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# The influence of low pressure operation on fluidization quality

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#### **Abstract**

The effect of low pressure operation on the fluidization quality of Group B powders has been studied. Pressure fluctuations have been analyzed—using the standard deviation, the power spectral density function (PSDF) and the auto- and cross-correlation functions for operating pressure down to 4 kPa. Low pressures were found to lead to slugging behavior at lower gas velocities than predicted using correlations applicable to higher pressure operation. Although the fluidization quality was found to be little affected by small decreases in pressure below the ambient level, quite drastic decrease on the fluidization quality were observed at the approach to the 4 kPa lower limit investigated in this study.

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# **1. Introduction**

The hydrodynamic behavior and, hence, the fluidization quality of fluidized beds is well known to depend on the conditions of operation. A great many industrial fluidized beds operate at pressures and temperatures higher than ambient. For this reason, fluidization under these conditions has been the subject of considerable study. Relatively little of this work, however, has addressed the question of fluidization quality—which Rapagná et al. [\[1\]](#page-6-0) and Llop and Arnaldos [\[2\]](#page-6-0) have shown to be affected by temperature and pressure, respectively. Recent applications of fluidized bed technology under partial vacuum conditions have focused interest on the study of fluidization at reduced pressure. Fuchs and Zeller [\[3\]](#page-6-0) and Arnaldos et al. [\[4\]](#page-6-0) have studied the drying process of thermolabile powders under reduced pressure as a means of reducing the operating temperature. Fletcher at al. [\[5\]](#page-6-0) in order to reduce the temperature and thereby the severity of oil sand cracking, have used a fluidized bed pyrolyser operated at reduced pressure. Caussat et al. [\[6,7\]](#page-6-0) have treated powder surfaces by chemical vapor deposition in a fluidized bed operated at low pressure and high temperature. Kusakabe et al. [\[8\]](#page-6-0) and Llop et al. [\[9\]](#page-6-0) have proposed that at low pressures the molecular flow must be considered, and the slip term contribution added to the pressure drop equation for an improved estimate of minimum fluidization velocity.

Rodríguez Ruvalcaba et al. [\[10\]](#page-6-0) considered fluidization at reduced pressure to take place in a progressive manner: they obtained an initial incipient fluidization velocity, and a final one corresponding to complete fluidization—both well predictable by means of the Ergun equation.

Fluidization quality has been related to bed pressure fluctuations by a number of authors on the basis that the number, velocity and size of the bubbles affect both these phenomena [\[11\]. L](#page-6-0)irag and Littman [12] showed that bed pressure fluctuations can provide considerable insight into the fluidization process. Dhodapkar and Klinzing [\[13\]](#page-6-0) found it more useful to analyze the frequency domain than the amplitude of the fluctuating pressure signal for relating pressure fluctuations to the state of fluidization in a bed. Nicastro and Glicksman [\[14\]](#page-6-0) and Di Felice et al. [\[15\]](#page-6-0) used the power spectral density function (PSDF) in applying the scaling relationships to fluidized systems—as did Rodríguez Ruvalcaba et al. [\[10\],](#page-6-0) together with the standard deviation of the pressure record, in comparing differences in fluidization behavior under low and ambient pressure conditions. There are, however, few studies of the fluidization process under partial vacuum and, as a consequence, the behavior of fluidized beds under these conditions is not well known.

In this paper, the quality of fluidization at low pressures is characterized in terms of instantaneous differential pressure measurements. The analysis of the pressure fluctuations has been carried out in the frequency and amplitude domains in an attempt to relate the fluidization quality to the power spectral density function (PSDF). For this purpose,

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several periodograms have been averaged—as suggested by Brown and Brue [\[16\]](#page-6-0) for obtaining accurate estimates of the PSDF. Standard deviations and the auto-correlation of pressure fluctuations have also been obtained, as well as the cross-correlation for pressure fluctuations recorded at two different locations in the bed.

# **2. Experimental**

The scheme of the experimental set-up is shown in [Fig. 1.](#page-2-0) The fluidization column was of glass in order to allow for visual observation. Its inside diameter and height were 76 and 600 mm, respectively. A perforated plate 2-mm thick was used for the distributor. A nylon mesh was placed above this to avoid the weeping of particles when the flow was stopped. Rashing rings were placed in the calming section below the distributor, in order to increase the uniformity of the gas distribution. The fluidizing medium was air, at  $20^{\circ}$ C and at several pressure levels ranging from 4 to 101 kPa. A membrane valve, located between the pump suction and the top of the column, and a pin valve, connected before the entrance of the membrane valve to atmosphere, controlled the vacuum in the column. A vacuum-meter, installed on the column exit pipe, measured the pressure. Pressure drop fluctuations in the bed were measured by pressure probes connected across two differential pressure transducers, both located in the top section of the column. Both transducers measured the local pressure relative to that in the freeboard: one close to the distributor; and the other some 90 mm above the first. This second transducer was used for the cross correlation analysis of the pressure signal. The voltage signals from the two transducers were sent simultaneously to an A/D board located in a personal computer; the data were stored and analyzed off-line using the adequate software. The sampling frequency was higher than 100 Hz, and 4096 data points were obtained for each channel. At each pressure level, more than 10 increasing values of gas velocity were investigated—from minimum fluidization conditions to the maximum bed expansion permitted by the height of the column. The inlet air flow rate was measured under ambient condition using a rotameter manifold.

The bed consisted of silica sand particles, of  $2650 \text{ kg/m}^3$ density and of shape factor in the range of 0.75–0.8. Three mean particle diameters (225, 300, and 475  $\mu$ m) were tested. The settled bed of particles was 130 mm high. The minimum fluidization velocity was obtained experimentally by plotting the bed pressure drop against decreasing superficial gas velocity.

# **3. Results and discussion**

### *3.1. Analysis of pressure fluctuations amplitude*

The standard deviation of the pressure fluctuations increases with the size of bubbles in the bed: high values relate to large bubble diameters and correspondingly strong heterogeneous fluidization—which is associated with low fluidization quality. For homogeneous fluidization, on the other hand, pressure fluctuations are of low amplitude resulting in low standard deviation values.

Homogeneous fluidization results in minimal by-pass of gas, so that good contact is maintained with the solid phase. Heterogeneous fluidization, on the other hand, results in greater gas–solid mixing which may lead to improvements in mass and heat transfer. It follows from these considerations that the optimal fluidization quality depends on the specific application in hand. Instantaneous pressure fluctuations, although insufficient for a complete characterization of the state of fluidization, can nevertheless offer appreciable information on its quality.

In [Fig. 2,](#page-2-0) the standard deviation of the pressure fluctuations, for all studied bed pressures, are shown plotted against  $u/u_{\text{mf}}$  for particles of 475  $\mu$ m. No clear influence of pressure level on the standard deviation is observed, except at the lowest bed pressure (4 kPa): where the standard deviation is clearly smaller than for the other recorded pressure levels at the same fluidization velocities. Similar behavior is observed with the  $225$  and  $300 \,\mu m$  particles. These results are in agreement with the data of Rodríguez Ruvalcaba et al. [\[10\]:](#page-6-0) they observed the standard deviation of pressure

<span id="page-2-0"></span>

Fig. 1. Schematic view of the experimental set-up.



Fig. 2. Standard deviation of pressure bed fluctuation vs. velocity ratio at several pressures for particle of  $475 \mu m$  average diameter.

fluctuations at 13.3 kPa to be lower than at atmospheric pressure for air fluidization of  $100 \mu m$  alumna particles.

The smaller values of the standard deviation of pressure fluctuations at 4 kPa is not due to an enhanced homogeneity of fluidization. Pressure fluctuations generally originate from three principal causes: the formation of jets at the distributor, the travelling of bubbles through the bed, and their eruption at the bed surface. It was visually observed that at 4 kPa the fluctuations of bed surface were smaller, in accord with lower standard deviation values obtained. This decrease in standard deviation was not due to a decrease in bubble size but in the development of an unusual type of slug formation, consisting of dense phase regions of higher void fraction, travelling upwards through the bed and erupting periodically at the bed surface.

At gas velocities close to incipient fluidization, the lower zone of the bed was observed to remain unfluidized. This lack of fluidization near the distributor is a typical feature of slugging systems, and could explain the decrease in standard deviation at 4 kPa pressure. In addition, as the velocity is increased and the whole bed is fluidized, a sudden jump is observed in the standard deviation—almost bringing it to values typical of higher pressure conditions.

## *3.2. Effect of low pressure on fluidized bed structure*

In order to relate the state of fluidization in the bed to the different operating conditions, the pressure fluctuations records were processed to obtain the PSDF for each particle diameter, pressure level and gas velocity. For the three diameters investigated at atmospheric pressure, the variation of the spectrum with fluidizing velocity follows the same trends previously observed by other investigators [\[1,13\]. F](#page-6-0)or velocities near to those at minimum fluidization, the PSDF is distributed over a quite wide range of frequencies. Distinct frequencies start to appear at the minimum bubbling point. Further increases in gas velocity increases the magnitude of the power due to increases in bubbles size by coalescence. At very high gas velocities, very few peaks with high magnitude are observed—as is typically encountered for fluidization in the slugging regime.

Fig. 3 shows the influence of low pressure on the PSDF. From atmospheric pressure to 60 kPa (Fig. 3(a) and (b)), a slight decrease in the intensity of the peaks is observed, together with a small increase in band width. This could be due to a slight decrease in the bed surface oscillations, bubble size and the extent of coalescence. At this pressure, the PSDF appears with narrower bands, composed of few dominant frequencies with strong amplitudes. This tendency becomes more pronounced as the bed pressure is decreased (Fig. 3(c) and (d)). Visual observations, however, reveal that the bed surface fluctuations diminish. For all pressures, the dominant frequencies lie between 5 and 6 Hz, and for the lowest pressure investigated (4 kPa) only one dominant frequency is observed (as shown in Fig. 3(d)). The magnitude of the power spectrum in this case is almost 10 orders of magnitude greater than at atmospheric pressure, and the dominant frequency has a very low value. This is clearly indicative of strong slugging flow: the bubbles coalesce rapidly into a single void, which travels through the bed.

At the lowest pressure investigated, the slugging characteristics appear as soon as the bed is fluidized for  $475 \mu m$ diameter particles—as shown in Fig. 4; the signal amplitude increases as the superficial gas velocity is increased. Frequency values in Fig. 4 have been divided by the dominant frequency, to illustrate better the growth of dominant amplitude of the PSDF.

The dominant frequency, defined as the frequency with the maximum amplitude, is analyzed for each run from the corresponding power spectrum. Although a certain dispersion of the data is observed, the gas velocity only marginally influences the dominant frequency—as shown in [Fig. 5. H](#page-4-0)owever, the operating pressure affects the dominant frequency: when the mean value of the dominant frequency is plotted for each



Fig. 3. Power spectral density function (PSDF) at investigated pressures for 225  $\mu$ m diameter particles for  $u/u_{\text{mf}} = 1.65$ .

pressure and particle diameter, as in [Fig. 6,](#page-4-0) a general influence of pressure is observed. Decreasing the pressure from 101 to 40 kPa changes the dominant frequency smoothly, but a further lowering of the pressure towards 4 kPa decreases



Fig. 4. Influence of gas velocity on PSDF at 4 kPa.

<span id="page-4-0"></span>

Fig. 5. The dominant frequency vs. the gas velocity ratio for several pressures.

the dominant frequency strongly. Lower amplitudes and higher frequencies should correspond to smoother fluidized states, where the bubbles are small and the gas–solid contact is better. This suggests that a small increase in fluidization quality can be brought about by decreasing the operating pressure. Fluidization quality decreases visibly, however, when the pressure approaches the lowest level investigated in this study. This behavior could well be related to a minor penetration of the gas jets generated in the holes of the distributor plate. An increase in vacuum conditions causes bubbles to coalesce more and, hence, result in slugging behavior.

The auto-correlation function provides an estimate of the time delay between similar events in the fluid bed. The events are the pressure oscillations in the bed due to the travelling of bubbles or slugs and their eruption at the surface. The time delay can be obtained from the maximum value of the auto-correlation function. It relates to the inverse of the dominant frequency in the PSDF of the same signal [\[17\].](#page-6-0) Fig. 7 compares the auto-correlation functions for the same velocity ratio at two different pressure values for the  $475 \mu m$ 



Fig. 6. Dominant frequency vs. the operation pressure.



Fig. 7. Auto-correlation function of the differential pressure fluctuations at the extreme pressures investigated for the particle diameter of  $475 \mu m$ .

particles. At the lower pressure, the periodic component is stronger and the lag time higher, which confirms complete slugging behavior. It can be seen that as the pressure decreases from ambient to 60 kPa (Fig. 8(a)), the periodic component almost do not change and the lag time decreases smoothly (the frequency increases). These may indicate a small increase in fluidization quality in agreement with the findings of Fletcher et al. [\[5\].](#page-6-0) Decreasing further the pressure to 4 kPa (Fig. 8(b)), causes a progressive increase in the periodicity, where the maximum is observed demonstrating the transition to slugging regime with the higher lag time.

### *3.3. Wave pressure velocity*

Fan et al. [\[18\]](#page-6-0) used the cross-correlation function to determine bubble and slug velocities in gas fluidized beds. The velocity was calculated from the time taken by pressure perturbations to travel between two pressure taps placed on



Fig. 8. Auto-correlation function of the differential pressure fluctuations for several pressures investigated for the particle diameter of  $225 \,\mu m$ .



Fig. 9. Influence of operating pressure on the wave velocity.

a vertical line in the bed. The shift time is obtained from the maximum value of the cross-correlation function of the two pressure signals. In fact, the velocity calculated in this way is the average velocity of pressure wave propagation. Different wave forms give rise to interference [\[19\].](#page-6-0) The influence of pressure on the wave velocity is shown in Fig. 9. It seems that this increases slightly with decreasing pressure down to about 40 kPa, and then decreases rapidly with further pressure reduction. No significant change in this behavior has been observed for the different particle diameters investigated.

## *3.4. Slugging behavior*

The analysis of pressure fluctuations described earlier establishes that slugging behavior tends to increase with decreasing pressure below the ambient level. This clearly follows from the plot of PSDF for group B particles. The observed decrease in both bubble frequency and velocity confirms this behavior. Although slugging is more common with large particles and deep beds, it is possible to observe this regime with group B particles and relatively shallow beds. Slugging in beds with  $H_s/D_c > 2$  is frequently observed, but less so for  $2 > H_s/D_c > 1$ , and never for  $H_s/D_c$  < 1 except for the case of very large or very dense particles [\[20\].](#page-6-0) Broadhurst and Becker [\[20\]](#page-6-0) proposed a correlation for estimating the minimum slugging velocity as a function of several parameters, for  $H_s/D_c < 3$ ,

$$
u_{\rm ms} = 7.17 \left(\frac{D_{\rm c}}{H_{\rm s}}\right)^{0.895} \left(\frac{g(\rho_{\rm p} - \rho_{\rm g})d}{\rho_{\rm p}}\right)^{0.5} \left(\frac{\rho_{\rm g}}{\rho_{\rm p}}\right)^{0.045} \tag{1}
$$

Even if the correlation predicts a decrease in minimum slugging velocity with decreasing pressure, but it over estimates  $u_{\text{ms}}$  for the 225 and 300  $\mu$ m diameter particles, mainly at 4 kPa, the lowest pressure investigated. Precisely, the *u*ms predicted is  $0.13 \text{ m/s}$  for  $225 \mu \text{m}$  diameter particles (the work range was from 0.05 to 0.135 m/s) and 0.15 m/s for  $300 \mu m$  diameter particles (the work range was from 0.09



Fig. 10. Influence of the Knudsen number on the wave pressure velocity.

to 0.19 m/s). For these particles, the slugging appears at gas velocities close to that for minimum fluidization. For the  $475 \mu m$  diameter particles, the predictions justifies reasonably that the slugging appears as soon as the bed is fluidized.

Roth et al. [\[21\]](#page-6-0) defined three different flow regimes at partial vacuum condition; depending on the pressure and the bed diameter these are laminar, intermediate, or slip and molecular flow. Using the same criteria as Roth et al. [\[21\],](#page-6-0) Llop [\[22\]](#page-6-0) established for fluidizing by air at ambient temperature, the boundaries of these regimes up to the viscous limit as a function of the particle  $Kn_p$ , defined by

$$
Kn_p = \frac{\lambda}{d} = \frac{kT}{2^{12}\pi\xi^2 P d}
$$
\n(2)

These boundaries become: for molecular flow  $Kn_p$ 0.49; for intermediate flow  $4.9 \times 10^{-3} < K n_{\rm p} < 0.49$ ; and for laminar or viscous flow,  $Kn_p < 4.9 \times 10^{-3}$ .

The Fig. 10 shows the pressure wave velocity; calculated by cross-correlation analysis, versus *Kn*p. The particle Knudsen number depends inversely to the gas density and particles mean diameter. This figure shows clearly that there is a variation of fluidization quality close to the Knudsen number of 0.0007, that achieved for 225, 300, and 475  $\mu$ m particles diameter, respectively, at the operating pressures of 40, 30, and 20 kPa. At this Kundsen number far from slip flow, begins the transition from vigorous bubbling to slugging regime. Decreasing the pressure and, therefore, the density of the gas, increases its compressibility and promote the slug formation that will be completed when the flow regime is next to the slip flow.

#### **4. Conclusions**

Pressure fluctuations in a fluidized bed have been used to investigate behavior and fluidization quality under partial vacuum conditions for Geldart group B particles. The technique is shown to be helpful for characterization of the state of a fluidized bed.

<span id="page-6-0"></span>Analysis of the standard deviation of the pressure fluctuations indicates no significant changes in bed structure at pressures near atmospheric; at lower pressures, however, slugging (slip–flow regime) action occurs and intensifies with increasing vacuum. The absence of fluidization in the bottom part of the bed, which is an important characteristic of the slugging regime for reasons described by Chen et al. [23], was observed at velocities much higher than  $u_{\text{mf}}$ , while the upper part of the bed remained well fluidized. Kusakabe et al. [8] observed similar features at low pressures with fine particles. Rodríguez Ruvalcaba et al. [10] suggested that progressive fluidization occurs under slip–flow conditions which is the flow regime relevant at high vacuum. In fact, the results of the present work suggest that the observations of these authors could well be due to slugging fluidization, which prevails in deep beds and, at low velocities, is characterized by the absence of fluidization in the bottom part of the bed.

The quality of fluidization is not so excessively affected by modest pressure reductions below the ambient level, in any case it seams to increase smoothly in agreement with the observations of Fletcher et al. [5]. When the absolute pressure decreases below about 40 kPa the fluidization quality progressively decreases and, at 4 kPa, a drastic decrease is observed. Under these vacuum conditions, slugging behavior becomes evident also at very low gas velocities, close to that for minimum fluidization.

This study highlights the importance of knowledge of fluidization characteristics under vacuum conditions, which can lead to significant departures from hitherto expected behavior, and which is necessary for the development of potential applications. It also points out the need for further research into the behavior of fluidized beds under low pressure conditions.

#### **References**

- [1] S. Rapagná, P.U. Foscolo, L.G. Gibilaro, The influence of temperature on the quality of gas fluidization, Int. J. Multiphase Flow 20 (1994) 305–313.
- [2] M.F. Llop, J. Arnaldos, Influencia de la presión sobre las fluctuaciones de pérdida de carga en lechos fluidizados sólido-gas, Información Tecnológica 110 (1998) 51–58.
- [3] G. Fuchs, A. Zeller, Vacuum fluidized bed drying of granular powders in pharmaceutical production, Pharm. Ind. 54 (10) (1992) 881–885.
- [4] J. Arnaldos, B. Kozanoglu, J. Casal, Vacuum fluidization: application to drying, in: Proceedings of the Ninth International Fluidization Conference, Engineering Foundation, New York, 1998, pp. 709– 715.
- [5] J.V. Fletcher, M.D. Deo, F.V. Hanson, Fluidization of a multi-sized Group B sand at reduced pressure, Powder Technol. 76 (1993) 141– 147.
- [6] B. Caussat, M. Hémati, J.P. Couderc, Silicon deposition from silane or disilane in a fluidized bed. Part I: Experimental study, Chem. Eng. Sci. 50 (1995) 3615–3624.
- [7] B. Caussat, M. Hémati, J.P. Couderc, Silicon deposition from silane or disilane in a fluidized bed. Part II: Theoretical analysis and modeling, Chem. Eng. Sci. 50 (1995) 3625–3635.
- [8] K. Kusakabe, T. Kuriyama, S. Morooka, Fluidization of fine particles at reduced pressure, Powder Technol. 58 (1989) 125–130.
- [9] M.F. Llop, J. Madrid, J. Arnaldos, J. Casal, Fluidization at vacuum conditions: a generalized equation for the prediction of minimum fluidization velocity, Chem. Eng. Sci. 51 (1996) 5149–5157.
- [10] J.R. Rodríguez Ruvalcaba, B. Caussat, M. Hemati, J.P. Couderc, Ëtude hydrodynamique des lits fluidisés sous vide et sous haute température, Can. J. Chem. Eng. 77 (1999) 35–44.
- [11] W.W. Shuster, P. Kisliak, The measurement of fluidization quality, Chem. Eng. Prog. 48 (1952) 455–458.
- [12] R.C. Lirag, H. Litman, Statistical study of the pressure fluctuations in a fluidized bed, AIChE Symp. Ser. 116 (1971) 11–22.
- [13] S.V. Dhodapkar, G.E. Klinzing, Pressure fluctuation analysis for a fluidized bed, AIChE Symp. Ser. 296 (1993) 170–182.
- [14] M.T. Nicastro, L.R. Glicksman, Experimental verification of scaling relationships for fluidized bed, Chem. Eng. Sci. 39 (1984) 1381– 1391.
- [15] R. Di Felice, S. Rapagná, P.U. Foscolo, Dynamic similarity rules: validity check for bubbling and slugging fluidized beds, Powder Technol. 71 (1992) 281–287.
- [16] R.C. Brown, E. Brue, Resolving dynamical features of fluidized beds from pressure fluctuations, Powder Technol. 119 (2001) 68–80.
- [17] N.N. Clark, E.A. McKenZie Jr., M. Gautam, Differential pressure measurements in a slugging fluidized bed, Powder Technol. 67 (1991) 187–199.
- [18] L.T. Fan, H. Tho-Ching, W.P. Walawender, Measurements of the rise velocities of bubbles, slugs and pressure waves in a gas–solid fluidized beds using pressure fluctuations signals, AIChE J. 29 (1983) 33–39.
- [19] Z. Fan, G.T. Chen, B.C. Chen, H. Yuan, Analysis of pressure fluctuations in a 2D fluidized beds, Powder Technol. 62 (1990) 139– 145.
- [20] T.E. Broadhurst, H.A. Becker, Onset of fluidization and slugging in beds of uniform particles, AIChE J. 21 (1975) 238–247.
- [21] A. Roth, Vacuum Technology. North-Holland, Amsterdam, 1976.
- [22] M.F. Llop, Contribució a l'estudi dels llits fluiditzats sotmesos a pressió i temperatura, Ph.D. Thesis, Universitat Politècnica de Catalunya, Barcelona, Spain, 1997.
- [23] Z. Chen, L.G. Gibilaro, P.U. Foscolo, Fluid pressure loss in slugging fluidized beds, Chem. Eng. Sci. 52 (1997) 55–62.