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## **Powder Technology**

journal homepage: www.elsevier.com/locate/powtec



## Effects of acoustic vibration on nano and sub-micron powders fluidization

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#### ARTICLE INFO

Article history: Received 16 March 2010 Received in revised form 9 February 2011 Accepted 13 March 2011 Available online 21 March 2011

Keywords:
Fluidization
Nano-particles
Sub-micron particles
Acoustic vibration
Minimum fluidization velocity

#### ABSTRACT

Fluidization of nano and sub-micron powders with and without acoustic vibration was investigated. The effects of sound pressure level and frequency were studied. Loudspeakers located under the distributor plate were used as the sound source to disintegrate larger agglomerates concentrated at the bottom of the bed. Nanoparticles showed fluid-like behavior similar to Geldart's A group and application of sound vibration improved their fluidization quality. Submicron particles were hard to fluidize and their fluidization quality was partially improved by sound excitation. Bed compaction, caused by rearranging of the agglomerates, was observed for submicron particles at low gas velocities while the bed was fixed. Nanoparticles did not experience any bed compaction. Sound vibration led to a decrease in minimum fluidization velocity and an increase in bed pressure drop and bed expansion for both types of particles. The fluidization quality of both particles increased at low frequencies, while the reverse was observed at higher frequencies. Fluidization of these particles was improved by increasing sound pressure level. There was a critical sound pressure level of 110 dB, below which the effect of sound vibration was insignificant. A novel technique was employed to find the apparent minimum fluidization velocity from pressure drop signals.

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#### 1. Introduction

Chemical industry is experiencing a growing interest in processing of ultrafine particles due to their exceptional physical and chemical properties. Fluidization of ultrafine particles is a promising technology for a number of industrial applications [1] such as pharmaceuticals, chemicals, foods, paints, dyes, inks, ceramics, cosmetics, powder metallurgy and powder coating. Recently, high purity nanoparticles with a narrow size distribution have helped to make advanced materials for the aerospace, transportation, electronics and health care industries. The unique property of ultrafine particles is their high surface area per unit mass, which enhances their activity enabling them to meet the process requirements of many products.

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 [2] 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 interparticle forces make the individual particles stick together and form agglomerates that can lead to severe

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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 [3–7]. It was shown that uniform fluidization of these powders was related to the existing agglomerates and 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 was the object of many researchers [8–15] in recent years. Nanoparticles with low density generally form porous agglomerates showing fluid-like fluidization with no bubbles and thus termed as Agglomerate Particulate Fluidization, APF [8]. Heterogeneous bubbling fluidization was observed for dense nanoparticles at high gas velocities [16].

In order to improve the fluidization quality of fine/ultrafine powders, various fluidization aids have been proposed such as mechanical vibration [17,18], sound wave vibration [19,20], mechanical stirring [21], magnetic field disturbance [22] and addition of fine particles as flow conditioner [23]. Application of fluidization aids reduces the gas velocity well below the minimum fluidization velocity of the particles for smooth fluidization. Valverde et al. [9] used different gasses 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. Morooka [4] reported that even some sub-micron particles could be fluidized fairly, since these particles agglomerated into larger particles and thus fluidized at higher gas velocities. Dutta and Dullea [18] used external vibration to improve the fluidization quality of fine cohesive powders,

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which simultaneously increased the bed pressure drop and the bed expansion as well as decreasing elutriation losses. Jung and Gidaspow [24] reported that nanoparticles could be fluidized due to the formation of light agglomerates. Barletta et al. [25] studied the effect of mechanical vibration on the fluidization of aeratable powders.

Due to the strong interparticles forces between submicron/nanoparticles, agglomeration is a common phenomenon observed in the fluidization of such powders [26]. The size and apparent density of agglomerates differ from the primary particles and therefore the physical properties of primary particles cannot be used for the prediction of their fluidization behavior. Depending on the type of particles, they may be self agglomerated forming stable and roughly mono-sized agglomerates [5,27]. In general, formation of agglomerates in a fluidized bed is the result of a dynamic equilibrium between formation and breakage of the agglomerates due to multiple collisions between the particles and also between the agglomerates and the column wall. If the interparticle forces are weak the agglomerates would be fragile, which makes the sampling of these agglomerates quite a challenge. Formation of agglomerates also depends on the type of the fluidizing gas, as well as its humidity and velocity.

A number of research groups have arbitrarily defined the boundary between submicron and nano powders using different particle sizes. In this study, particles between 100 nm and 1  $\mu$ m in size are considered as sub-micron powder and smaller particles (<100 nm) as nanoparticles.

Different techniques have been employed in the study of agglomerates such as freezing technique [7], particle/droplet image analysis [28], SEM analysis [10], X-ray imaging [28,29], online sampling [30].

The objective of this study is to improve the fluidization quality of nano and sub-micron powders using acoustic vibration to reduce interparticle forces. In most studies, sound wave generators (loud-speakers) were located in the freeboard; consequently the sound waves reached the particles/agglomerates on top of the bed first [24,31,32]. Due to the bed attenuation, a small fraction of sound waves may reach the particles/agglomerates at the bottom of the bed, where the larger agglomerates were concentrated. In this study, the loudspeakers were located under the distributor. The effects of sound frequency and sound pressure level 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 employed to find the apparent minimum fluidization velocity of submicron and nano powders.

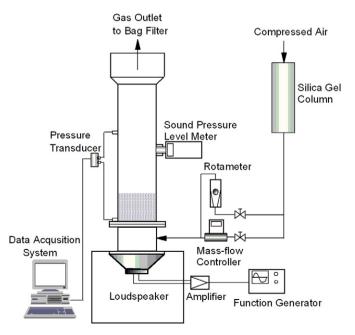


Fig. 1. Experimental setup.

#### 2. Experimental setup and procedure

The schematic diagram of the experimental setup is shown in Fig. 1. Compressed air stripped of 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. An 8-inch loudspeaker, located under the distributor, was used to generate an acoustic field. A digital signal generator (Wavetek 182A, 4 MHz Function Generator) provided an electric sine wave of specified frequency, which was further amplified (PB-110PX, 30W RMS) before sending to the loudspeaker. A digital sound pressure meter was mounted above the bed to measure sound pressure level.

In gas-solid fluidized beds, one or more resonant frequencies may exist depending on physical parameters of the vibrating objects. The agglomerates are assumed to act like rigid solid particles and the effect of external sound excitation on natural frequency of these agglomerates is negligible. Therefore the natural or resonant frequency of particle oscillation can be estimated as [33]:

$$f_{\rm r} = \frac{1}{4L} \sqrt{\frac{\rm RT} \rho_{\rm g}}{\epsilon (1 - \epsilon) \rho_{\rm p}} \tag{1}$$

where  $f_r$  is the resonant frequency, L is the bed height, R is the universal gas constant, T is absolute temperature,  $\rho_g$  and  $\rho_p$  are gas and particle densities and  $\varepsilon$  is the bed voidage. Acoustic oscillation strength may be expressed in dimensionless form as [34]:

$$Z = \frac{2\pi f P_m}{\rho_b C_b g} \tag{2}$$

where f and  $P_m$  are frequency and sound pressure and  $\rho_b$  and  $C_b$  are the bulk density and speed of sound of the fluidized bed emulsion phase.

The sound pressure and sound pressure level (SPL) are related as:

$$SPL = 20\log \frac{P_m}{P_{ref}}$$
 (3)

where  $P_{ref}$  is the reference sound pressure. The sound velocity in the emulsion phase can be estimated by [35]:

$$C_o = \sqrt{\frac{\left[\frac{\epsilon}{K_g} + \frac{(1-\epsilon)}{K_p}\right]^{-1}}{\epsilon \rho_g + (1-\epsilon)\rho_p}} \tag{4}$$

**Table 1**Properties of solid particles.

Powder	Average size	Density (kg m³) (aerated)	Density (kg m³) (tapped)	Hausner ratio (HR)	Angle of repose (degree)	Manufacturer
SiO <sub>2</sub> Al <sub>2</sub> O <sub>3</sub> TiO <sub>2</sub> ZrSi	App. 16 nm App. 16 nm 0.53 μm 0.74 μm	34 40 743 919	53 58 1209 1460	1.55 1.45 1.62 1.59	36.5 40.9 44.8 42.9	Degussa Al <sub>2</sub> O <sub>3</sub> _AluC Degussa Fujian Sanxiang Metallurgy Co., Ltd. Degussa

Particle size: Volume-weighted mean diameter by laser diffraction (Malvern Mastersizer). Other particle properties: Analyzed by Hosokawa Powder Tester.

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