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

Carla Freitas, António A. Vicente, Manuel Mota and José A. Teixeira*

I.B.Q.F. (Instituto de Biotecnologia e Química Fina), Departamento de Engenharia Biológica, Universidade do Minho, Campus de Gualtar, 4709 Braga codex, Portugal

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_j	solids volume in the sample (mL)
V_{sample}	total sample volume (mL)

Greek letters

ϵ_j	solids hold-up in one section of the reactor
ϵ_{st}	overall solids hold-up

Introduction

Three-phase reactors (fluidized beads 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 Janekeh, 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 hydro-dynamic 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.
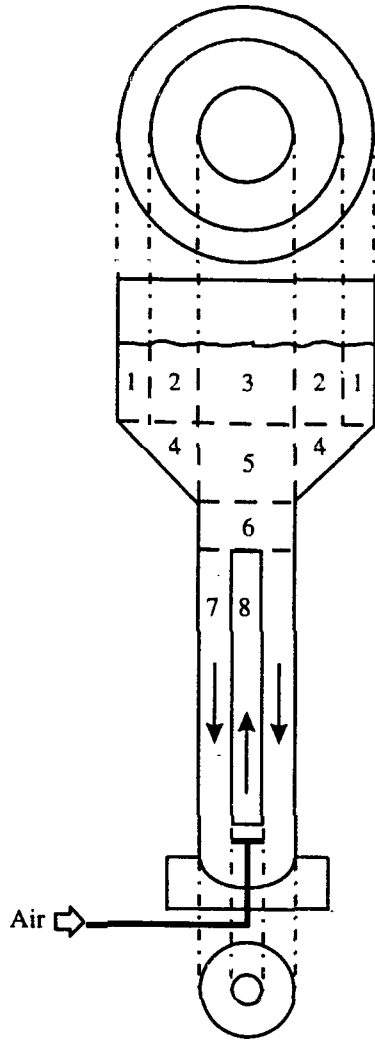
Figure 1 Schematic diagram of the airlift reactor. The reactor is divided in eight different sections for sampling purposes (Sections S_1 to S_5 correspond to the degassing zone and Section S_6 is a transition section between the downcomer – S_7 – and the riser – S_8). The arrows indicate the fluid flux.

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 ± 1 kg/m³ and a diameter of 2.151 ± 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
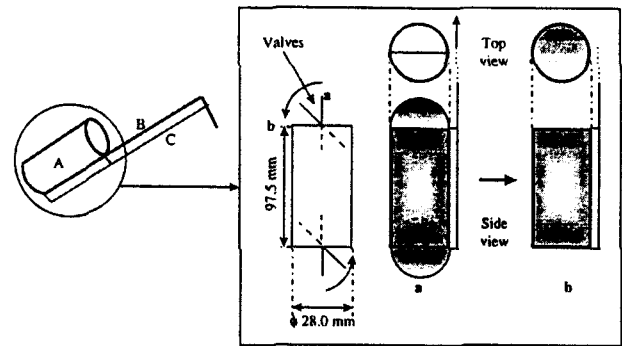

Figure 2 The sampler (A – sampler body; B – rod; C – trigger wire). Sampling is achieved by closing two valves that switch from position **a** (valves open) to position **b** (valves closed).

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 $(V_{sample} = 60.0$ mL), the solids hold-up (ϵ_s) was given by the following equation:

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

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 (S_1 to S_8) of the airlift reactor

Section	Volume (L)
1	14.70
2	8.69
3	2.69
4	9.12
5	2.53
6	3.17
7	14.27
8	3.59

Into five sections (S_1 to S_5), Section S_6 being a transition between the degassing zone and both the downcomer (S_7) and the riser (S_8).

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 S_1 and S_4 to S_3 and S_5, respectively) and from the top to the bottom (from S_1 and S_3 to S_4 and S_5, respectively). Also, solids hold-up values in the degassing zone (S_1 to S_5) are lower than in both the downcomer (S_7) and the riser (S_8), demonstrating the efficiency of the degassing zone while acting as a
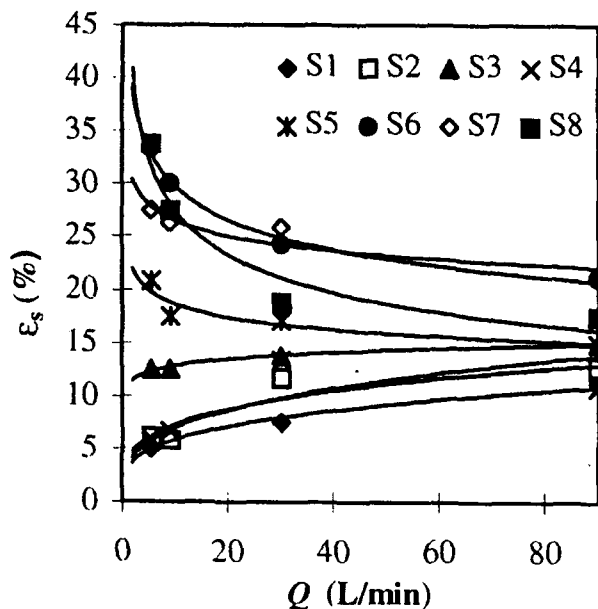

Figure 3 Solids hold-up (ϵ_s) in different sections of the airlift reactor *versus* airflow rate (Q).

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 S_6, 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 (S_7) and in the riser (S_8), in the majority of the sections of the degassing zone (S_1 to S_4) solids hold-up *increases*. In Sections S_5 and S_6 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 (S_8), downcomer (S_7) and in Sections S_5 and S_6 is higher. After they leave the riser, the particles go upwards to Section S_5 and only a few of them reach the top of the degassing zone (S_3). Increasing the airflow rate, increases the rising force. Solids rise in higher amounts until Section S_3 and, on their way down, they pass through Sections S_1, S_2 and S_4, 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 (ϵ_s), 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_{sl}) and the corresponding relative error, for each airflow rate

Q (L/min)	ϵ_{sl} (% v/v)	Relative error (%)
5.5	15.1	0.5
9.1	14.4	4
30.3	15.8	5
90.2	15.1	0.6

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

Acknowledgement

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