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Hydrodynamic behavior of liquid–solid micro-fluidized beds determined from bed expansion

Xiangnan Li[a,b], Mingyan Liu[a,b,*], Yanjun Li[a,b]

[a] Collaborative Innovation Center of Chemical Science and Engineering (Tianjin), School of Chemical Engineering and Technology, Tianjin University, Tianjin 300072, China
[b] State Key Laboratory of Chemical Engineering (Tianjin University), Tianjin 300072, China

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ABSTRACT

The bed-expansion characteristics of liquid–solid micro-fluidized beds were experimentally studied. Bed columns with inner diameters of 0.8, 1.45, and 2.3 mm were fabricated based on capillaries. Five particle sizes in a range of 22–58 μm were investigated. Bed-expansion curves were plotted using visually recorded bed-expansion heights. The bed expansion and initial fluidization behavior were compared with predictions for conventional-scale beds. Evident differences are reflected in lower expansion ratios and higher minimum fluidization velocities for micro-fluidized beds. These were attributed to the increase in the internal surface area of the particle beds and specific surface area of wall contact. The wall effect for micro-fluidized beds at higher particle/bed diameter ratios caused higher local voidage and an increase in expansion ratio. Correlations for the exponent and proportional coefficient in the Richardson–Zaki equation for micro-fluidized beds were proposed. The minimum fluidization velocities were correlated using a modification of the Ergun equation.

Introduction

Fluidized beds are an essential operation unit in chemical engineering, providing intensification of mass- and heat-transfer characteristics by virtue of their excellent phase contact and mixing properties. The miniaturization of fluidized beds widens their areas of application and extends this means of conventional process intensification to mini- and micro-scale fluidic devices. Micro-fluidized beds are a promising type of micro-reactor, because the fluidized particles offer extra-high specific surface area of the solid phase while improving the diffusion-dominated interphase transfer of regular micro-fluidic devices. Use of such equipment avoids not only the need to fabricate complex internal structures but also the high pressure drop associated with micro-packed beds (Li, Liu, & Li, 2016a, 2016b; Liedtke, Bornette, Philippe, & de Bellefon, 2013; Losey, Schmidt, & Jensen, 2001). Although proportional scaling down of conventional-sized fluidized beds seems to be the easiest approach, micro-fluidized beds with extremely high height-to-diameter ratios have unique advantages in terms of radial distribution, wall flux, and residence-time controllability. For instance, a high wall flux of heat or light greatly improved the performance of pyrolysis or photo-catalytic reactions in micro-fluidized beds (Guo et al., 2016; Yang, Liu, & Lin, 2016; Yu et al., 2010; Zhang, Scott, & Silveston, 1994). These advantages, combined with operating features of fewer particles required for filling and convenience of loading and recycling particles, make micro-fluidized beds suitable for reaction kinetics studies and high-throughput catalyst evaluation.

Whether "micro" represents a laboratory scale in a conventional reactor engineering context or a column diameter of less than a millimeter, micro-fluidized beds show apparent changes in hydrodynamic characteristics. Many recent studies have aimed to describe and explain these changes. Especially for gas–solid micro-fluidized beds, experimental methods, which included common pressure-drop analysis (Rao, Curtis, Hancock, & Wassgren, 2010; Xu & Yue, 2009) and visual investigation via high-speed camera (Liu, Xu, & Gao, 2008; Wang & Fan, 2011), and computational fluid dynamics based on the discrete particle model (Wang, Tan, Van der Hoef, van Sint Annaland, & Kuipers, 2011) were employed to determine important hydrodynamic parameters. Correlations for minimum fluidization, minimum bubbling velocity, and terminal velocity were proposed to quantify the differences shown by micro-fluidized beds. The wall effect resulting from the reduction of

* Corresponding author at: School of Chemical Engineering and Technology, Tianjin University, Tianjin 300072, China.
E-mail addresses: myliu@tju.edu.cn, mingyanliu@126.com (M.Y. Liu).

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Nomenclature

D_{eq}	Equivalent tube diameter
D_t	Column or tube diameter
d_p	Mean particle diameter
F	Liquid viscous drag
f	Wall friction force
G	Gravitation
H	Bed height
k	Proportional coefficient of Richardson–Zaki equation
L	Equivalent tube length
n	Exponent of Richardson–Zaki equation
ΔP	Liquid pressure drop
Re_p, Re_t	Particle and terminal Reynolds numbers
U	Superficial liquid velocity
U_{mf}	Minimum fluidization velocity
U_t, $U_{t'}$	Calculated and extrapolated terminal velocities

Greek letters

ε	Voidage
ϕ	Sphericity
μ	Liquid viscosity
ρ	Density

Subscripts

0	Initial or static state
p	Particle
f	Fluid
w	Wall region of particle bed
b	Bulk region of particle bed
exp	Experimental value
cal	Calculated value

column diameter was most involved in interpretations of these differences. The actual influences of the wall effect generally included intensified wall friction and column diameter restriction. The wall effect inevitably had significant impact owing to the increase of the proportion of the wall region in micro-fluidized beds.

Work is still limited with respect to use of micro-fluidized beds for liquid fluidization. Doroodchi, Peng, Sathe, Abbasi-Shavazi, and Evans (2012) reported the fluidization characteristics of 225 μm glass particles in capillary tubes with a smallest inner diameter of 0.8 mm. As the bed diameter decreased, deviation of pressure drop and expansion ratio from previously reported correlations appeared. This was attributed to the resultant high shear rate of liquid; because of this, the particles were pushed toward the center of the tube, thereby forming a small gap between the fluidized bed and tube wall, as observed in their experiments. Zivkovic, Biggs, and Alwahabi (2013) fabricated a liquid micro-distributor in a rectangular polydimethylsiloxane micro-channel with a cross section of 400 μm × 175 μm and achieved ethanol fluidization of soda lime glass microspheres of 30 μm diameter. They obtained the minimum fluidization velocity and parameters of the Richardson–Zaki equation from linear regression of the visually measured expansion curve. In other work (do Nascimento, Reay, & Zivkovic, 2016), two rectangular poly(methyl methacrylate) (PMMA) micro-channels of 1 mm × 1 mm and 2 mm × 2 mm were used as micro-fluidized bed columns. Minimum fluidization velocities of various sized glass and PMMA particles in a range of 20–60 μm were determined. The values were higher than theoretical predictions and found to linearly increase with increasing adhesion force between the solid surfaces. Tang, Liu, and Li (2016) experimentally investigated the effects of particle size distribution and column diameter on the fluidization behavior of mini-fluidized beds. Differences between the exponent of the Richardson–Zaki equation obtained from experiment and that calculated by conventional-scale correlations were compared and discussed.

Although homogeneous expansion behavior of liquid fluidization seems to be common and simple, hydrodynamic differences caused by miniaturization still could be very complicated; however, for liquid fluidization, it is easier to give quantitative descriptions and explanations of the differences between macro and micro scales. This study focused on the size effect on important hydrodynamic characteristics, including expansion ratio and minimum fluidization velocity. Experimental results were compared with the values calculated from conventional-scale correlations. Differences resulting from the reduction of column diameter, particle size, and fluidization velocity were discussed. Proposed quantitative relationships are critical in the prediction and control of hydrodynamic performance of micro-fluidized beds and contribute to further study of more complex systems, such as those of gas–liquid–solid fluidization.

Experimental

Experimental setup

An annotated schematic diagram of the experimental setup is depicted in Fig. 1(a). All experiments were performed in three micro-fluidized bed columns with different inner diameters (0.8, 1.45, and 2.3 mm) and identical transparent height of 60 mm. A photograph of the micro-fluidized bed column fabricated based on a 0.8 mm inner diameter and 6 mm outer diameter quartz capillary is shown in Fig. 1(b). Profiles of the overflow weir and liquid distributor are illustrated in Fig. 1(c). A polytetrafluoroethylene (PTFE) overflow weir with a filter mesh-covered liquid outlet connected the enlarged conical outlet of the capillary. The liquid distributor, which was made of a polyethylene mesh (mean pore size of 20 μm) sandwiched between PMMA and silicone tubes, was sleeved on the entrance of the capillary by 5 mm height. The entire bed column was joined and sealed by epoxy adhesive. The two other sizes of column had similar configurations, but with different inner (1.45 and 2.3 mm) and outer (3 and 4 mm) diameters. The entrance diameter of the distributor was constant at 3 mm, which was larger than the largest inner column diameter.

Each micro-fluidized bed was vertically fixed between a light-emitting diode (LED) light source (HL-6500K, Vitt, China) and a high-speed camera (i-SPEED LT, Olympus, Japan). Distilled water was fed as the fluidization medium from a syringe pump (LSP01-2A, Longer, China). Expansion behavior of the micro-fluidized beds was recorded using two lenses: a wide-angle lens (AF FX 35 mm f/2D, Nikon, Japan) for recording the entire bed and a macro lens (MP-E 65 mm f/2.8 1–5×, Canon, Japan) for observation of local fluidization characteristics.

Flint glass microspheres of five different sizes were used as the solid particles. The wettability of the particles was improved to prevent their aggregation and column-wall adhesion that was reported in previous research (Zivkovic & Biggs, 2015). Before performing the experiments, the particles were etched with 4% HF for several minutes, and then washed and dried. The particle size distributions, measured by a laser diffraction particle size analyzer (Mastersizer 3000, Malvern, UK), are shown in Fig. 2. The detailed properties of the particle beds are listed in Table 1. The terminal velocities were calculated using the mean particle diameter (surface area equivalent mean) using Stokes' law. The bulk densities were measured using the capillary method (Zivkovic et al., 2013) and were the average values for the three different-sized columns. Each parti-