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Fluidization behavior in high-pressure water at temperature from ambient to supercritical

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ABSTRACT

The fluidization behaviors of Geldart-A and B particles in high pressure, sub-critical and supercritical water were investigated experimentally with the conditions of pressure up to 27.2 MPa and temperature up to 482 °C. Minimum fluidization velocity was determined by measuring pressure drop. Minimum bubbling velocity was obtained not only by analysis of the experimental differential pressure fluctuation signals, also by the relationship between bed voidage and superficial velocity. Correlations for the minimum fluidization velocity, homogeneous bed expansion rate and the minimum bubbling velocity were proposed. Transitions of fluidization regimes: fixed–homogeneous (F-H), fixed-homogeneous-bubbling (F-H-B) and fixed-bubbling (F-B) were uncovered at temperature from ambient to supercritical. The fluidization of F-H was found when discrimination number D_n is below 1.2×10^4, and the fluidization of F-H-B was found when discrimination number D_n is between 1.2×10^4 and 6×10^4, the fluidization of F-B was found when discrimination number D_n is above 6×10^4. These results are very useful for the design of the high pressure, sub-critical and supercritical water fluidized bed.

1. Introduciton

High temperature water (HTW), which is defined as liquid water above 200 °C and supercritical water (SCW, $T > 374$ °C, $P > 22.1$ MPa), is attracting attention as a medium for organic chemistry [1]. In HTW, organic wastes or biomass can convert to fuel, such as H_2 and CH_4, by hydrothermal reaction. Fuel generation by the hydrothermal reaction typically includes aqueous phase reforming, hydrothermal liquefaction and gasification. Hydrogen production by aqueous phase reforming of carbohydrate or alcohols typically occurs in liquid water at temperatures of 220–250 °C and pressures of 1.5–5 MPa [2]. Bio-oil can be produced by hydrothermal liquefaction in water with higher temperature and pressure (270–370 °C, 6–25 MPa) [3–4]. Clean gaseous fuels including CH_4, H_2 and CO are produced by catalytic wet gasification (in sub- and near-critical water 350–400 °C) and supercritical water gasification (500–750 °C) [5]. These chemical processes for fuels production have been demonstrated to be effective and promising.

One of the challenges for industrial applications of chemical technology of the hydrothermal reaction is the reliability of continuous running of reactor. Usually, tubular reactor is applied. However, some problems, such as plugging and loading catalyst, are difficult to solve by the tubular reactor. A fluidized bed reactor is typically used for coal gasification and combustion, drying agricultural particles, coating, catalytic cracking of crude oil, and washing and roasting mineral materials etc. Recently, a fluidized bed has proved to be a reliable reactor for continuously gasifying biomass in supercritical water for hydrogen production [6–7]. The reactor could avoid plugging, increase the hydrogen yield and improve gasification efficiency. Because of lower operation temperature and pressure, the fluidized bed as an effective chemical reactor is used more easily in the aqueous phase reforming and hydrothermal liquefaction.

The design and operation of the fluidized bed reactor are greatly depending on the understanding of the two-phase flow characteristics in the fluidized bed. The two-phase flow structures in the reactor greatly affect the chemical reaction. The determination of flow patterns of the fluidized bed reactor will help to keep the chemical reaction under suitable conditions and avoid bad mixing of the solid and fluid phases. The two-phase flow characteristics of water-solid fluidized bed in ambient conditions have been studied widely [8–10]. The evolution of flow patterns of the water-solid fluidized bed with increasing superficial velocity is from fixed bed, to homogeneous bed expansion, to turbulence zone, and then to transport zone. However, for the fluidized bed in near-critical and supercritical zone, the flow patterns and bed expansion characteristics are still unclear.

Tarmy et al. studied the three-phase flow characteristics of fluidized beds under a pressure of 17 MPa and temperature of 450 °C [11]. Jiang et al. investigated bed contraction and expansion in a gas–liquid–solid fluidized bed at pressures from 0.1 to 17.4 MPa and temperatures from 20 to 94 °C [12]. Liu et al. experimentally studied the fluidization of Geldart

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Group A, B and D particles in CO_2 under ambient to supercritical conditions [13]. They found that fluidization of the supercritical CO_2 fluidized bed was intermediate between the classical aggregative (gas-solid system) and particulate (liquid-solid system) fluidizations. Marzocchella and Salatino fluidized Geldart Group A and B particles by CO_2 at temperature of 35 °C with pressure ranging from1 to 8 MPa [14]. The fluidization regimes of fixed bed, homogeneous bed expansion, bubbling zone, and turbulent regime were mapped. Vogt et al. investigated the fluidization behaviors of supercritical CO_2 fluidized bed with pressure up to 30 MPa [15]. They fitted the correlations for the minimum fluidization velocity and bed expansion. Besides, they suggested a method to determine the minimum bubbling velocity and measured the bubble size and visible bubble flow. They provided a comprehensive picture for the supercritical CO_2 fluidized bed when the superficial velocity was not very high. Due to the difference between supercritical CO_2 and water, the research results of the supercritical CO_2 fluidized bed are hard to guide the design of near-critical and supercritical water fluidized bed. Potic et al. studied the fluidization process of Geldart Group A particles in high pressure water within a cylindrical quartz reactor with an internal diameter of 1.0 mm in the range of 0.1–24.4 MPa and 20–500 °C. Homogeneous bed expansion was observed for the typical conditions of 300–320 °C and 16 MPa, and slugging fluidization was observed for typical conditions of 370 °C and 16 MPa. In this work, the authors studied visually the fluidization process in high pressure and high temperature [16]. However, the fluidization regime, such as slugging, may be also induced by the small ratio of inner diameter of the fluidized bed to the particle diameter, which restricted the universality of the conclusion of the work. In our previous work, we measured the pressure drops of a SCW fluidized bed with a diameter of 35 mm for temperature ranging from 360 °C to 420 °C and pressure ranging from 23 MPa to 27 MPa [17]. It was found that Ergun equation was suitable for calculating the fixed bed pressure drop. Although this work solved the problem of onset fluidization in supercritical zone, the experimental range was narrow and the bed expansion or flow patterns evolution were not involved.

Few numerical investigations studied bed expansion process within the SCW fluidized bed. Wei et al. simulated the feeding methods of a SCW fluidized bed reactor based on the Eulerian two-flow model incorporating particle kinetic theory [18]. Bubbling phenomenon of Geldart Group B particles was observed in the simulation results. More recently, Lu et al. studied the fluidization process of Geldart Group B particles in SCW by a CFD-DEM model. Fluidization transition of fixed–homogeneous-bubbling zone was observed by the simulation work [19–20]. Particle-scale investigations including the flow separation and heat transfer process from a spherical particle to the SCW flow were conducted numerically [21–22]. The results show that the decrease in density and viscosity of water in supercritical zone leaded a decreasing particle drag, which may be the reason for the transitions from homogeneous to bubbling.

To the best of our knowledge, no experimental investigations have been conducted to the bed expansion and flow patterns in a near-critical or supercritical water fluidized bed. In this paper, we investigated experimentally on a fluidization behavior of Geldard-B and Geldard-A particles in a high-pressure fluidized bed at the temperature ranging from ambient to supercritical zone. The flow patterns and their boundaries of the fluidized bed were obtained, and the correlations of the minimum fluidization velocity, homogenous bed expansion rate and minimum bubbling velocity of a fluidized bed were also proposed based on the experimental data.

1. Experimental apparatus and process

Fig. 1a displays the schematic diagram of the experimental setup of the fluidized bed. The setup consists of a fluidized bed test section and other additional elements including a tank, two high-pressure pumps, a damper, two heat regenerators, an electric heater, bypass pipes, two coolers, two regulating valves, two mass flow meters, a back pressure valve and a cooling system. The fluidized bed section is made of stainless steel with 1.2 m height and 35 mm inner diameter. It is designed for the temperature up to 550 °C and the pressure up to 30 MPa. A porous metal foam with bore diameter of 30 μm is used as the distributor of the fluidized bed test section, and a metal foam filter is installed in the exit of the fluidized bed to avoid the escape of the bed materials. The temperature of fluidized bed is measured by two type K thermocouples in good thermal contact with the inlet and outlet of the fluidized bed. The temperature difference between inlet and outlet can be <1 K because of good thermal insulation of the test section. Therefore, the average of the two measured temperatures is used as the bed temperature.

The mass flow rates of the fluidized bed and bypass pipe are measured by two mass flow meters (Siemens, Germany; Rhoenik, Germany). The present system provides mass flow rate in a wider range of up to 250 $kg \cdot h^{-1}$. The superficial velocity can be determined by the mass flow rate, water density and geometry of the test section. The properties of water are calculated by ISAWP-IF97 equations based on the operating temperature and pressure [23]. Deionized water is pumped from the tank to the damper, heat regenerator I and II and the electric heater, in turn. Then, the water is divided into two parts, one passing through the fluidized bed and the other getting into the bypass pipe. The control method of mass flow is different from our previous work [17], in which the mass flow rate was changed by adjusting the flow rate of the pump. It will lead to a variation of temperature in the inlet of fluidized bed because of a limited heat power of the electric heater. In present work, the flow mass changes in fluidized bed by adjusting the mass flow of bypass pipe and keeping the pump flow steady. By using the regulating valves, a linear increase or decrease in flow into the fluidized bed can be achieved without regulating the pump flow, and the temperature and pressure of the inlet of fluidized bed can be kept constant.

Absolute pressure (AP) signal is measured by a pressure sensor (Rosemount, USA). Differential pressure (DP) ($\triangle P1$, $\triangle P2$, $\triangle P3$, and $\triangle P4$) are measured using four differential pressure transducers with high response frequency (Foxboro, USA), and the locations of the transducers are shown in the Fig. 1b. The locations of the four pressure transducers are kept flush with the top of the reactor. Pressure line was fully exposed to the ambient air. The fluctuation signals of DP are measured with a sampling frequency of 1000 Hz, and sampling number of 60,000. Signals from the DP transducers were recorded by a data acquisition card (NI CompactDAQ-8174, USA) data and stored in a computer. The frequency of pressure differences signals in a fluidized bed is not high. Johnsson et al. used 20 Hz as cut off frequency when analyzing pressure difference signals. The signals of >20 Hz in frequency could be considered as noise [24]. According to a detailed investigation on delay time correlations of pressure fluctuation signals by Zhao et al., the signals of >30 Hz in frequency are not of linear correlation. The signals of >40 Hz in frequency are not of nonlinear correlation, which could be considered as noise for no correction in any time interval [25]. To be safe, 40 Hz as the filtered frequency was used to remove the effect of noise signals in this paper. The measured pressure difference between the pressure ports includes two parts: the frictional pressure drop and supercritical water's gravity pressure drop. The bed frictional pressure drop should equal the measured differential pressure minus the fluid gravitational pressure drop. Average bed voidage of fluidized bed can be evaluated by the pressure drops of $\triangle P1$, $\triangle P2$ and $\triangle P3$. The bed height for the bed is determined by measuring the differential pressures, and the details can be found in the literature [15,17].

We used quartz sand with two different sizes as bed materials. The real density of quartz sand is 2650 $kg \cdot m^{-3}$. The volumetric equivalent diameters of the quartz sand were measured by a Malvern laser particle size analyzer (Malvern Spraytec 300). Fig. 2 displays the diameter distribution of the bed materials. The average volumetric equivalent diameters of the particles are 65 μm and187 μm, which can be classified as Geldart A and B particles, respectively. The experimental study was conducted at the conditions of pressure up to 27.2 MPa and temperature up

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