Chemical Engineering and Processing 78 (2014) 27–36

Contents lists available at ScienceDirect

Chemical Engineering and Processing: Process Intensification

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

Two-phase flow hydrodynamic study in micro-packed beds – Effect of bed geometry and particle size

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ARTICLE INFO

Article history:
Received 26 November 2013
Received in revised form 7 February 2014
Accepted 10 February 2014
Available online 20 February 2014

Keywords:
Hydrodynamics
Micro-packed bed
Visualization
Pressure drop
Hysteresis
Capillary shape

ABSTRACT

Microscopic visualizations nearby the wall region of micro-fixed beds and hydrodynamic measurements during gas–liquid two-phase flows were carried out with an aim to investigate the effect of particle size and capillary tube shape on the bed pressure drop, flow regime transition, hysteresis and bed transient response to flow-rate step perturbations. Visualizations through inverted microscopy revealed that a decrease in particle size leads to early inception of a high interaction flow regime whereas changing capillary shape from circular to square had no effect on flow regime changeover. The effect of particle size on the wetting pattern hysteresis in square micro-packed beds was also investigated in both imbibition and drainage paths. It was found that wetting pattern hysteresis decreases with a decrease in particle size. Finally, the transient behavior of micro-fixed beds of circular and square geometries packed with particles of two different sizes were studied by monitoring the bed pressure drop variations upon step changes in liquid flow rate at iso-G conditions. Larger particle sizes and square geometry showed shorter transient times as compared to smaller particle sizes and circular geometry.

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1. Introduction

Miniaturized reactors currently serve as the basic tool for kinetic studies, synthesis and catalyst testing due to their high surface-to-volume ratios, enhanced heat transfer, safety, etc. This is especially the case for heterogeneously catalyzed highly exothermic reactions carried out in micro-fixed bed reactors [1–3]. Although the literature is rife with studies on micro-fixed beds [1,2,4–6] with the aim of enhancing their efficiencies in terms of conversion, mass transfer, etc., only a few of them went beyond and to the root cause, that is, the nature of contacting patterns between the phases [7–9]. This is what hydrodynamic studies attempt to address so that by adjusting the contacting scheme of the phases involved, multiphase reactions in micro-reactors could proceed toward more favorable conditions and performances [7]. Occasional examples of combining hydrodynamic analysis with mass transfer and reaction studies in micro-reactors can be found in the literature [10–12].

Possible research initiatives in this field come from two different aspects: (1) similarity between micro-fixed beds and macro-scale fixed beds (packed beds) from the operation point of view and (2) resemblance between micro-fixed beds and micro-channels from the scale perspective. The two approaches have major benefits; first, there are a number of key parameters concerning the hydrodynamics of both packed beds and micro-channels that could be suggestive to prospective investigations on micro-fixed beds (e.g., pressure drop, liquid holdup, residence time distribution (RTD), system geometry, etc.). Second, the appropriate method that could be adopted for measuring/determining the above parameters on micro-fixed beds can be decided by customizing the available methods used for packed beds [13,14] or micro-channels [15,16]. However, there is still a narrow band of knowledge on micro-fixed beds hydrodynamics as compared to existing information about other miniaturized flow devices due to the numerous challenges and difficulties that arise in the experiments. Description of the common technical challenges and measurement approaches in micro-fixed bed experiments has been addressed recently [17].

As for studies on micro-fixed beds in particular, Márquez et al. did RTD tests to determine a global liquid holdup in micro-fixed beds using non-volatile [8] and volatile [18] tracers. They also observed minor hysteresis in pressure drop and liquid holdup in micro-fixed beds [9]. Faridkhou and Larachi [7], on the other hand, reported a major hysteresis in pressure drop and wetting patterns by combining pressure drop measurements with microscopic wall visualizations at higher gas and liquid flow-rates as compared to Márquez et al. [9]. Flow regimes study was also carried out [7] by processing images taken from the wall region at different gas-to-liquid flow ratios and introducing a phase characteristic length as a measure to differentiate the two observed flow regimes.

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http://dx.doi.org/10.1016/j.cep.2014.02.005
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Near-wall RTD measurements were also performed by injecting a dye tracer at the entrance of the micro-fixed bed and monitoring variations in the gray level intensity of the images taken within a frame downstream of the injection point [17]. The results were in accordance with the theoretically predicted maximum velocity in the high porosity zone close to the wall [19].

To further extend knowledge about the hydrodynamics of micro-fixed beds, the current work studies the effect of channel shape and particle size (or tube-to-particle size ratio) on the bed pressure drop, flow regime transition point, hysteresis and transient behavior. Since the utilization of square tubes rather than round ones offers more compact systems, as shown in the case of heat exchangers [20], the comparison between the two geometries at micro-scale hydraulic diameters becomes appealing. Also, numerous works have been done on microchannel geometry and its effect on hydrodynamics [20–22] and heat transfer [23] while similar studies on micro-fixed beds is still lacking in the literature. Despite a number of researches carried out on the effect of particle size (or column-to-particle size ratio) on the hydrodynamics of packed beds [24–27], studies of such kind have yet to be performed in their micro-scale lookalikes and hence, will be subjected to investigation in this contribution. Finally, since micro-fixed beds are ultimately aimed for commercial applications, the study of their transient behavior (which is an industrially important parameter especially during equipment startups) and factors affecting it seems crucial.

2. Experimental

2.1. Setup

The schematic of the setup for performing the experiments is shown in Fig. 1. It consists of two separate transparent borosilicate tubes one with square ($W \times H = 1\,\text{mm} \times 1\,\text{mm}$, $L = 11.5\,\text{cm}$) and another with circular cross-section ($D = 1\,\text{mm}$, $L = 10.8\,\text{cm}$) used as micro-beds. The capillaries are inserted in carved Plexiglas blocks so that they could be protected against any inadvertent stroke. The tubes are packed with spherical glass beads with diameters in the ranges of either 106–125 μm or 55–63 μm (depending on the experiment) serving as the micro-packed bed. Both beds have optically accessible windows for microscopic visualization. Liquid (water, surface tension $\sigma = 0.0728\,\text{N/m}$) and gas (air) delivery to the micro-fixed bed are enabled by a syringe pump (Teledyne ISCO, 500D) and mass flow controller (EL-Flow Bronkhorst), respectively. Introduction of gas and liquid into the bed is such that their mixing takes place right at the start of the packed bed. Otherwise, the existence of any void space upstream of the micro-fixed bed bringing gas and liquid phases in contact prior to entering the packed section, will lead to instabilities and therefore, should be avoided [4,17]. The micro-fixed bed is mounted on an inverted microscope (Olympus GX-51) and visualized through a 10X objective lens (MPlanFLN-BD series, resolution: 1.12 μm) at a constant light intensity. The microscope is coupled with a camera (TSI, PowerView$^{\text{TM}}$ Plus) which is itself connected to a computer with a frame-grabber port and image acquisition is performed at a frequency of 15 Hz using Insight3G software. Pressure drop measurement in all the experiments is performed between the gas-feed line (inlet section) and bed outlet via a piezoelectric differential pressure transducer.

2.2. Methodology

To begin each set of experiments, the micro-beds were densely packed with glass beads. Particles could be easily loaded inside the micro-tube through a handmade tiny metal funnel within multiple steps in each of which a certain portion of the total bed volume is loaded with particles. Frequent tapping is required between the steps to completely compact the bed [17]. For the first part of flow regime transition experiments, only the square micro-fixed bed is used as it not only enables microscopic visualizations but also provides images with appropriate quality for subsequent processings due to its flat wall (the latter is not the case for circular geometry). Air superficial velocity is kept constant (21.3 cm/s) while water flows at different superficial velocities (12.8, 14.9, 17.0 and 19.2 mm/s) through the micro-fixed bed at room temperature. Upon reaching a constant pressure drop at each set of gas and liquid superficial velocities (indicating steady state conditions), a set of 50 images were taken at 15 Hz rate from each of the four regions within the bed as shown in Fig. 2. Two of the regions are located at the first half of the bed and the other two at the second half of it and imaging is performed successively from regions of one extreme to the other by turning the *x*-axis stage knob of the microscope for axial displacement through the micro-fixed bed. The aim of this part is to show how the flow regime transition starts sequentially within the bed length. The transition from low interaction to high interaction regime can be observed by microscopic visualization and graphically depicted via changes in the dimensionless liquid phase characteristic length ($\Lambda_{\text{L}}/d_{\text{p}}$) [7]. The characteristic length for the liquid phase is based on the weighted mean area it occupies on each image determined by image thresholding and binarization in MATLAB software [7,17]. A smooth evolution of the characteristic length with time indicates low interaction flow regime whereas abrupt jerks are signs for high interaction flow regime.

To study the effect of tube cross-sectional shape and particle size (or tube-to-particle diameter ratio) on pressure drop, flow regime transition point and bed dynamics, micro-fixed beds of both geometries were packed with glass beads of both size distributions and tested at different operating conditions according to Table 1. Upon increasing liquid flow from L_{i} to L_{j} (at iso-G conditions), steady state pressure drop was registered (at a frequency of 10 Hz) by first maintaining the liquid flow rate at L_{i} for 30 s and then switching to L_{j} until establishment of a new steady state. Meanwhile, microscopic visualizations from the near-inlet windows of both micro-fixed beds monitored the prevailing flow regimes within the bed. If the initial steady state pressure drop (for the initial liquid flow, L_{i}) and the final steady state pressure drop (for the final liquid flow, L_{j}) are named as ΔP_{i} and ΔP_{j}, respectively, then the transient time for the bed (as a measure of the bed dynamic behavior) is taken as the time when pressure drop values starts to exceed $\Delta P_{\text{i}} + \sigma_{\text{i}}$ and reach $\Delta P_{\text{j}} - \sigma_{\text{j}}$ where σ_{i} and σ_{j} are the standard deviations for pressure drop data at the initial and final steady states (designated as ΔP_{i} and ΔP_{j}), respectively. This has been shown schematically in Fig. 3a and b where the transient time is the time span between the blue and red bands representing the ascending part of the pressure drop variations versus time in Fig. 3b.

As for the study of wetting pattern hysteresis in micro-fixed beds, Faridkhou and Larachi [7] performed the experiments for $D/d_{\text{p}} \approx 10$ with particles in the range of 106–125 μm by taking wall-region images from the bed texture during both imbibition (increasing liquid flow rate) and drainage (decreasing liquid flow rate) paths within the low interaction flow regime. To investigate the effect of using smaller particle size (or increased tube-to-particle ratios) on the wetting patterns, particles in the range of 55–63 μm are used in the current work ($D/d_{\text{p}} \approx 20$). The gas superficial velocity is fixed at 25.5 cm/s while liquid superficial velocity is gradually increased from 1.1 mm/s to 13.8 mm/s and then decreased to its initial value. At each liquid velocity, for both imbibition and drainage paths, 150 images were taken from the bed after pressure drop stabilization in order to compare visualizations from the bed texture at same superficial liquid velocities in both

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