Nanopowder Fluidization Using the Combined Assisted Fluidization Techniques of Particle Mixing and Flow Pulsation

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Abstract: In the present study, we report the fluidization behavior of ultrafine nanopowder using the assisted fluidization technique of particle mixing, which was further superimposed with the pulsation of the inlet gas flow to the fluidized bed. The powder selected in the present study was hydrophilic nanosilica, which shows strong agglomeration behavior leading to poor fluidization hydrodynamics. For particle mixing, small proportions of inert particles of Geldart group A classification were used. The inlet gas flow to the fluidized bed was pulsed with a square wave of frequency 0.1 Hz with the help of a solenoid valve controlled using the data acquisition system (DAQ). In addition to the gas flow rate to the fluidized bed, pressure transients were carefully monitored using sensitive pressure transducers connected to the DAQ. Our results indicate a substantial reduction in the effective agglomerate size as a result of the simultaneous implementation of the assisted fluidization techniques of particle mixing and flow pulsation.

Keywords: Assisted fluidization; particle mixing; flow pulsation; agglomeration; hysteresis

1. Introduction

Process industries often employ fine and ultrafine powders to enhance surface-based rate processes such as gas–solid-catalyzed reactions and various separation processes [1,2]. An intimate contact between the solid and the fluid phase is an important prerequisite for their efficient utilization. Ultrafine nanopowders, whose size typically ranges from 1 to 100 nm, have a wide range of applications, from in research laboratories to large-scale industrial applications. Their extremely high surface area-to-volume ratio can significantly enhance the processes that depend upon the availability of the surface area. However, the efficacy of nanopowders is often compromised due to their agglomeration behavior, which occurs as a result of strong interparticle van der Waals forces [3–7].

Fluidized beds enhance the interphase mixing, and impart high mass and heat transfer rates while limiting the pressure drop to effective bed weight, even at high fluid velocities. Their hydrodynamics, however, strongly depend on the physical characteristics of the solid particles such as particle diameter and density [1,8,9]. Classified as Geldart group C particles (\(D_p < 30 \mu m\)), nanopowders usually show strong agglomeration behavior, which induces non-homogeneities in the bed during their fluidization. This leads to poor interphase mixing, thereby affecting the effectiveness of the fluidized bed [10–13].

Assisted fluidization techniques are often used to improve the fluidization behavior by providing additional energy to overcome the interparticle forces and promote deagglomeration [14]. Such techniques sometimes involve internal or external vibrations of the fluidized beds using various
devices and configurations, such as ultrasonic comminution devices [15], high-shear mixers [16], mechanical vibrations [17], acoustic vibrations [18–20], and oscillating magnetic particles under a magnetic field [21,22]. In addition to being energy intensive, these techniques are also difficult to implement on an industrial scale. Some assisted fluidization techniques, which do not require a direct supply of energy, include tapered and inclined fluidized beds [23,24], fluid flow pulsation [8,25–29], and inert particle mixing [3,4,30–34]. In some cases, a combination of two assisted fluidization techniques has also been used to further enhance the performance of fluidized beds [35–38]. Levy and Celeste [6] studied the hydrodynamics of fine powders with sizes ranging from 12 nm to 15 µm in bubbling fluidized beds. They used the combined assisted techniques of horizontal mechanical vibrations and acoustic vibrations to enhance the fluidization behavior. The combined technique was found to be more effective than mechanical vibrations alone. Both the agglomerate diameter and the minimum fluidization velocities were substantially reduced by the application of combined assisted fluidization techniques. Liu and Guo [39] studied the effect of acoustic and magnetic vibrations on the hydrodynamics of fluidized beds containing fine powders ranging from 5 nm to 45 µm. The combination of assisted techniques helped to reduce the minimum fluidization velocity to a greater extent as compared to the case when these techniques were used separately. In the present study, a combination of two assisted fluidization techniques, namely, particle mixing and flow pulsation, is used to improve the fluidization behavior of a bed of nanopowder.

In the assisted fluidization technique of particle mixing, the premixing of external particles with nanoparticles improves the bed homogeneity and promotes deagglomeration, and thus enhances the fluidization quality. Ali and Asif [30] reported a significant improvement in the fluidized bed hydrodynamics by the addition of even a small proportion of inert Geldart group A particles. The hysteresis effect, which occurs due to bed non-homogeneity, was suppressed, and a substantial reduction in the size of agglomerates was observed. Moreover, a substantial bed contraction occurred during the defluidization owing mainly to increased bed homogeneity. Duan et al. [33] used a binary solid fluidized bed of SiO₂ and ZnO nanoparticles, and added FCC and Al₂O₃ of size range 38–1000 µm in proportions varying from 15 to 45 wt %. This strategy helped to improve the fluidization behavior. The minimum fluidization velocity decreased with the increase in the fraction of external particles in the bed. The size of the inert particles played a crucial role in fluidized bed hydrodynamics.

In the assisted fluidization technique of flow pulsation, the inlet flow to the fluidized bed is pulsed. Akhavan et al. [8] used pulsed fluid flow for silica nanoparticles by using two flow inlets. The fluid flow was kept steady in one input while flow pulsation was applied to the other input. This configuration helped to maintain minimum fluid flow to the bed, even when the valve was closed in the other input during pulsation. The fluidization behavior improved remarkably, leading to as much as a 72% reduction in the minimum fluidization velocity. Ali and Asif [27] used pulsed flow at different frequencies of 0.25, 0.1, and 0.05 Hz. They reported that fluidization quality, in terms of bed homogeneity, hysteresis, and minimum fluidization velocity, was significantly enhanced. Also, the pulsed airflow reduced the agglomerate size by 50%.

In the present work, we have investigated the fluidization hydrodynamics of a fluidized bed of hydrophilic nanopowder that was subjected to unassisted fluidization, assisted fluidization of particle mixing, and a combination of both assisted fluidization techniques of particle mixing and flow pulsation. In addition to the bed expansion data, the pressure transients during the experiments were recorded using highly sensitive pressure transducers at a rate of 100 data/s. The experimental data were then used to compute the minimum fluidization velocity and the agglomerate diameter.

2. Experimental

We used hydrophilic fumed SiO₂ (Aerosil 200; Evonik Industries, Essen, Germany) in our experiments. Its primary size was 12 nm and it had a surface area of 200 ± 25 m²/g. However, due to agglomeration, dry-particle size analysis yielded a wide size distribution, ranging from 2 to 100 µm with a Sauter mean diameter of 20 µm [30]. Though its true density was 2200 kg/m³, the
tapped density was found to be only 50 kg/m$^3$, which indicated the highly porous nature of the bed with a void fraction of 0.98. For mixing, we used inert Geldart group A particles (sand) with a size range of 38–75 µm (Sauter mean diameter = 56.5 µm) and a density of 2664 kg/m$^3$. Compressed air, at room temperature, was used as fluidizing medium in the experiments.

The experimental setup (Figure 1) consisted of a test section, which was a transparent Perspex column of height 1.5 m and internal diameter 0.07 m. It was preceded by a 0.50 m-long calming section. We used a nylon mesh-covered perforated distributor with a 4% fractional open area to ensure uniform airflow in the test section.

![Figure 1. Schematic of the experimental setup. DAQ: data acquisition system.](image)

The pressure transients of the nanoparticle bed were recorded using a highly sensitive fast-response bidirectional differential pressure transducer (Omega PX163-005BD5V; Norwalk, CT, USA) between pressure taps located at distances of 110 and 230 mm above the distributor. A data acquisition system (DAQ) with LabVIEW software (National Instruments, Austin, Texas, USA) was used to record pressure transients at a rate of 100 data/s from the pressure transducers' voltage signals.

The inlet air flow to the fluidized bed was pulsed at a frequency of 0.1 Hz using a solenoid valve (Omega SV 3310; Norwalk, CT, USA). The valve was kept open for 5 s followed by 5 s of closing time, thus completing a cycle of 0.1 Hz. As shown in Figure 2, the 0.1 Hz pulsation allowed complete settling of the bed once the flow was stopped and, moreover, led to complete fluidization when the solenoid valve was open during the flow pulsation [26,40]. The solenoid valve was controlled using a digital I/O signal from the data acquisition system (DAQ) and LabVIEW software.
The following experimental strategy was followed in the present work:

1. Unassisted fluidization experiments were carried out by first gradually increasing the velocity followed by a gradual defluidization.
2. Before carrying out the fluidization and defluidization experiment, 4.5 vol % Geldart group A particles were added and thoroughly mixed with the nanopowder.
3. The inlet air flow to the fluidized bed was pulsed at 0.1 Hz.
4. Steps 2 and 3 were repeated after increasing the fraction of group A particles to 8.6 vol %.

Two Gilmont flow meters of different ranges were used to control the airflow to the fluidized bed. The superficial velocity was gradually increased up to a maximum of 171.5 mm/s to avoid the elutriation of nanoparticles at higher velocities.

3. Mathematical Equations Used

The average pressure drop and standard deviation were calculated from the pressure drop data using the following equations:

\[
\Delta P = \frac{1}{n} \sum_{i=1}^{n} (\Delta P_i), \quad (1)
\]

\[
\sigma = \sqrt{\frac{1}{n-1} \sum_{i=1}^{n} (\Delta P_i - \Delta P)^2}, \quad (2)
\]

The pressure drop in the packed bed is described by the well-known Ergun equation:

\[
\frac{\Delta P}{\Delta L} = \frac{150u U_0}{D_{av}^2} \frac{(1 - \varepsilon)^2}{\varepsilon^3} + \frac{1.75 \mu (U_0)^2}{D_{av} \varepsilon^3} (1 - \varepsilon), \quad (3)
\]

where \(\varepsilon\) is the bed void fraction, \(U_0\) is the fluid superficial velocity, \(\mu\) is the viscosity, and \(D_{av}\) is the average diameter of the agglomerates of nanoparticles.
Since the fluidized bed pressure drop is equal to the effective weight of the solid in the bed, one can, therefore, write
\[
\frac{\Delta P}{\Delta L} = \left( \rho_b - \rho_f \right) g = \left( 1 - \epsilon \right) \left( \rho_p - \rho_f \right) g.
\] (4)
where \(\rho_p\), \(\rho_f\), and \(\rho_b\) are true solid density, fluid density, and bulk density, respectively.

\[
\epsilon = 1 - \frac{M_{\text{bed}}}{\rho_p V_{\text{bed}}}.
\] (5)
Here, \(M_{\text{bed}}\) is the total solid mass in the bed while \(V_{\text{bed}}\) is the volume of the solids in the bed.

\[
\rho_b = (1 - \epsilon) \rho_p + \epsilon \rho_f.
\] (6)

When the fluidized bed contains a binary mixture of different-sized particles, the average diameter of the particles can be written as
\[
\frac{1}{D_{av}} = X_1/D_1 + X_2/D_2.
\] (7)
where \(D_i\) is the diameter of the \(i^{th}\) component and \(X_i\) is the fluid-free volume fraction of the \(i^{th}\) component.

4. Results and Discussion
The dependence of the pressure drop on the superficial velocity is reported in Figure 3. The ordinate in the figure is the normalized pressure drop, which was computed by dividing the pressure drop by the effective weight of the bed. During the unassisted fluidization, a significant hysteresis is visible between the fluidization and defluidization. This is caused by initial non-homogeneities due to the presence of cracks, channels, and plugs, which leads to non-uniform flow of air through the bed. The bed is more homogenous during the defluidization. The difference in bed homogeneity usually results in hysteresis as discussed in detail by Gomez-Hernandez et al. [41].

The addition of even a small proportion of Geldart group A particles helps to mitigate the effect of hysteresis, which is further diminished when the flow is pulsed, as seen in Figure 4. The fluidization behavior is clearly further improved with the combination of assisted fluidization techniques of flow pulsation and particle mixing.

![Figure 3. Effect of velocity and particle mixing on the pressure drop behavior of the fluidized bed.](image-url)
Figure 3. Effect of velocity and particle mixing on the pressure drop behavior of the fluidized bed. 

(a) 

(b) 

Figure 4. Effect of velocity on the pressure drop behavior of the fluidized bed subjected to the combined effect of flow pulsation and particle mixing; (a) 4.5 vol % particle mixing; and (b) 8.6 vol % particle mixing.

Another interesting feature of Figure 3 is that the particle mixing causes a higher pressure drop at low velocities when the bed is not fluidized. The higher the fraction of external particles, the greater the pressure drop, because the addition of external particles lowers the bed void fraction due to the volume-contraction phenomenon, which is reflected in the increase of the pressure drop in the bed [28]. In fact, it was pointed out that a decrease in bed expansion during long-term fluidization of hydrophilic nano-titania at high velocities cannot be attributed to a reduction in the size of agglomerates [42]. Rather, it appears that since high velocities promote greater solid mixing in the bed, smaller agglomerates tend to occupy the interstitial spaces of their large counterparts, thus contributing to the bed volume contraction. The effect of the flow pulsation on the pressure drop profile is more pronounced for a fluidized bed containing a small amount of external particles (Figure 4a). At higher proportions of external particles, the pressure drop behavior is not affected so much by the flow pulsation (Figure 4b).

It is clear from the pressure drop profiles shown in Figure 4 that hysteresis is present even with particle mixing, especially when higher amounts of external particles are added. The use of
flow pulsation, however, eliminates the hysteresis, which is a clear indication of the removal of bed non-homogeneities.

The minimum fluidization velocity \( (U_{mf}) \) is an important parameter that characterizes the hydrodynamics of the fluidized bed. At incipient fluidization, the packed pressure drop is equal to the effective bed weight. Using this approach, we determined the minimum fluidization velocity as shown in Figure 5. The results are reported in Table 1. The void fraction at incipient fluidization was computed from the bed expansion data. Clearly, particle mixing lowered the \( U_{mf} \) by more than one-half of the corresponding value obtained using unassisted fluidization. When flow pulsation was introduced, a further substantial reduction in the \( U_{mf} \) was seen. Thus, the use of the combined assisted fluidization techniques of particle mixing and flow pulsation helped to reduce the \( U_{mf} \) from 118 to 25 mm/s, which is, indeed, a significant reduction.

![Figure 5. Minimum fluidization calculations for nanoparticle bed fluidization at different assistance conditions.](image)

**Figure 5.** Minimum fluidization calculations for nanoparticle bed fluidization at different assistance conditions.

### Table 1. Incipient fluidization parameters of the fluidized bed.

| Incipient Fluidization          | \( U_{mf} \) (mm/s) | \( \varepsilon_{mf} \) (/-) |
|---------------------------------|----------------------|-----------------------------|
| Unassisted                     | 118.3                | 0.984                       |
| 4.5 vol % particle              | 47.0                 | 0.973                       |
| 4.5 vol % + 0.1 Hz pulse        | 25.0                 | 0.971                       |
| 8.6 vol % particle              | 49.4                 | 0.970                       |
| 8.6 vol % + 0.1 Hz pulse        | 33.0                 | 0.969                       |

However, when the fraction of external particles was increased to 8.6 vol %, no reduction in the \( U_{mf} \) was noticed as compared to the case of 4.5 vol %. Instead, there was an increase, however small. Introducing flow pulsation to the mixed fluidized bed, nonetheless, once again significantly lowered the \( U_{mf} \) to 33 mm/s, which is, however, higher than the 25 mm/s achieved in the case of 4.6 vol %.

We further analyzed our pressure drop data using the Ergun equation (Equation (4)), which is valid for velocities in the packed bed region when the bed is not fluidized. According to the Ergun equation, the pressure drop \( (\Delta P) \) depends on the bed void fraction \( (\varepsilon) \) and agglomerate diameter \( (D_{av}) \). The bed void fraction \( (\varepsilon) \) was computed from the bed height using Equation (5). As seen in Table 2, these values are lower than the corresponding incipient values \( (\varepsilon_{mf}) \) reported in Table 1. This leaves diameter as the only remaining unknown parameter in the Ergun equation. As shown in Figure 6, we fitted our experimental data with the Ergun equation in order to determine the effective
diameter of the agglomerates of nanoparticles present in the bed. The results obtained using regression analysis are reported in Table 2. The \( R^2 \) values in most cases indicated an excellent fit with the experimental data (more than 90% in all cases). The effective diameter in the table is the average diameter for both nanoparticles as well as external particles, while the mean agglomerate diameter is the average diameter of agglomerates only, which was calculated using Equation (9). Clearly, particle mixing helped to decrease the size of nanoparticles agglomerates in the packed bed. The agglomerate diameter decreased by 33.2% and 48.4% with the addition of 4.5 vol % and 8.6 vol % group A particles, respectively. The agglomerate diameter further reduced to 39.6% with the combined effect of 4.5 vol % particle mixing and pulsed airflow as compared to the case of particle mixing alone. However, for the case of 8.6 vol % particle mixing and flow pulsation together, the agglomerate size was further reduced to 50.7%. This reduction is not as significant as the one seen in the case of 4.5 vol % when flow pulsation was applied.

Table 2. Agglomerate size reduction under different fluidization conditions (external particle diameter: 56.5 \( \mu \)m).

| Assisted Fluidization Techniques | Porosity (\( \varepsilon \)) | Effective Diameter | Mean Agglomerate Diameter | Agglomerate Size Reduction | \( R^2 \) |
|--------------------------------|-----------------------------|--------------------|---------------------------|---------------------------|----------|
| Unassisted                     | 0.979                       | 14.05              | 14.05                     | -                         | 0.997    |
| 4.5 vol % particle             | 0.971                       | 11.62              | 9.50                      | 32.4                      | 0.910    |
| 4.5 vol % + 0.1 Hz pulse       | 0.970                       | 10.65              | 8.49                      | 39.6                      | 0.992    |
| 8.6 vol % particle             | 0.967                       | 11.50              | 7.25                      | 48.4                      | 0.924    |
| 8.6 vol % + 0.1 Hz pulse       | 0.969                       | 11.21              | 6.93                      | 50.7                      | 0.975    |

Figure 6. Nanoparticle agglomerate diameter calculation by equation fitting on average pressure drop results.

These results indicate that the use of flow pulsation with particle mixing further enhanced the deagglomeration phenomenon. However, the effect of pulsed flow is more prominent in 4.5 vol % particle mixing than in 8.6 vol % particle mixing. The size reduction can be attributed to the presence of larger micron-sized Geldart group A particles in the bed of nanoparticles. The repeated collision of denser and larger external particles tends to fragment agglomerates of nanoparticles. Moreover, the interagglomerate force equilibrium is disturbed, owing to the presence of different external particles in their midst, which leads to deagglomeration.
5. Conclusions

The hysteresis effect is reduced by the assisted fluidization technique of particle mixing and almost completely eliminated by the combined application of particle mixing and flow pulsation. Since hysteresis mainly arises due to non-homogeneities present in the bed, it is obvious that flow pulsation in conjunction with particle mixing is quite effective in improving the fluidization hydrodynamics by eliminating various kinds of bed non-homogeneities.

Though the assisted fluidization technique of particle mixing was effective in lowering the $U_{mf}$, better results were obtained with 4.5 vol % as compared to the case of 8.6 vol %. When the flow was also pulsed for mixed beds, a further substantial reduction was obtained in the value of the $U_{mf}$. This reduction was, however, higher in the case of 4.5 vol %.

Particle mixing using Geldart group A promoted deagglomeration behavior in the fluidized bed of nanopowder. As much as a 48% size reduction was achieved with 8.6 vol % particle mixing. The assisted fluidization technique of flow pulsation was more effective for 4.5 vol % as compared to the case of 8.6 vol %.

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Nomenclature

| Symbol  | Description                                      | Units   |
|---------|--------------------------------------------------|---------|
| $D_{av}$ | mean diameter of nanoparticle agglomerates (μm) |         |
| $D_i$   | diameter of the $i^{th}$ component (μm)         |         |
| $\Delta L$ | distance between pressure taps (m)             |         |
| $M_{bed}$ | solid mass in the bed (kg)                     |         |
| $\Delta P$ | pressure drop calculated from the Ergun equation (Pa) |     |
| $\overline{\Delta P}$ | average pressure drop (Pa)                   |         |
| $\Delta P_i$ | pressure transient recorded at $i^{th}$ time (Pa) |     |
| $U_0$   | superficial velocity of the fluid (m/s)        |         |
| $U_{mf}$ | minimum fluidization velocity (m/s)            |         |
| $V_{bed}$ | volume of the solids in the bed (m³)            |         |
| $V_i$   | specific volume of the $i^{th}$ species (-/-)  |         |
| $V_M$   | specific volume of the mixture (-/-)           |         |
| $X_i$   | volume fraction of the $i^{th}$ species (-/-)  |         |

Greek Symbols

| Symbol | Description            | Units   |
|--------|------------------------|---------|
| $\varepsilon$ | bed porosity (-/-) |         |
| $\varepsilon_{mf}$ | bed porosity at incipient fluidization (-/-) |     |
| $\rho_b$ | bulk density (kg/m³)  |         |
| $\rho_f$ | air density (kg/m³)   |         |
| $\rho_p$ | true solid density (kg/m³) |     |
| $u$    | air viscosity at room temperature (N·s·m⁻²) |         |
| $\sigma$ | standard deviation (Pa) |         |

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