the fluidization cycle, followed by a gradual reduction during the defluidization cycle to delineate the effect of the hysteresis on the APF hydrodynamics. The electronic flow controllers were controlled using the AO signal of the DAQ. The bed transients were analyzed in both time as well as frequency domain.

In addition, the experimental data were processed to examine the influence of the velocity on the mean pressure drop values in the upper, middle, and lower regions of the bed. The overall pressure drop data were used to evaluate the $U_{mf}$, while the overall bed expansion was used to assess the terminal velocity and the agglomerate’s size.

2. Experimental

The schematic of the experimental setup used in the present investigation is shown in Figure 1. A 1.5 m-long 70 mm internal diameter Perspex column was employed as the test section. Below the test section, a 500 mm long column was used as the calming section. A perforated plate distributor covered with fine polymeric mesh separated the two sections. The distributor perforations’ diameter was 2 mm, with a fractional open area of less than 4% [21]. At the top of the test-section, a disengagement section with an internal diameter of 140 mm helped to suppress the solid particle entrainment with the fluidizing gas. A stable supply of compressed air at ambient conditions was used as the fluidizing gas.

For measuring the pressure transients in the fluidized bed, eight sensitive bidirectional pressure transducers were found along with the fluidized bed’s height (Figure 1). The configuration is similar to the one employed before for the pulsed bed of hydrophilic nanosilica [11,12]. The upper region dynamics were recorded using $\Delta P_1$ and $\Delta P_2$, located at the height of 230 mm from the bottom of
The middle region of the bed (i.e., from 110 mm to 230 mm) was monitored using \( \Delta P_3 \) and \( \Delta P_4 \). The dynamics of the lower region of the bed were monitored using \( \Delta P_5 \) and \( \Delta P_6 \). The lower tap of these transducers, i.e., \( \Delta P_5 \) and \( \Delta P_6 \), was at a distance of 11 mm from the distributor plate, while the upper tap was located at a height of 110 mm. Besides, two transducers, \( \Delta P_7 \) and \( \Delta P_8 \), with their lower port connected with the lowest pressure tap, monitored the overall pressure transients. All the pressure taps were covered with a fine polymeric mesh to prevent the bed material from clogging the transducer’s lines. The pressure transducers were calibrated using a Fluke 718 Pressure Calibrator (Everett, WA, USA). An 18-bit, 32-analog input (AI, 625 kS/s), 4-analog output (2.86 MS/s) data acquisition system (USB-6289, National Instruments Corporation, Austin, TX, USA) was used for signal acquisition at a sampling rate of 100 Hz.

![Figure 1. Experimental setup. (1) Calming section; (2) Fluidized bed test section; (3) Disengagement section; (4) Data acquisition system; (5) Analog input signals (red broken lines); (6) Compressed air supply; (7) Mass flow controller; (8) Analog output signals (blue broken lines); (9) Laptop with LabVIEW software.](image)

An electronic mass controller was used to control the fluidizing gas’ flow rate to the test section of the fluidized bed. The velocities were gradually increased in regular steps up to a maximum of 10 mm/s, then decreased similarly to investigate both fluidization and defluidization cycles of the experiment. An analog output (AO) signal from the data acquisition system (DAQ) was used to control
the mass controllers, as shown with a broken blue line in Figure 1, while AI signals from pressure transducers and the flow controller are shown with broken red lines.

A sieved sample of ultrafine fumed nanosilica (Aerosil R812, Evonik GmbH, Wolfgang, Germany) of 7 nm primary size was used as the bed material. It is strongly hydrophobic due to the vapor phase treatment with hexamethyldisilazane (HMDS). Introducing hydrophobicity to the catalytic surface, for example, can significantly alter their surface dynamics [22]. Figure 2a shows the particle size distribution of the sample. Because of agglomeration, the size was found to vary in the range of 0.5 µm to 500 µm, with a median diameter of approximately 8.0 µm. The scanning electron micrograph of the sample, shown in Figure 2b, confirms the particle size distribution analysis results. Overall, 83.2 g of the sieved sample was loaded into the test section. After settling, the bed height was 370 mm, with a bulk density of 58.4 kg/m³.

Figure 2. Size characterization of the sieved sample of hydrophobic nanopowder; (a) Particle size distribution; (b) Agglomerate morphology in SEM micrographs.

The present experimental strategy involved a regular stepwise increase in the velocity, followed by a similar stepwise decrease to investigate the bed hydrodynamics during the fluidization and defluidization cycles (Figure 3). Each step was 150 s long. In addition to the velocity, the overall and local bed dynamics in different bed regions were monitored using eight different pressure transducers, as shown in Figure 1, with the data acquisition frequency of 100 Hz. Thus, there were approximately 15,000 data points for each analog voltage signal at a given velocity. The mean velocity was computed by carefully selecting a data window, as shown in Figure 3, and computing the average value. The mean values of the local and overall pressure drops were similarly computed.

Figure 3. Velocity variation strategy used and evaluation of mean velocities.
Two experimental runs were carried out to verify the data reproducibility. Since the pressure transients were used to characterize the bed dynamics, it was therefore essential to verify the pressure measurement system’s accuracy in this investigation. To this end, the local pressure drop values were added together to obtain the overall pressure drop from the right-hand side of the following equation, which was then compared with the direct monitoring of the overall pressure drop.

\[
\frac{(\Delta P_7 + \Delta P_8)}{2} = \frac{(\Delta P_1 + \Delta P_2)}{2} + \frac{(\Delta P_3 + \Delta P_4)}{2} + \frac{(\Delta P_5 + \Delta P_6)}{2},
\]

The results of the comparison are shown in Figure 4. Clearly, the actual values of the overall pressure drop, i.e., the left-hand side of Equation (1), offer excellent agreement with the sum of the local pressure drops (i.e., the above equation’s right-hand side).

![Figure 4](https://example.com/f4.png)

**Figure 4.** A comparison of the sum of the local pressure drops with the actual overall pressure drop, as represented by Equation (1).

### 3. Results and Discussion

#### 3.1. Overall Bed Transients

Figure 5 shows the overall pressure drop and velocity transients in different fluidized bed regions. The velocity is depicted on the right side while the pressure is represented on the figure’s left side. Initially, the velocity changes were instantaneously reflected on the bed dynamics. The upper region of the bed, represented by \( \Delta P_1 \), was more sensitive to changes to the velocity than the middle and lower regions, i.e., \( \Delta P_3 \) and \( \Delta P_5 \). The step changes in the velocity were initially reflected in the pressure drops. At 600 s, however, when the velocity was increased to 3 mm/s, the dynamics completely changed. There was a steep decline in the pressure drop, indicating a transition from the fixed to fluidized bed contact mode. Further increase in the gas flow above 3 mm/s then led to a different transients’ evolution in different fluidized bed regions. While the upper region showed a relatively consistent increase in the pressure drop, its magnitude in the lower and middle regions hardly increased, despite the velocity increase. This behavior was caused by the velocity-induced bed expansion after the onset of fluidization. Higher velocities caused more significant bed expansion, leading to the migration of bed material from the middle and lower regions to the bed’s upper region. Given that the fluidized bed’s pressure drop corresponds to the effective bed weight, the increase in the solid mass in the upper region was thus reflected in the pressure drop rise. The defluidization experiment started at 1800 s with a stepwise velocity decrease. The upper region transients were slow to respond to the velocity change up to 5.6 mm/s at 2560 s. Further velocity reduction led to a consistent but sharper decrease in the pressure drop in the bed’s upper region. On the contrary, the middle region...
pressure drop ($\Delta P_3$) showed a consistent increase, albeit small, during the defluidization cycle due to lower bed expansion, which caused the reverse migration of the solids from the upper to the middle region of the bed. The pressure drop in the lower region of the bed remained mostly unaffected by the velocity changes.

![Figure 5](image.png)

**Figure 5.** Velocity and pressure transients in different region of the fluidized bed.

Figure 6 highlights the overall pressure drop dynamics (i.e., $\Delta P_7$) and the upper region dynamics monitored by diametrically opposite pressure transducers, $\Delta P_1$, and $\Delta P_2$. At the onset of the fluidization, the overall pressure drop at 600 s showed a steep decline, followed by an increase with the velocity increase. Its value remained relatively steady when the velocity was increased to 8.2 mm/s and then decreased to 4.7 mm/s during the defluidization cycle. A further decrease in the velocity below 4.7 mm/s was clearly reflected in the overall pressure drop profile. On the other hand, the bed dynamics monitored by $\Delta P_1$ and $\Delta P_2$ showed excellent agreement, indicating the absence of radial non-homogeneities in the upper region of the fluidized bed. Similarly, overall pressure drop transients, i.e., $\Delta P_7$ and $\Delta P_8$ (not reported here), showed excellent agreement that can be attributed to the absence of radial non-homogeneities in the APF fluidization of the hydrophobic nanosilica.

![Figure 6](image.png)

**Figure 6.** Velocity, overall pressure drop ($\Delta P_7$), and upper region transients (i.e., $\Delta P_1$ and $\Delta P_2$) for the fluidization and defluidization cycles.

### 3.2. Frequency Response of Bed Transients

In this study, bed transients were also analyzed in the frequency domain. In particular, the effect of the velocity changes on the region-wise bed dynamics was examined. Figure 7a considers
dynamics in the lower bed region. Both x- and y-axes present normalized values. While the amplitude was normalized with the maximum value of the amplitude in the range of interest, the frequency axis was normalized with the frequency of the velocity step changes, which regularly took place at an interval of every 150 s. Thus, velocity changes occurred at a frequency of $6.67 \times 10^{-3}$ Hz. Therefore, the event occurring at the normalized frequency of unity represents the velocity step changes. Higher frequency peaks on the velocity signal appearing at the integral multiple of unity are higher-order sinusoid approximating the velocity step changes. However, the effect of velocity step changes was not strongly felt on $\Delta P_1$, since the peak at 1 (Hz/Hz) was relatively subdued compared to lower frequency peaks occurring at 0.7 and 0.25, as shown in the figure. A typical case of hydrophilic nanosilica with similar experimental conditions is presented in Figure 7b for comparison. There are prominent peaks at 1 (Hz/Hz) in the lower region pressure drop signal. Unlike hydrophobic nanosilica case, however, no low-frequency event was visible in the pressure drop signal. This means that the bed dynamics are more sensitive to velocity transients in ABF than those in APF.

![Figure 7](image)

**Figure 7.** (a) Frequency response of the velocity and lower region pressure drop signals for the hydrophobic nanosilica. (b) Frequency response of the velocity and lower region pressure drop signals for the hydrophilic nanosilica.

Figure 8 shows the dynamics of the upper region. Surprisingly, the 150 s event associated with the velocity change was hardly visible on the frequency response of the pressure drop signal. Rather, two low frequency (i.e., 0.46 and 0.25 Hz/Hz) events were more prominent. This means that the effect of the velocity step changes was mitigated by the time it reached the fluidized bed’s upper region. Instead, it appeared as a low-frequency event since no other event except the velocity changes took place in the system in the given frequency region.

![Figure 8](image)

**Figure 8.** Frequency response of the upper region pressure drop and velocity signals for the fluidized bed of the hydrophobic nanosilica.
3.3. Region-Wise Pressure Drop Behavior

The dependence of the pressure drop on the velocity is shown in Figure 9a for the fluidized bed’s upper region. Results of both cycles, i.e., gradual velocity increase (fluidization) and gradual velocity decrease (defluidization), are presented together in the figure. At low velocities, the velocity increase led to the pressure drop increase due to the fixed bed mode of gas-solid contact. When the velocity was increased above 2 mm/s, there was a significant decline in the pressure drop due to the onset of fluidization that caused motion of solids in the bed. Further increase in the velocity beyond 4 mm/s then led to a rise in the pressure drop due to the bed’s expansion. As mentioned earlier, bed expansion caused the mass (solid materials) upward movement from lower bed sections to the upper region. This phenomenon was reflected in the pressure drop increase. Unlike the fluidization cycle, the velocity decrease led to a consistent pressure drop reduction during the defluidization cycle. As a result, there was a pronounced hysteresis effect observed in the upper region of the bed.

![Figure 9a](image1)

![Figure 9b](image2)

![Figure 9c](image3)

![Figure 9d](image4)

**Figure 9.** Effect of velocity on the pressure drop in different regions of the fluidized bed during the fluidization and defluidization cycles (a) Upper (b) Middle (c) Lower (d) Overall.

Figure 9b shows the velocity dependence of the pressure drop in the middle region of the fluidized bed. The behavior at low velocities, in this case, was similar to the one observed for the upper region owing to the fixed bed mode of gas-solid contact. At the onset of the fluidization, the pressure drop dropped significantly due to the solid motion. As the velocity was raised above 4 mm/s, there was no noticeable change in the pressure drop profile. On the other hand, decreasing the velocity during the defluidization cycle led to a steady increase, albeit small, in the pressure drop behavior, due to the decrease in bed expansion, which caused the downward migration of the bed material upper region of the bed. A significant effect of hysteresis at low velocities was seen in this region. The pressure drop profiles in the lower region are shown in Figure 9c. The hysteresis effect, is at least in this case, compared to the middle and upper regions of the fluidized bed.

The overall pressure drop profiles are presented in Figure 9d. These profiles represent the combined behavior of all the three regions shown before. The upper region behavior dominated
the overall behavior of the fluidized bed. Therefore, the hysteresis phenomenon was prominent in the overall pressure drop profiles as well. Another noteworthy feature in this figure was the overall pressure drop for the fully fluidized bed. In this case, the theoretical pressure drop can be defined as

\[ (\Delta P)_{\text{theoretical}} = (\rho_b - \rho)gL_s \]

where \(\rho_b\) and \(\rho\) are the bulk and fluid densities in kg m\(^{-3}\), respectively. Notably, \(g\) is the acceleration due to gravity, and \(L\) is the height of the bed. Accounting for 83.2 g of the bed material and correcting for the height of the lowest pressure tap from the distributor plate, one obtains \((\Delta P)_{\text{theoretical}} = 201\) Pa. However, the experimental overall pressure drop for the fully fluidized bed obtained in this study was approximately 145 Pa, which was substantially lower than the actual value of 201 Pa, even though no instance of dead region or gas bypassing was observed. This reduction in the pressure drop can be attributed to the upward motion of solids, thereby lowering the upward moving fluidizing gas’s relative velocity. This phenomenon reduced the gas-solid drag, resulting in a lower pressure drop.

3.4. Bed Height and Bulk Density

The dependence of the bed height on the velocity is shown in Figure 10a. The bed height remained stagnant till the velocity was raised to 6 mm/s. Then, there was a steep rise in height as the velocity was increased. On the other hand, the defluidization cycle showed a gradual decrease in height with a reduction in the velocity. Thus, the hysteresis phenomenon was once again very prominent in the expansion of the hydrophobic nanosilica. The overall bed expansion affects the overall bulk density, which has been lately utilized to develop dry beneficiation technologies [23,24]. The bed bulk density profiles are shown in Figure 10b for both fluidization and defluidization cycles. The hysteresis phenomenon is once again very obvious from the figure. However, it is interesting that the bed shows a several-fold increase in the bed height, resulting in the bulk density, which is extremely low and has not been reported in the fluidization of micron-sized particles.

![Figure 10](image-url)  
**Figure 10.** Effect of velocity on the bed expansion (a) Bed height; (b) Bulk density.

3.5. Incipient Fluidization and Terminal Velocity

Figure 11 shows the overall pressure drop, which was normalized by dividing the actual pressure drop by its theoretical value obtained using Equation (2). The experimental data for the defluidization cycle were used for evaluating the \(U_{mf}\), which is approximately 4 mm/s here for the hydrophobic nanosilica, as seen in the figure. On the other hand, the \(U_{mf}\) of hydrophilic nanosilica was approximately 22 mm/s, almost five times higher than the value obtained here [12]. This shows that the surface treatment of the nanosilica can significantly affect its dry dispersion characteristics. Moreover, given that the \(U_{mf}\) depends on the particle size, a lower \(U_{mf}\) indicated smaller agglomerates in the present case. Besides the minimum fluidization velocity, there was another aspect of the fluidization hydrodynamic, which was noteworthy as well. The normalized pressure drop for the
fully fluidized bed was 0.72. This means that the actual pressure drop was significantly lower than
the theoretical pressure drop, despite the particulate nature of the fluidization, which rules out the
occurrence of gas by-passing and other similar non-homogeneities in the bed.

\[ U_t/n = U_t^{1/n} - U_t^{1/n}(1 - \varepsilon_{g0})H_0/H \]  

(3)

where \( U_t \) is the terminal velocity of the agglomerate, \( H_0 \) and \( H \) are initial bed height and the bed
height at velocity \( U_0 \), respectively. \( \varepsilon_{g0} \) is the inter-agglomerate bed void fraction at the initial bed
height conditions, and \( n \) is the Richardson–Zaki exponent. The above equation describes the linear
relationship between the \( U_t^{1/n} \) (y-axis) and the \( H_0/H \) (x-axis), thereby yielding \( U_t^{1/n} \) as the y-intercept
and \( U_t^{1/n}(1 - \varepsilon_{g0}) \) as the slope. The results are shown in Figure 12 using \( n = 5 \). Previous studies pointed
out that the above equation’s parameters were relatively independent of the value of \( n \) [5]. From the
figure, the terminal velocity of the hydrophobic nanosilica was found to be 21.6 mm/s. Using the Stokes
law and the terminal velocity data, the values of the agglomerate diameter and the terminal Reynolds
number was found to be 87 µm, and 0.12, respectively.

\[ \text{Figure 11. Minimum fluidization velocity from the overall pressure drop profile of the defluidization cycle.} \]

\[ \text{Figure 12. Evaluation of the terminal velocity from the dependence of the bed expansion on the gas velocity.} \]
4. Conclusions

The fluidized bed dynamics of agglomerated hydrophobic nanosilica with APF behavior were investigated here by monitoring the pressure transients in different regions of the bed, which was subjected to regular step changes in the velocity of the fluidizing gas. The radial non-uniformities were mostly absent in the bed during both the fluidization and defluidization cycles. The effect of the velocity changes was observed on the lower region dynamics. In the upper region of the bed, however, the effect of the velocity changes was substantially mitigated and mostly appeared as low-frequency events. Strong hysteresis effects on the pressure drop profiles were noted in the upper region, which was also clearly reflected in the bed’s overall behavior. The hysteresis phenomenon was also found in the overall bed expansion and the bulk density profiles. Although neither gas by-passing nor other non-homogeneities were observed in the bed, the actual pressure drop in the fully fluidized bed was significantly lower than the theoretical pressure drop obtained from the bed’s total mass. The minimum fluidization velocity was substantially lower than the ones usually obtained for the hydrophilic nanosilica. Similarly, the agglomerate diameter evaluated from the bed expansion data, when fitted with the Richardson–Zaki correlation, predicted the agglomerate size to be 86 mm while the actual primary nanoparticle size was reportedly 7 nm only.

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