Fluidization and Hydraulic Behaviour of Natural Zeolite Particles Used for Removal of Contaminants from Wastewater

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Fluidization, bed expansion, pressure drop, and hydraulic characteristics of beds of natural zeolite particles have been studied for their potential application in the treatment of aqueous waste. The measurements in the bed of zeolite particles are compared with regular shaped glass beads. The bed of zeolite particles required to be fully fluidized before getting reproducible pressure measurements in the bed. This is attributed to their wider size distribution and resulting segregation. The mean size of the particles ranged from 550 to 900 μm. Minimum fluidization velocity was determined from pressure measurements below the distributor and compared with values from pressure measurements inside the bed. Experiments conducted with slurry out the bed showed that it could be easily drained in expanded mode from the column through a small diameter opening near the bottom.

La fluidisation, l'expansion du lit, la perte de pression et les caractéristiques hydrauliques des lits de particules de zéolite naturelles ont été étudiées pour leur application potentielle dans le traitement des déchets aqueux. Les mesures du lit de particules de zéolite sont comparées à des billes de verre de taille régulière. Le lit de particules de zéolite doit être entièrement fluidisé avant que l'on puisse obtenir des mesures de pression reproductible dans le lit. On attribue cette situation à leur distribution de plus grande taille et à la ségrégation qui en résulte. La taille moyenne des particules allait de 550 à 900 μm. La vélocité de fluidisation minimale était déterminée à partir de mesures de la pression sous le distributeur et comparée aux valeurs des mesures de la pression à l'intérieur du lit. Les expériences réalisées avec la boue qui sortait du lit ont indiqué qu'on pouvait facilement la drainer en mode expansé de la colonne par une ouverture de petit diamètre près de la partie inférieure

Keywords: natural zeolites, pressure drop profile, fluidized bed, bed expansion, spent bed removal

INTRODUCTION

In the past liquid fluidized beds have taken a backseat to gas fluidized beds because of their limited application in industry, but more recently they are becoming more widely used and the research concerning them is more abundant. Liquid fluidized beds are most commonly used in wastewater treatment for the adsorption of contaminants, for separation of pharmaceuticals, proteins, and other components from broths as well as for applications in the mining industry such as mineral classification and elutriation. The expanded or fluidized bed allows processing of feedstock with suspended particles such as cell debris and precipitated fines. In several of these applications, having a classified fluidized bed or a bed containing particles with a wide-size distribution which readily segregates according to size and/or density is beneficial to the process (Anspach et al., 1999). Take expanded bed adsorption for example. This is a fluidized bed operation where the degree of expansion is very carefully controlled. The success of the adsorption process depends on a classified bed because this classification results in reduced local mixing, and therefore a more efficient adsorption process (Thommes et al., 1997). A classified fluidized bed is also important in the mining industry because the main purpose of many of the fluidized beds used in this industry is to separate particles of a certain size or density.

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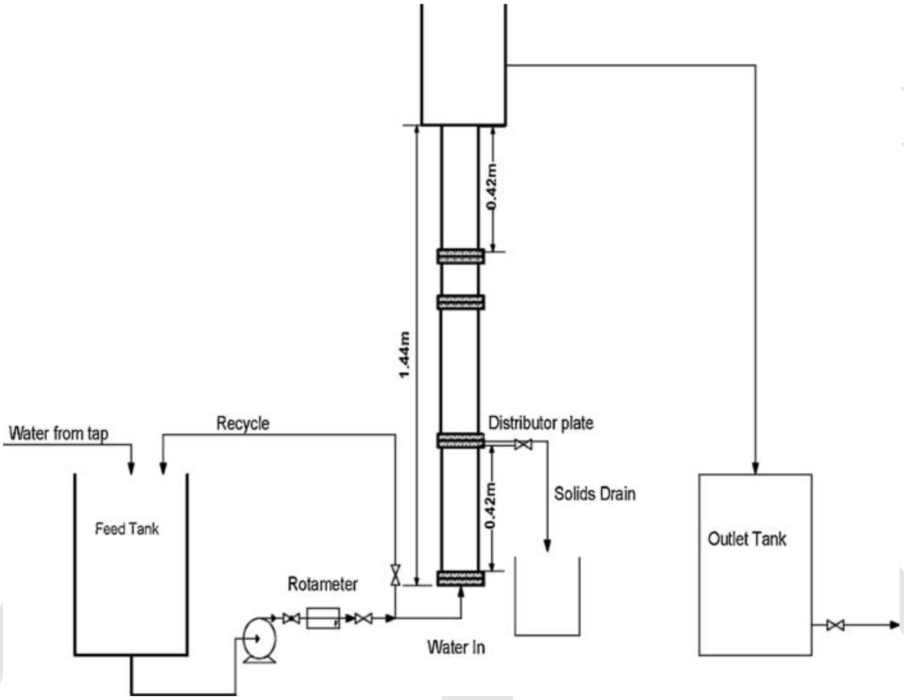

Figure 1. Schematic diagram of the experimental set-up.

The materials of interest in this study are two natural zeolites called chabazite and clinoptilolite. Natural zeolites are hydrated alumino-silicate minerals which possess an open cage-like structure and have a net negative charge. It is this negative charge which makes them particularly good sorbents for cations (Erdem et al., 2004). Moreover, their low cost and easy availability has made them attractive for a number of industrial applications. As a result interest in natural zeolites has been growing for sometime (Ouki et al., 1994; Stylianou et al., 2006). The material is generally used "as-received" having wide-size distribution and irregularly shaped particles making prediction of its behaviour in a fluidized bed rather difficult. Most of literature studies on hydrodynamics in fluidized and expanded beds have focused on beds containing narrow-sized or binary spherical particles. This study investigates pressure drop profile, bed expansion, and segregation in a bed of as-received particles of chabazite and clinoptilolite for application as adsorbents in expanded and fluidized beds. Slurrying out of particles bed is studied to facilitate the addition and removal of particles from the column. Comparisons are made with spherical glass beads of similar settling velocity to point out differences in hydrodynamic behaviour related to size and shape.

EXPERIMENTAL

Experiments were conducted in an acrylic column with an inner diameter of 0.1 m and a height of about 2 m. The column consisted of four sections, which could be moved around as needed. *O*-ring seals between the flanges of each section ensured that no water would leak out. The bottom section of the column served as the calming section. A perforated plate distributor installed in the second flange from the bottom of the column separated the particle bed from the calming section, and helped provide an even distribution of water into the bed. The distributor had 30 holes of 2.5 mm diameter on a triangular pitch. A wire mesh was also placed over the distributor plate to prevent the holes from becoming plugged with the particles.

The test column had a number of ports on its side wall. Two of these ports were used to measure pressure drop in the bed by connecting directly to water manometer tubes placed side-by-side on a flat vertical board with a measuring scale in between for accurate reading. A third port was used to measure the pressure below the distributor plate using a high accuracy digital pressure gauge from Omega (DPG-7000-10). The other ports were used to drain the particle bed from the column, and to take samples of the bed for analysis of the particle size distribution in different sections of the bed. The water flow rate into the column was controlled using rotameters with different ranges of flow, and the superficial velocities ranged from 1 to 8 mm/s. Both the manometer and the rotameters were calibrated using standard practices. During operation, water was fed to the bottom of the column from a 75 L holding tank, using a centrifugal pump. The water left the column through a square outlet tank installed on the top of the column, and was fed by gravity from this outlet tank into another 230 L holding tank. From here the water could either be sent back to the column or to the drain. A flow diagram of the experimental set-up is shown in Figure 1.

A number of different techniques were used to determine particles characteristics shown in Table 1. The skeletal density of the particles was determined using Helium pycnometry, and BET analysis allowed the determination of the particle density using the pore volume. The particle size analysis was performed using a Malvern Mastersizer 2000, which is based on laser diffraction technique. The particle size distribution of chabazite and glass beads are compared in Figure 2. It can also be noted that chabazite had a significantly wider-size distribution than the glass beads.

Experiments were performed to study variations of pressure below the grid, bed pressure drop, and bed expansion, etc., as a function of superficial liquid velocity. The total system pressure was measured using a digital pressure gauge below the distributor plate to record highest pressure in the system following a common practice in industry. For each set of experiments, the superficial liquid velocity was increased incrementally and a mea-

Table 1. Properties of particles used in the study

Particle type	Mean diameter (μm)	Particle shape factor	Skeletal density (kg/m^3)	Particle density (kg/m^3	BET pore volume (cc/g)	Static bed height (m)
Glass beads	551	Spherical	2490	2490	N/A	0.248
Chabazite	930	0.85	2106	1630	0.359	0.252
Clinoptilolite	890	0.85	2200	2010	0.083	0.25

surement was taken after waiting for the reading to stabilize. When the top of the flow range was reached, the bed was fully fluidized, and the measurements were then taken while decreasing the superficial velocity incrementally. Observations about segregation, particles movements, and instabilities in the particle bed were also recorded. In order to compare these results to those obtained for spherical particles, the same sets of experiments were performed with glass beads. A number of experiments were performed with irregularly shaped zeolite particles to test reproducibility of results. Experiments were also conducted to slurry out the bed of particles at the end of a run. The measured parameters were the time taken for the bed and column to drain and the volume of water collected with the particle bed. A Model was also developed to predict this time based on mass and momentum balance equations.

RESULTS AND DISCUSSION

Pressure Profile and Minimum Fluidization Velocity

Figure 3 compares the measured pressure below the distributor plate with chabazite particles for increasing and decreasing liquid velocity. It can be observed that there is a significant difference between the increasing and decreasing velocity curves. The increasing velocity run results in pressure fluctuations and significantly higher pressure measurements. It was observed that during increasing liquid flow, a "plug" of particles formed slowly dividing the bed into two sections. An air gap was created by the air driven out from porous chabazite particles as pores got filled by water. Higher pressure measurements were recorded during this process of plug formation in the bed. The pressure fluctuations recorded during the increasing velocity runs could be attributed to changes in the bed voidage as fine particles moved upwards in the bed.

Such a behaviour of irregularly shaped particles has been recorded in earlier literature studies. In their study of the fluidization characteristics of biobone particles, Ellis et al. (1996) found that pressure drop data obtained using increasing superficial velocity pressure data could not be used to accurately predict the minimum fluidization velocity because of mechanical interlocking of the needle-shaped particles. While this effect does not appear to be as severe with chabazite particles, the wide-size distribution and irregular shape of the chabazite particles had an effect on pressure measurements with increasing liquid velocity. However, measured pressure with decreasing velocity is much smoother and shows two clearly different slopes. A linear regression of the data set can be represented by two trend lines with intersection at liquid velocity of about 3.2 mm/s (Figure 3). A significant decease in the slope of the second line indicates that the bed was fluidized above this velocity—the minimum fluidization velocity.

Comparison with Glass Beads

A comparison of the pressure measurements below the distributor plate for chabazite and glass beads is shown in Figure 4. It can be seen that for glass beads both the increasing and decreasing velocity curves are very similar, essentially overlapping thus showing good reproducibility. This can be attributed to their nonporous structure and their narrow-size distribution. For the glass beads it can also be observed that there is a significant change in the slope of the curve above liquid velocity of about 3 mm/s. The minimum fluidization velocity for glass beads determined through linear regression is 2.7 mm/s. It can be seen that the chabazite particles do not show a similar distinct change in slope. Because of their wide-size distribution, the onset of fluidization is much less pronounced.

A number of correlations have been reported in the literature for the estimation of minimum fluidization velocity. These corre-

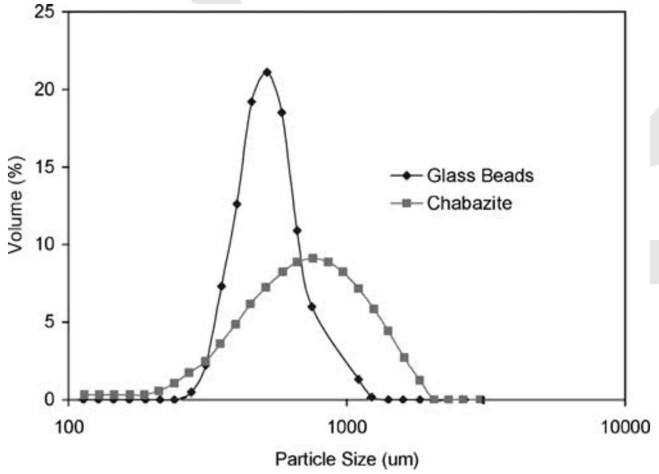

Figure 2. Comparison of particle size distributions of chabazite and glass beads.

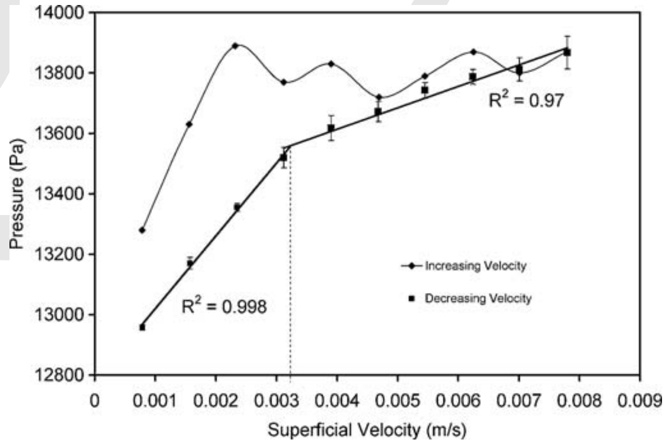

Figure 3. Measured pressure below distributor as a function of superficial velocity for chabazite particles with increasing and decreasing flow.

Table 2. Comparison of literature correlations for minimum fluidization velocity

Refs.	Correlation constants	Glass beads $U_{\mathrm{mf,calc}}$ (mm/s) (% Abs Error)	Chabazite $U_{\mathrm{mf,calc}}$. (mm/s) (% Abs Error)
Wen and Yu (1966)	$C_1 = 33.7$; $C_2 = 0.0408$	2.63 (2.6)	2.81 (12.0)
Saxena and Vogel (1977)	$C_1 = 25.28$; $C_2 = 0.057$	4.76 (76)	5.0 (56.0)
Grace (1982)	$C_1 = 27.2$; $C_2 = 0.0408$	3.2 (18.5)	3.42 (6.9)
Chitester et al. (1984)	$C_1 = 28.7$; $C_2 = 0.0494$	3.7 (37.0)	3.91 (22.2)
Lucas et al. (1986)	$C_1 = 29.5$; $C_2 = 0.0357$	2.62 (3.0)	2.8 (12.5)

$U_{\mathrm{mf,exp}}$: chabazite particles: 3.2 mm/s; glass beads: 2.7 mm/s, %AbsError = $\left|\frac{U_{\mathrm{mf,calc.}} - U_{\mathrm{mf,exp}}}{U_{\mathrm{mf,exp}}}\right|$

lations have the general form shown in Equation (1):

$$Re_{\mathrm{mf}} = -C_1 + (C_1^2 + C_2 Ar)^{0.5} \tag{1}$$

where Re_{mf} is the Reynolds number at minimum fluidization, Ar is the Archimedes number. The values of the two constants C_1 and C_2 vary somewhat with suggested correlations. These correlations were tested and compared to the experimental results using absolute percent error and the results are summarized in Table 2. It can be seen that the correlations by Wen and Yu (1966), Grace (1982), and Lucas et al. (1986) which have similar theoretical basis provided reasonable predictions. For glass beads the correlations by Wen and Yu (1966) and Lucas et al. (1986) gave excellent predictions with error of $<4\%$ and for chabazite particles the correlation from Grace (1982) provided the best predictions of the experimental value with an error of 6.9%. These differences can be related to the data set used by the authors for estimation of constants C_1 and C_2.

The pressure drop within the bed of particles was also measured using a water manometers and the results with decreasing velocity are compared in Figure 5 with the measurements of pressure below the distributor plate. Linear regression performed for the run resulted in a minimum fluidization velocity of about 3.8 mm/s which is slightly higher than the one obtained from the pressure measurements below the distributor (3.2 mm/s). This difference could be attributed to different particle size distribution between the two taps of the manometer in the bed due to particle segregation. It was observed that the bed of zeolite particles segregated easily even at low superficial velocities and distinct layers of different particle sizes could be seen. Particle samples were withdrawn and analysed from top, middle, and bottom layers of the bed and the average size was 512, 717, and 1125 μm, respectively. Segregation of particles of different sizes in liquid fluidized bed has been reported in literature studies (Epstein and Pruden, 1999; Chen et al., 2002; Chavan and Joshi, 2008). For segregation to occur, the size ratio of the segregated fractions generally need to be about 1.4 or more while for lower size ratios, there is intermixing of particles (Chavan and Joshi, 2008). The measured particle size in three segregated layers roughly met this criterion. A mixed bed of zeolite, chabazite, and clinoptilolite containing 50 wt.% of each was also studied. These two zeolites have different sets of adsorption characteristics which could be better utilized in a mixed bed for some applications. The two zeolites were added separately into the column as would be practiced in industry. It was observed that the two zeolite particles mixed well (the two had different colours) as the liquid velocity increased but did segregate in layers of different sizes. The observed mixing of the two zeolites can be attributed to a low ratio (~1.05) of their mean size. The behaviour of measured pressure below the distributor was similar to bed of chabazite particles. The minimum fluidization velocity in the bed of mixed zeolite was higher by about 20% which could be attributed mainly to higher density of clinoptilolite.

The bed expansion measurements obtained with chabazite and glass beads are shown in Figure 6. It can be observed that with glass beads, there is very little bed expansion until minimum fluidization velocity above which bed expansion is uniform and linear. This behaviour can be attributed to their narrow-size distribution. Chabazite on the other hand shows a more gradual change in slope as a result of their wide-size distribution and a distinct change in slope occurs around minimum fluidization velocity of

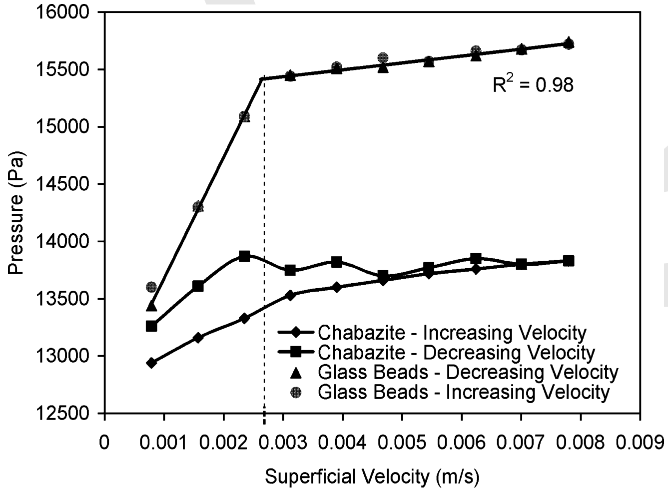

Figure 4. Comparison of pressure measurements below the distributor plate for chabazite and glass beads.

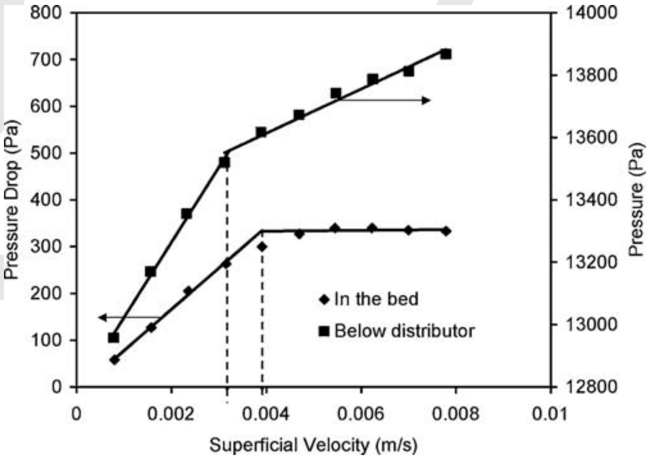

Figure 5. Comparison of pressure profile below the distributor and pressure drop measurements in the bed to determine the minimum fluidization velocity for chabazite particles.

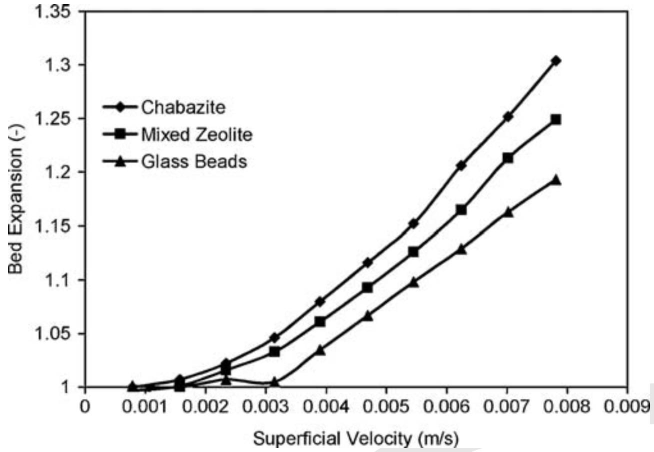

Figure 6. Relationship between bed expansion and superficial velocity for different particles.

the particles. The expansion characteristics of fluidized beds can be obtained from the Richardson–Zaki (1954) equation which predicts a linear relationship between superficial fluid velocity (U) and bed voidage (ε) on a log–log plot:

$$\frac{U}{U_t} = \varepsilon_b^n \tag{2}$$

$$\log(U) = n\log(\varepsilon_\mathrm{b}) + \log(U_\mathrm{t}) \tag{2a}$$

The Richardson–Zaki parameter (n) and terminal settling velocity were determined from linear regression of the log (ε) − log (U) plots. The corresponding bed voidage was calculated from the known particles weight and density:

$$\varepsilon_\mathrm{b} = 1 - \frac{(W_\mathrm{s}/\rho_\mathrm{s})}{A_\mathrm{c} h_\mathrm{b} g} \tag{3}$$

The values of the Richardson–Zaki parameter were found to be 2.5 and 3.7 for glass beads and chabazite, respectively. Correlations have been proposed in the literature for prediction of Richardson–Zaki index (Di Felice, 1995). Three of these correlations were used to estimate the index for the two particles and the results are reported in Table 3. It can be seen that estimations are reasonable (although on higher side) for glass beads and quite a bit lower for chabazite. It should be noted that the literature correlations are based on spherical particles with narrow distribution. The higher values for the chabazite particles is attributable to their wide-size distribution and less spherical shape (Di Felice, 1995). However, the wide-size distribution of chabazite particles seems to be having a larger effect here. As pointed out earlier bed segregation resulted in smaller particles layer at the top of the bed. When average particle size of the top layer was used, the predicted value of "n" were within 10% of the experimental value. This indicated that bed expansion behaviour was governed by the particle size of the top layer which occupied about 25% of bed height.

Spent Bed Removal

For an anticipated application of the zeolite particles, the bed of spent particles need to be removed by remote operation. Experiments were performed to determine the suitable flow rate of water, size of the drain line, time required to drain out the bed as, and measure the volume of water exiting with the particles. The slurry velocity in the drain pipe was assumed to be twice the critical velocity for slurry flow in pipeline (Oroskar and Turian, 1980) to get an estimate of pipe size. After initial experiments with different flowrates, the bed was fluidized close to minimum fluidization and it took about 20 s to slurry out the bed from column. The dynamic process of particles slurring out was modelled as below for scale up purposes.

Water and solids balances:

$$A_\mathrm{c}\frac{\mathrm{d}h_\mathrm{w}}{\mathrm{d}t} = (F_\mathrm{w}^\mathrm{in} - \varepsilon_\mathrm{p} F_\mathrm{sl}^\mathrm{bot} - F_\mathrm{w}^\mathrm{top}) \tag{4}$$

$$A_\mathrm{c}\frac{\mathrm{d}h_\mathrm{b}}{\mathrm{d}t} = \frac{(1-\varepsilon_\mathrm{p})F_\mathrm{sl}^\mathrm{bot}}{(1-\varepsilon_\mathrm{b})} \tag{5}$$

The bed voidage (ε_b) was assumed to be 1 at minimum fluidization and obtained from experimental data. The time-dependent voidage in the pipe (ε_p) was estimated based on change in height of water and bed level in the column and observation that all the inlet water flow was leaving from the drain pipe.

Momentum balance equation:

Rate of change of momentum = dynamic hydrostatic head − frictional losses.

The dynamic hydrostatic head was based on varying bed and water column heights and observation that total height was dropping during the drain time:

$$\text{Hydrostatichead} = \rho_\mathrm{b} g h_\mathrm{b} + \rho_\mathrm{w} g (h_\mathrm{w} - h_\mathrm{b}) \tag{6}$$

The fanning friction factor for the slurry flow (f_sl) was calculated based on correlations recommended in literature (Wasp et al., 1977; McCabe et al., 2001; Abulnaga, 2002).

Frictional losses:

$$\begin{gathered} h_\mathrm{f} = \frac{2 f_\mathrm{sl} L_\mathrm{P} U^2}{D_\mathrm{P}} \\ f_\mathrm{sl} = f_\mathrm{w}\left(1 + K_\mathrm{f}(1-\varepsilon_\mathrm{p})\left[\frac{g D_\mathrm{P}(\rho_\mathrm{s}-\rho_\mathrm{w})}{U^2 \rho_\mathrm{w}\sqrt{C_\mathrm{D}}}\right]^{1.5}\right) \\ C_\mathrm{D} = \frac{24}{\mathrm{Re_p}}(1 + 0.173\,\mathrm{Re_p^{0.657}}) + \frac{0.413}{1 + 16300\,\mathrm{Re_p^{-1.09}}} \\ f_\mathrm{w} = 0.0014 + \frac{0.125}{\mathrm{Re_w^{0.32}}} \end{gathered} \tag{7}$$

The drag coefficient in the above equation was calculated using correlation proposed by Turton and Levenspiel (1986) and the

Table 3. Summary of Richardson–Zaki predictions for glass beads

Refs.	Correlation	Glass beads n_calc (Relative Error)	Chabazite n_calc (Relative Error)
Richardson and Zaki (1954)	$n = 4.4\,\mathrm{Re_t^{-0.1}}$, For $1 < Re_\mathrm{t} < 500$	3.0 (20%)	2.88 (−22%)
Rowe (1987)	$\frac{(4.7-n)}{(n-2.35)} = 0.175\,\mathrm{Re_t^{0.75}}$	2.9 (16%)	2.8 (−24%)
Khan and Richardson (1989)	$\frac{(4.8-n)}{(n-2.4)} = 0.043\,Ga^{0.57}$	2.95 (18%)	2.8 (−24%)

n_calc: chabazite: 3.7; glass beads: 2.5, Relative Error = $[(n_\mathrm{calc} - n_\mathrm{exp})/n_\mathrm{exp}]100$

value of coefficient K_f was assumed to be 81 from Govier and Aziz (1972).

CONCLUSIONS

Pressure profile obtained below the distributor plate can be used to determine minimum fluidization velocity of a bed of particles. This approach is more practical in some industrial applications where number of pressure taps in the bed need to be minimized to avoid potential leakage problems. A gradually increasing pressure profile above the fixed bed region indicates the presence of wide-size distribution of particles and a sharp threshold from fixed bed to fluidized bed indicates narrow-size distribution in the bed. The bed expansion behaviour of particles with wide distribution can be dominated by the smaller size fraction in the bed. The procedure and simplified model developed to remove the spent bed of adsorbent particles from the column can be easily adapted to larger scale industrial application.

NOMENCLATURE

A_c	cross-sectional area of column (m^2)
Ar	Archimedes number $= \rho_l(\rho_p - \rho_l)gd_p^3/\mu^2$
C_1 and C_2	constants in Equation (1)
C_D	drag coefficient
d_p	particle diameter (m)
D_P	pipe diameter (m)
f	friction factor
F	flow (m^3/s)
F_w^{in}	inlet flow to column (m^3/s)
F_w^{top}	outlet flow from column top (m^3/s)
F_{sl}^{bot}	outlet flow from column top (m^3/s)
g	acceleration due to gravity (m/s^2)
Ga	Galileo number (same as Archimedes number)
h	height (m)
L_P	length of pipe (m)
n	Richardson–Zaki index
Re_{mf}	Reynolds number at minimum fluidization velocity
Re_p	Reynolds number based on particle diameter
Re_t	Reynolds number based on particle terminal velocity
t	time (s)
U	superficial velocity (m/s)
U_t	terminal settling velocity (m/s)
U_{mf}	minimum fluidization velocity (m/s)
W_s	weight of solid particles in bed (kg)

Greek Symbols

ε	voidage
ρ	density (kg/m^3)
μ	viscosity (Pa/s)

Subscripts

b	bed
f	friction
p	particle
P	pipe
s	solid
sl	slurry
w	water

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