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# Fluidization of nano and sub-micron powders using mechanical vibration

Souresh Kaliyaperumal, Shahzad Barghi, Lauren Briens, Sohrab Rohani, Jesse Zhu*

*Powder Technology Research Center, Department of Chemical & Biochemical Engineering, The University of Western Ontario, London, Ontario, Canada, N6A 5B9*

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ABSTRACT

The fluidization behavior of nano and sub-micron powders belonging to group C of Geldart's classification was studied in a mechanically vibrated fluidized bed (vibro-fluidized bed) at room temperature. Pretreated air was used as the fluidizing gas whereas $SiO_2$, $Al_2O_3$, $TiO_2$, ZrSi, $BaSO_4$ were solid particles. Mechanical vibration amplitudes were 0.1, 0.25, 0.35, 0.45 mm, while the frequencies were 5, 20, 30, 40 Hz to investigate the effects of frequency and amplitude of mechanical vibration on minimum fluidization velocity, bed pressure drop, bed expansion, and the agglomerate size and size distribution. A novel technique was employed to determine the apparent minimum fluidization velocity from pressure drop signals. Richardson–Zaki equation was employed as nano-particles showed fluid like behavior when fluidized. The average size of agglomerates formed on top of the bed was smaller than those at the bottom. Size distribution of agglomerates on top was also more uniform compared to those near the distributor. Larger agglomerates at the bottom of the bed formed a small fraction of the bed particles. Average size of submicron agglomerates decreased with increasing the frequency of vibration, however nano particles were less sensitive to change in vibration frequency. Mechanical vibration enhanced the quality of fluidization by reducing channeling and rat-holing phenomena caused by interparticle cohesive forces.


## 1. Introduction

Fine and ultrafine (nano) particles are being widely used in many industries due to their attractive physical and chemical properties. Approximately 30% of the chemicals produced in the world are in the form of powder (Krupp & Sperling, 1966). The high surface area of fine and nano powders, which results in their enhanced activity enables them to meet the process requirements of many products, including pharmaceuticals, chemicals, foods, paints, dyes, inks, ceramics, cosmetics, powder metallurgy and powder coating. Recently, very fine and pure powders with a narrow size distribution have helped to make advanced materials for the aerospace, transportation, electronics and health care industries (Wang, Rahman, & Rhodes, 2007).

Fluidization is a widely used technique for powder handling in various industries because of a favorable gas–solid contact efficiency. Among the well known Geldart's groups (Geldart, 1973) in gas–solid fluidization (based on the size and density difference between gas and solid particles), group C powders are very fine and hard to fluidize due to strong interparticle cohesive forces between them. The cohesive nature of group C powders comes from the fact that when the particle size becomes smaller, the relative magnitude of the inter-particle forces increases. Such strong inter-particle forces make the individual particles stick together and form agglomerates that can lead to severe agglomeration, channeling, and rat holing or even complete defluidization. Micron and submicron particles belong to group C powders and remarkable reports have been published about fluidization behavior of these powders (Chaouki, Chavarie, Klvana, & Pajonk, 1985; Chavarie, Dobson, Clift, & Seville, 1987; Li, Legros, Brereton, Grace, & Chaouki, 1990; Morooka, Kusakabe, Kobata, & Kato, 1988; Pacek & Niwnow, 1990). It was shown that nonuniform fluidization of these powders was related to the existing agglomerates or their formation during fluidization. Due to the increasing applications of ultrafine powders and nanoparticles, which are at the extreme end of Geldart's group C particles, fluidization of these particles has been the object of many researchers (Guo, Liu, Shen, Yan, & Jia, 2006; Guo, Yang, Shen, & Liu, 2007; Quevedo et al., 2006; Valverde & Castellanos, 2007; Valverde, Quintanilla, Castellanos, Lepek, & Quevedo, 2008; Wang, Palero, Soria, & Rhodes, 2006a, 2006b; Wang et al., 2007) in recent years. Nanoparticles with low bulk density generally form porous agglomerates showing fluid-like fluidization with no bubbles and thus termed as Agglomerate Particulate Fluidization, APF (Valverde & Castellanos, 2007). Heterogeneous bubbling fluidization was observed for dense nanoparticles at high gas velocities (Zhu, Yu, Dave, & Pfeffer, 2005).

* Corresponding author.
*E-mail address:* jzhu@uwo.ca (J. Zhu).

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**Nomenclature**

| | |
|---|---|
| $A$ | cross sectional area, m$^2$ |
| $A_m$ | mechanical vibration amplitude, m |
| $A_1$ | minimum of $y$-axis data in Eq. (6) |
| $A_2$ | maximum of $y$-axis data in Eq. (6) |
| $C_D$ | drag coefficient |
| $d_a$ | particle diameter, m |
| $d_{a,ave}$ | average size of agglomerates in the bed, m |
| $d_{a,t}$ | average size of agglomerates on top of the bed, m |
| $d_{a,b}$ | average size of agglomerates at the bottom of the bed, m |
| $d_p$ | particle diameter, μm |
| F.I. | fluidization index |
| $f$ | frequency of mechanical vibration, Hz |
| $g$ | acceleration due to gravity, m/s$^2$ |
| $H$ | height of the bed, m |
| $H_0$ | static bed height, m |
| $n$ | exponent in Eq. (4) |
| $P$ | hill slope in Eq. (6) |
| $\Delta P$ | pressure drop across the bed, Pa |
| $U_g$ | superficial gas velocity, m/s |
| $U_{mf}$ | minimum fluidization velocity, m/s |
| $U_t$ | terminal velocity, m/s |
| $W$ | weight of the solids, N |
| $X$ | $x$-axis data |
| $X_0$ | $X$ at $y$ 50% of $xy$ data |
| $Y$ | fluidization index in Eq. (6) |

*Greek letters*

| | |
|---|---|
| $\rho_b$ | bed density, aerated, kg/m$^3$ |
| $\rho_f$ | fluid density, kg/m$^3$ |
| $\rho_p$ | particle density, kg/m$^3$ |
| $\Gamma$ | mechanical vibration strength ratio |
| $\varepsilon$ | bed voidage |

In order to improve the fluidization quality of fine/ultrafine powders, various fluidization aids have been proposed such as mechanical vibration (Dutta & Dullea, 1991; Mori, Yamamoto, Wata, Harahan, & Yamada, 1990), sound wave vibration (Chirine & Massimilla, 1994; Nowak, Hasatani, & Derczynski, 1993), mechanical stirring (Brekken, Lancaster, & Wheellock, 1970), magnetic field disturbance (Zhu & Li, 1996) and addition of fine particles as flow conditioners (Zhou & Li, 1999). Application of fluidization aids reduces the gas velocity well below the minimum fluidization velocity of the particles for smooth fluidization. Valverde et al. (2008) used different gases as fluidizing gas for nanoparticles and showed that the gas type had negligible effect on the size of agglomerates. They also showed that nanoparticles form fine agglomerates, which have a tendency to further agglomerate and make larger ones when fluidized.

A number of research groups have arbitrarily defined the boundary between submicron and nano powders using different particle sizes. In this study, particles between 50 nm and 1 μm in size are considered as sub-micron powder and smaller particles (<50 nm) as nanoparticles. Morooka et al. (1988) reported that even some sub-micron particles could be fluidized fairly, since these particles agglomerated into larger particles and thus fluidized at high gas velocities. Mori et al. (1990), using a vibro-fluidized bed, also found that a wide range of fine particles down to sub-micron level could be fluidized fairly well at relatively low gas velocities. Dutta and Dullea (1991) used external vibration to improve the fluidization quality of fine cohesive powders, which simultaneously increased the bed pressure drop and the bed expansion, and decreased elutriation loss. Jung and Gidaspow (2002) reported that nano particles could be fluidized due to the formation of light agglomerates. Wensrich and Collard (2006) studied the resonant behavior of vibro fluidized beds providing more insight to the harmonic response of various granular materials. Alavi and Caussat (2005) showed the effect of vibration strengths on fluidization of micronic powders in a cylindrical/conical fluidized bed. Barletta, Donsi, Ferrari, Poletto, and Russo (2008) studied the effect of mechanical vibration on the fluidization of aeratable powders.

Due to the strong interparticle forces between submicron/nano particles, agglomeration is a common phenomenon observed in the fluidization of such powders (Valverde & Castellanos, 2007). The size of agglomerates is larger than primary particles and therefore the physical properties of primary particles cannot be used for the prediction of fluidization behavior of these agglomerates. Depending on the type of particles, they may be self agglomerated forming stable and roughly mono-sized agglomerates (Chaouki et al., 1985; Jaraize, Kimura, & Levenspiel, 1992). In general, formation of fluidized agglomerates is the results of a dynamic equilibrium between formation and breakage of the agglomerates in the fluidized bed. If the interparticle forces are weak, the agglomerates would be fragile, making the sampling of these agglomerates a challenge. On the other hand, agglomeration also depends on the type of the fluidizing gas, its humidity and velocity.

Most of the studies on mechanical vibration have been focused widely on micron-sized and nanoparticles. Very few results have been reported on the fluidization of submicron powders. The objective of this study is to improve the fluidization quality of nano and sub-micron powders by reducing the interparticle forces using external mechanical vibration. In this study, the effects of vibration on the fluidization of nano and sub-micron powders with respect to minimum fluidization velocity, bed pressure drop, bed expansion, and size of agglomerates were investigated. In addition, a novel technique (curve fitting) was used to find the apparent minimum fluidization velocity of submicron and nano powders. The effect of amplitude and the frequency of mechanical vibration were also investigated.

## 2. Experimental setup and methods

The schematic diagram of the experimental setup is shown in Fig. 1. Compressed air, stripped off humidity through a fixed bed of silica gel to ensure a low and constant humidity, was used as the fluidizing gas. The gas flow rate was measured and controlled by a series of calibrated rotameters (Omega Eng. Inc.) and a digital mass flow controller (Fathom Technologies, GR series). The particles used in the experiments and their properties are shown in Table 1. The fluidized bed is a Plexiglas column, 10 cm I.D. and 30 cm high. A porous polymer plate was used as the gas distributor. A differential pressure transducer (Omega PX163) measured the bed pressure drop and the bed expansion was determined visually.

The fluidized bed and its wind box are fixed onto the upper surface of a vibro-stand, which is supported at its base by four springs. Mechanical vibration was generated with a pair of vibrators (LYNN Co. Ltd., BL-03, 60 W), fixed to two opposite sides of the vibro-stand. A variable frequency drive precisely controlled the vibration frequency between 0 and 50 Hz. The amplitude of the vibration was varied between 0 and 0.5 mm by changing the eccentric weight on the vibro-motors and was measured by a high-sensitivity accelerometer (Dalimar Instruments Inc.).

Experiments were carried out at amplitudes of 0.1, 0.25, 0.35, 0.45 mm, and frequencies of 5, 20, 30, 40 Hz. The bed height was