A new sampling device for measuring solids hold-up in a three-phase system

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Measurement of solids hold-up in gas-liquid-solid systems was achieved by using a cylinder with a valve in each extremity, which allowed direct sampling inside the reactor causing minimal disturbances to the fluid flow. The method was developed and tested in a 60 L concentric draught tube airlift reactor. Validation of the method was done by means of an overall balance to the solid phase in the bioreactor.

Notation

Q = airflow rate (L/min)
S = sample points
V_s = solids volume in the sample (mL)
V_total = total sample volume (mL)

Greek letters

\( \epsilon_s \) = solids hold-up in one section of the reactor
\( \epsilon_o \) = overall solids hold-up

Introduction

Three-phase reactors (fluidized beds and slurry reactors) have been extensively used in chemical industries on gas-liquid-solid catalytic processes. Systems using immobilized cells are receiving increasing attention, both in biochemical industries and in waste water treatment plants (Siegel et al., 1988). Airlift reactors have some advantages when operating with three-phase systems such as the elimination of dead zones, an increase in the maximum solids hold-up and lower gas flow for the complete fluidization of the solids. Nevertheless, only a few studies have been made with three-phase airlift reactors (Verlaan and Tramper, 1987; Smith and Skidmore, 1990; Kennard and Janeke, 1991; Livingston and Zhang, 1993; Lu et al., 1995; Vicente and Teixeira, 1995; Kawase and Hashimoto, 1996) and most of them do not make reference to the solids hold-up, due to measurement difficulties. Since solids hold-up has a great influence on the hydrodynamic behaviour and on mass transfer in three-phase airlift reactors, especially those operating at high cellular densities, it is necessary to develop systems that allow its correct determination. Three-phase systems are routinely sampled through a small port on the side wall of the reactor. Side wall sampling is known to give erroneous measurement of concentration and particle size distribution for all but the smallest particles having densities close to that of the suspending fluid (Wenge et al., 1995). As particles have to change direction to get into the sample port, small particles (with lower inertia) are sampled preferentially. Because of that, the sample has more liquid and small particles than the bulk flow and does not give the real value of the solids hold-up.

To avoid problems associated with side wall sampling, a device was developed to sample directly inside the reactor without disturbing the fluid flow. As a test, solids hold-up was determined in different parts of an airlift reactor working with 15% (v/v) of solids, for different airflow rates (5.5, 9.1, 30.3 and 90.2 L/min).

Materials and methods

The reactor

Measurements were done in a 60 L airlift reactor of the concentric draught tube type, with an enlarged degassing zone, made of plexiglass with a thickness of 8 mm (Fig. 1). The total height of the reactor was 1.986 m and the fluid level was 0.530 m above the top of the draught tube. The height and inside diameter of the downcomer were 1.190 m and 0.142 m, respectively. The draught tube height and inside diameter were, respectively, 1.190 m and 0.062 m and its bottom edge was 0.086 m above the bottom of the reactor.

The top section was of the cylindrical conical type. The angle of the conical sector with the main body of the reactor was of 51° and the height and diameter of the cylindrical part were, respectively, 0.350 m and 0.442 m.
Air injection was made 0.061 m below the bottom of the draught tube by means of a circular plate with a diameter of 0.030 m, with 30 holes of 1 mm each.

Ca-alginate beads, with a density of $1038 \pm 1$ kg/m$^3$ and a diameter of $2.151 \pm 0.125$ mm, were used as the solid phase while water was used as the liquid phase.

The sampler
The sampler consisted of a 60.0 mL cylinder with two valves, one at each end. The height and diameter of the sampler were 97.5 mm and 28.0 mm, respectively. The sampling cylinder (A) was connected to a long rod (B) that permitted sampling at different depths in the reactor. Along this rod there was a rigid wire (C) that was used to act on two lock springs that controlled the closing of the sampling unit. When the sampler was introduced into the reactor (Fig. 2a), the valves were kept open in the flow direction and closed simultaneously for sample collection (Fig. 2b). In each sample, retained solids volume ($V_s$) was measured. Knowing the sample (solids, liquid and gas) total volume ($V_{total}$ = 60.0 mL), the solids hold-up ($\epsilon_s$) was given by the following equation:

$$\epsilon_s = \frac{V_s}{V_{total}}$$

Experimental procedure
The reactor was filled with water and solids (15% v/v) and the airflow rate was adjusted to the desired value (5.5, 9.1, 30.3 and 90.2 L/min).

The gas-liquid-solid slurry was sampled at eight different points, each one representing a different section of the reactor (Fig. 1). For each set of experimental conditions, three samples were taken per sampling point.

Results and discussion
To characterize the distribution of the solid phase throughout the reactor, samples were taken in eight different points of the reactor. Each sample point was representative of a section of the reactor, with volumes presented in Table 1. The degassing zone was divided...
Table 1 Volumes of the different sampling sections (S1 to S8) of the airlift reactor

<table>
<thead>
<tr>
<th>Section</th>
<th>Volume (L)</th>
</tr>
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<tbody>
<tr>
<td>1</td>
<td>14.70</td>
</tr>
<tr>
<td>2</td>
<td>8.69</td>
</tr>
<tr>
<td>3</td>
<td>2.69</td>
</tr>
<tr>
<td>4</td>
<td>9.12</td>
</tr>
<tr>
<td>5</td>
<td>2.53</td>
</tr>
<tr>
<td>6</td>
<td>3.17</td>
</tr>
<tr>
<td>7</td>
<td>14.27</td>
</tr>
<tr>
<td>8</td>
<td>3.59</td>
</tr>
</tbody>
</table>

Into five sections (S1 to S5), Section S8 being a transition between the degassing zone and both the downcomer (S7) and the riser (S6).

Solids hold-up in each section of the reactor is shown in Fig. 3, plotted versus the airflow rate. An axial and radial distribution of the solid phase in the degassing zone is shown. Solids hold-up increases from the wall to the middle (from S1 and S6 to S2 and S5, respectively) and from the top to the bottom (from S3 and S4 to S4 and S3, respectively). Also, solids hold-up values in the degassing zone (S1 to S4) are lower than in both the downcomer (S7) and the riser (S6), demonstrating the efficiency of the degassing zone while acting as a solids settler. This characteristic is of major importance to the operation of continuous high cell density bio-reactors, where a good biomass retention is desired. In Section S8, solids hold-up is similar to those found in the riser and downcomer.

It can also be seen from Fig. 3 that, for lower values of airflow rate, while with an increase in airflow rate, solids hold-up decreases both in the downcomer (S7) and in the riser (S6), in the majority of the sections of the degassing zone (S1 to S4) solids hold-up increases. In Sections S3 and S5, there is a decrease as those observed in the riser and in the downcomer. This corresponds to the expected behaviour because, when the airflow rate is low, solids cannot reach the top of the reactor and the concentration of solids in the riser (S6) is lower than in Sections S3 and S5. After they leave the riser, the particles go upwards to Section S6 and only a few of them reach the top of the degassing zone (S1). Increasing the airflow rate, increases the rising force. Solids rise in higher amounts until Section S1, and, on their way down, they pass through Sections S4, S5, and S6, increasing the solids hold-up in these sections. Consequently, solids hold-up in the other sections of the reactor decreases. For higher values of the airflow rate, its influence on solids hold-up is very little and the distribution of the solid phase in the reactor is more homogeneous.

To confirm the validity of the method, a balance on the solid phase was made without considering the bottom of the reactor, since its volume is negligible when compared with the total volume of the reactor. Values of the measured overall solids hold-up (εs), for each airflow rate studied, are shown in Table 2. Those values present relative errors ranging from 0.5% to 5% when compared with the volumetric fraction of the solids in the total volume of the reactor (15%). These low relative errors confirm the validity of the method and are within the expected, suggesting that the airflow rate has no systematic influence on the magnitude of the experimental error.

Conclusions

A new method for the measurement of solids hold-up in gas-liquid-solid systems was developed. The method consists in sampling the slurry directly inside the reactor without disturbing the fluid flow, using a device specially developed for that purpose.

Results of laboratory experiments show that the method is sensitive to changes in the solids concentrations occurring in the different sections of the reactor, for a
Table 2 Measured overall solids hold-up (ε_s) and the corresponding relative error, for each airflow rate

<table>
<thead>
<tr>
<th>Q (L/min)</th>
<th>ε_s (% v/v)</th>
<th>Relative error (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5.5</td>
<td>15.1</td>
<td>0.5</td>
</tr>
<tr>
<td>9.1</td>
<td>14.4</td>
<td>4</td>
</tr>
<tr>
<td>30.3</td>
<td>15.8</td>
<td>5</td>
</tr>
<tr>
<td>90.2</td>
<td>15.1</td>
<td>0.6</td>
</tr>
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</table>

range of airflow rates. The validation of the method was done by means of a balance to the solid phase.

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References

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