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Characterizing Electrostatic Charges in High-Pressure Gas-Solid Fluidized Beds: Experimental Design and Preliminary Results

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ABSTRACT

Electrostatic charges in gas-solid fluidized beds such as polyethylene production reactors cause reactor wall fouling. An experimental technique to examine this phenomenon, described previously (1-5), was extended to a pilot-scale system designed for higher pressures (25 atm) and temperatures (100 °C) that approach industrial conditions. This work presents the experimental setup and preliminary results.

INTRODUCTION

Electrostatic charge generation in gas-solid fluidized beds is a long-standing problem. For instance, in polyethylene production reactors, electrostatic charge generation causes the adherence of a layer of particles on the wall or the dome of the reactor (6-9). If the particle layer is sufficiently large, it drops onto the distributor plate and partially blocks it, causing defluidization and necessitating reactor shutdown for cleanup. Commercial gas-solid fluidized bed reactors, such as those of polyethylene, operate at higher pressures of 7 to 20 bar (10). Under such conditions, bed hydrodynamics (e.g., gas bubble size and rise velocity) and thus solids mixing differs from those under atmospheric conditions. Since the study of electrostatics is dependent on the particles mixing and their interactions with the reactor wall, it is essential to investigate this phenomenon under industrial conditions. However, the majority of works found in open literature, with the exception of very few (11-13), investigated the electrostatic phenomenon under atmospheric conditions. Moughrabiah et al. (11) and Liu et al. (12) used electrostatic probes and determined the local particle charge in various locations within a fluidized bed operated at pressures up to 10 bar. Moughrabiah et al. (11) concluded that increasing the pressure from 1 to 8 bar intensified the bed electrification, whereas Liu et al. (12) found no clear effect of pressure on particles charging. In these works, due to the usage of electrostatic probes and thus very local charge measurements, no information on the influence of operating pressure on the degree of reactor wall fouling and the net charging of the particles in this layer was provided. In our previous works (1-5) a novel measurement technique was developed and successfully implemented in an atmospheric fluidized bed. The method, consisting of two removable Faraday cups, enabled the quantification of the degree of reactor wall

fouling, and provided information on particle charging simultaneously in three key areas of a fluidized bed: the bed particles, those adhered to the column wall, and those entrained. Given the success of this measurement technique, a new pilot plant fluidization system was built and commissioned that houses the same charge measurement technique and is able to operate at high-pressures of up to 25 atm (similar to those of commercial reactors such as polyethylene production). The present work discusses the newly commissioned high-pressure system and provides some preliminary results.

EXPERIMENTAL SETUP AND PROCEDURE

The high-pressure pilot plant fluidization facility as illustrated in Figure 1 consists of a three-dimensional gas-solid fluidization column 0.15 m in diameter and 4 m in total height, with a 2.5 m fluidization section. The two expanded sections at the top and bottom of the column are 0.34 m in diameter. The entire system is made of stainless steel and designed to be operated at pressures up to 25 atm.

The same charge measurement technique as detailed in Sowinski et al. (1, 2) was implemented where two copper Faraday cups were placed inside the top and bottom expanded sections of the column, each with the diameter of 0.254 m. Both cups are electrically isolated from the fluidization column which is grounded. The top Faraday cup houses a filter bag that is attached to the extension of the fluidization section of the column and used to capture the entrained fines for their charge to be measured by the Faraday cup while allowing the fluidizing gas to exit the system. Both cups are directly connected to Keithley digital electrometers Model 6514 to measure the cumulative charge of entrained particles throughout the fluidization period, and the net charge of the particles adhered to the column wall and those in the bulk of the bed upon the completion of a fluidization run. A commercially available pneumatic knife gate valve, 0.15 m in diameter and supplied by Orbinox Canada (QC, Canada), was modified and used as a perforated removable distributor plate to allow the discharge of the bed particles into the bottom Faraday cup. A stainless steel screen was mounted at the bottom of the valve blade to prevent any passage of fine particles. In order to enable removal of Faraday cups and the filter bag for measuring the particles mass and examining the extent of the reactor wall fouling; a mechanism was required to allow the frequent opening and closing of the top and bottom of the fluidization column. Since the fluidization column was designed for high pressures of up to 25 atm, a special mechanism was required which was achieved by replacing the top and the bottom flanges of the column with commercially available high pressure doors, referred to as manways or quick closures. The manways were supplied by GD Engineering (UK).

The fluidization system enables two modes of operation; once through and closed loop circulation. Under once through mode, the system can be operated from atmospheric to the maximum source pressure of the fluidizing gas (e.g., building compressed air). The closed loop circulation mode allows the usage of any type of fluidizing gas (e.g., nitrogen) from a gas cylinder, and thus operating pressures of up to 25 atm. A variable speed centrifugal compressor supplied by FIMA (Germany) allows the recirculation of fluidization gas in the system for operations from 5 to 25 atm. The fluidizing gas volumetric flow is measured and controlled by a Brooks (MN, USA) mass flow controller and a Rosemount (PA, USA) vortex meter. The room and

the fluidizing gas relative humidity (RH) and temperature are monitored by two Vaisala (USA) relative humidity transmitters. The system pressure and also pressure drop across the bed are monitored by ABB (UK) gauge and differential pressure transducers.

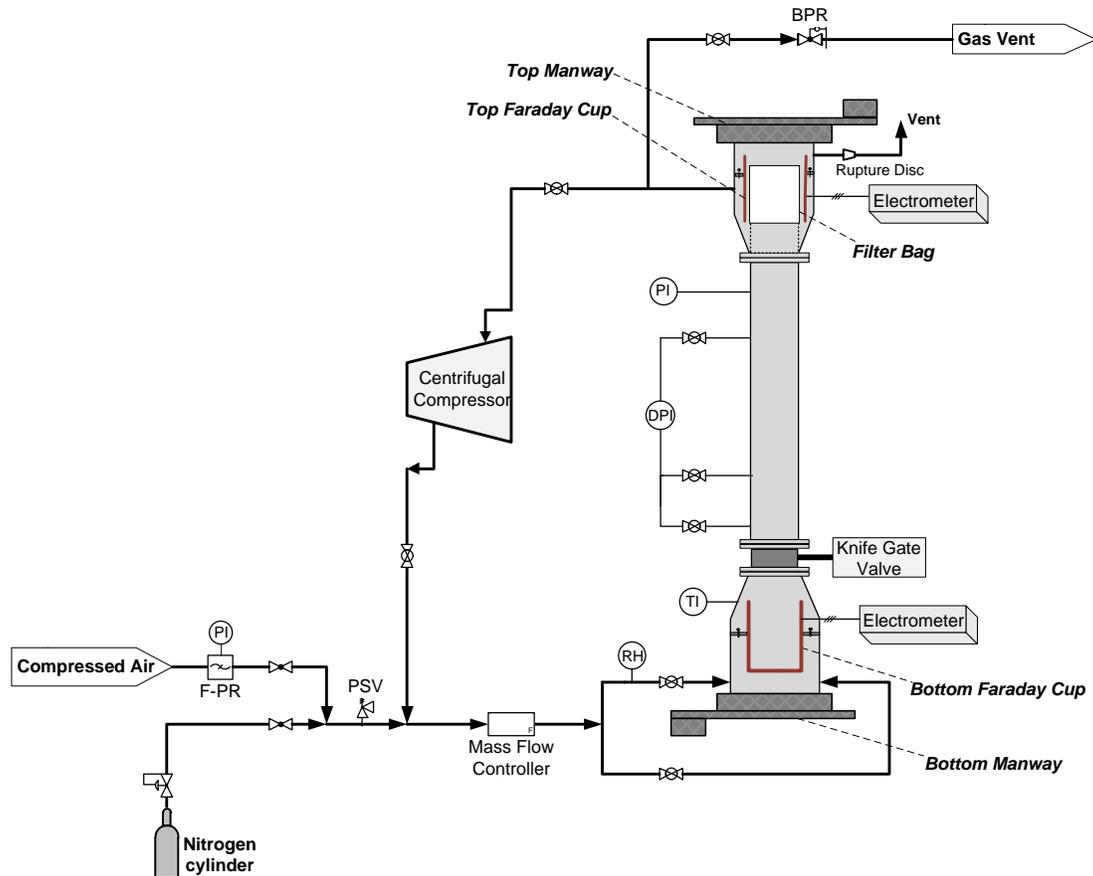


Figure 1. Schematic diagram of the high-pressure (up to 25 atm) fluidization system. PI (pressure indicator-pressure gauge); DPI (differential pressure indicator-pressure transducer); TI (temperature indicator-thermocouple); RH (relative humidity meter); PSV (pressure safety valve); BPR (back pressure regulator); F-PR (filter & pressure regulator).

The preliminary testing of the system was conducted by fluidization of a polyethylene resin produced using metallocene catalyst in an industrial gas-solid fluidized bed reactor. The resin had a particle density of 918 kg/m^3 and a wide size distribution of 20-2000 micron with an average particle size of approximately 700 micron. Compressed building air at 23°C and 3% RH was used as the fluidizing gas. Preliminary experiments were performed by running the system under once through mode at two operating pressure conditions of atmospheric and 4 atm. The high pressure condition was achieved by controlling the column pressure by the back pressure regulator located at the exit of the fluidization column. For both conditions, the fluidizing gas velocity was set at 1.25 times the minimum fluidization velocity (U_{mf}) to ensure bubbling flow regime. Experiments were repeated three times to confirm reproducibility. For all trials the bed height to the column diameter ratio (L/D) was held constant at 1.3, and the fluidization was conducted for 1 hour.

For each run the initial charge of the particles was measured by an external Faraday cup followed by placing the particles inside the fluidization column and mounting the filter bag at the top of the column. During the fluidization period, the charge of any fines entrained was measured by the top Faraday cup. Upon the completion of the fluidization period, the top manway was opened and the filter bag was removed for the entrained particles (referred to as “fines”) mass to be measured. This followed by opening the knife gate valve to allow the bulk of the bed (referred to as “dropped”) to discharge into the bottom Faraday cup for its net charge to be measured by the electrometer and recorded. The bottom manway was then opened to remove the bottom Faraday cup to measure the collected particles mass and to inspect the inner column wall for any fouling (i.e., pictures to be taken). The bottom Faraday cup was then placed back to allow the collection of the particles adhered to the column wall (referred to as “wall-1”). These particles were dislodged from the column wall by blowing air through a small stainless tube, connected to a dry air supply and inserted into the column from the top. The bottom cup was then removed for the collected particles mass to be measured and images to be taken from the inner column wall again. Through some preliminary testing at 4 atm, it was determined that the last layer of particles attached to the column wall had an opposite polarity to those forming the outer layer. Thus, these particles removal was conducted in two steps where the same procedure as described for wall-1 particles was repeated. The second batch of particles was referred to as “wall-2”. For all trials, a significantly thin layer of particles were left on the column wall as their removal was not possible by the aforementioned procedure. These particles were referred to as “wall-3”. Although samples of these particles were taken for their particle size distribution analysis, but their charge remains unknown. Since the column could be opened at the end of the each trial, it enabled the inner column wall to be thoroughly cleaned in between trials. All data collection and control was conducted by LabVIEW software.

RESULTS

The charge measurement technique developed in our earlier works ([1](#), [2](#)) was successfully implemented in the new custom-made high-pressure fluidization infrastructure. The inclusion of manways easily enabled accessing the inside of the fluidization column. Preliminary free bubbling fluidization experiments were performed under atmospheric conditions and at higher pressures of 4 atm. The average net charge-to-mass ratio (q/m) of initial particles was measured to be $-0.03 \pm 0.02 \mu\text{C}$, indicating that all trials were initiated with similar q/m values. Although initially the resin had a net negative charge, its exact charge distribution was determined by a charged particle separator apparatus, built in-house and detailed elsewhere ([14](#)). The resin initially consisted of both positively and negatively charged particles with specific charges found to be $17 \pm 1 \mu\text{C/kg}$ and $-17 \pm 1 \mu\text{C/kg}$, respectively.

A minute amount of entrainment was obtained during the fluidization, thus no charge results are discussed here for these particles. For all trials, under both operating conditions, the dropped particles and those adhered to the column wall occupied almost 95-97% and 2-4% by mass of the resin initially placed inside the fluidization column, respectively. Figure 2 illustrates the effect of system pressure on the magnitude of the reactor wall fouling. Elevating the pressure by only 4 atm, almost

doubled the magnitude of the particles adhered to the column wall (i.e., wall-1 plus wall-2).

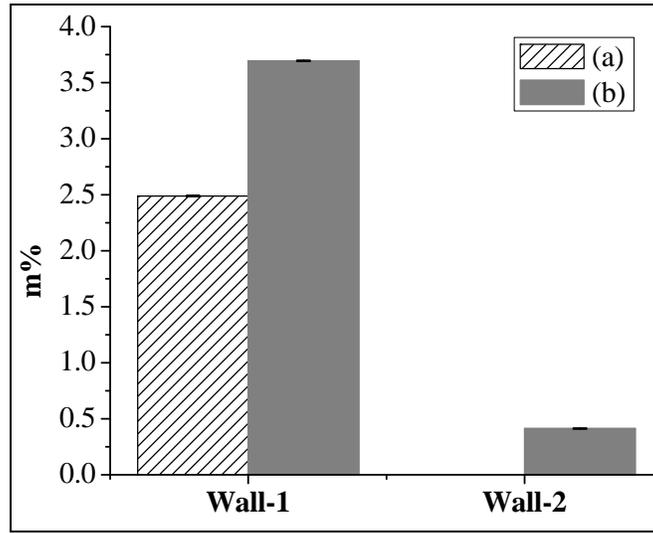


Figure 2. Results of the mass percentage of particles collected off of the column wall. (a) Atmospheric; (b) 4 atm.

The net q/m of particles collected from various regions of the fluidization column is presented in Figure 3. The dropped particles were dominantly positively charged with quite a low net specific charge, similar to that of the initial particles. At 4 atm, the wall coating consisted of two layers of wall-1 and wall-2 that were mainly positively and negatively charged, respectively. The net specific charge of the wall-1 particles, under two operating pressures, was found to be comparable, although their masses were different. Wall-2 particles were highly charged with an absolute value almost three times larger than wall-1 particles. The elevation of the particles mass under 4 atm operating condition might have been due to the presence of the highly negatively charged particle layer on the wall, the attraction to which constituted a driving force for the positively charged bulk particles to migrate to the wall region and thus resulting in a larger wall layer mass. It is significant to note that the specific charges of both wall-1 and wall-2 are much higher than those of the positive and negative portions of the initial particles, suggesting that the wall particles charge was acquired during fluidization and was not merely the result of the segregation of initially and oppositely charged particles.

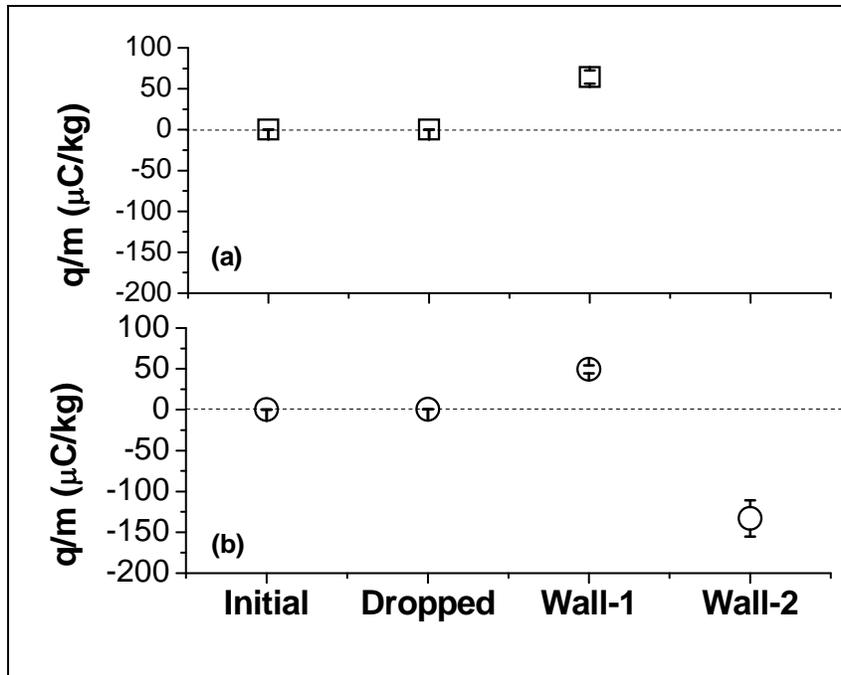


Figure 3. Results of the net charge-to-mass ratio of the initial, dropped, wall-1 and wall-2 particles. (a) Atmospheric; (b) 4 atm.

The degree of column wall fouling was also examined qualitatively by obtaining images of particle wall coating. Sample images, in Figure 4, show the inside of the column before and after the removal of the wall layer.

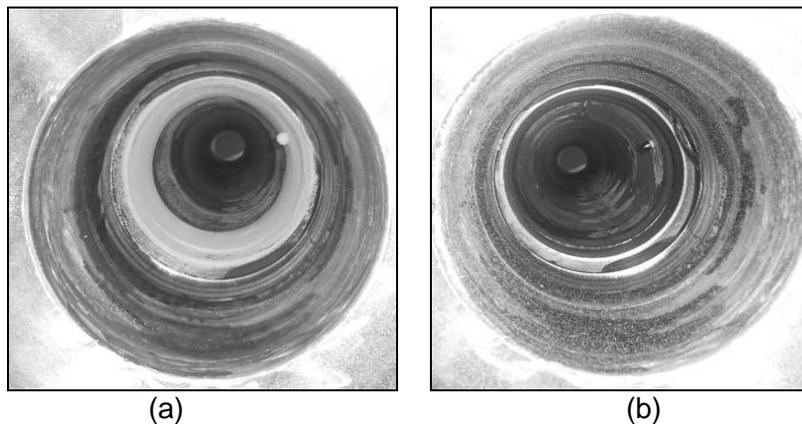


Figure 4. Images of the wall layer coating before and after their removal at atmospheric condition. (a) Prior to wall-1 particle removal; (b) After wall-1 particle removal.

Samples taken from dropped, wall-1, wall-2 and wall-3 particles were analyzed for their particle size distribution. As can be seen in Figure 5, particles adhered to the column wall were smaller than those in the bulk of the bed, with wall-3 particles being the smallest. This indicated that the particle size decreased in the radial direction from the center of the bed to the column.

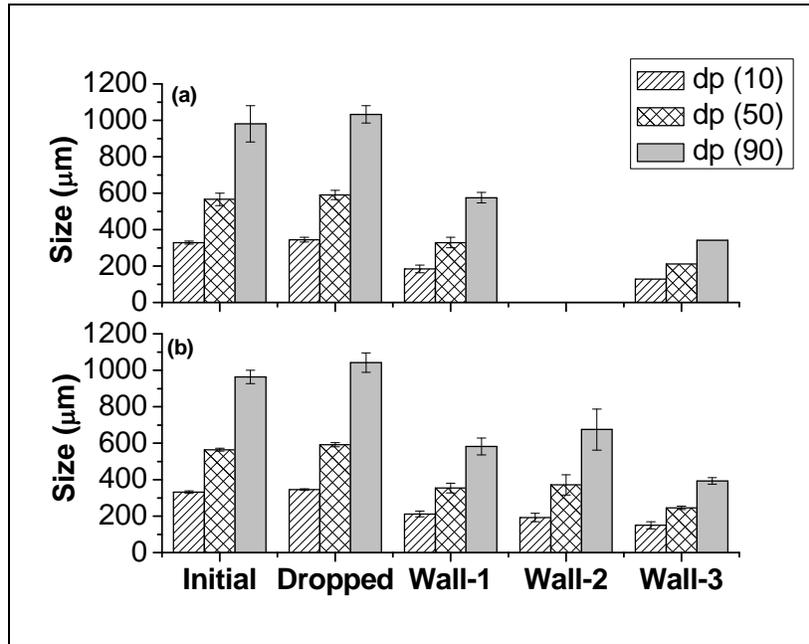


Figure 5. Particle size distribution (dp_{10} , dp_{50} , dp_{90}) in different regions of the fluidized bed. (a) Atmospheric; (b) 4 atm.

CONCLUSIONS

A pilot-scale experimental unit designed for operating under higher pressures (up to 25 atm) and temperatures (up to 100 °C) was used to examine wall fouling in the fluidization of polyethylene resin by air. The new system successfully allowed the measurement of the mass, charge, particle size distribution, as well as the spatial distribution of the fouling under high operating pressures. Comparing atmospheric and 4 atm operating conditions, it was found that at the higher pressure there was a substantial increase in the mass of the wall fouling layer. The wall layer at high pressure consisted mainly of positive particles but also included highly negatively charged and smaller particles. Future work will include a comprehensive experimental program to investigate the extent of reactor fouling at various pressures and temperatures.

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