Chemical Engineering Science 66 (2011) 3350–3357

Contents lists available at ScienceDirect

Chemical Engineering Science

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

Effect of spent grains on flow regime transition in bubble column

André Mota*, Antonio A. Vicente, José Teixeira

Institute for Biotechnology and Bioengineering, Centre of Biological Engineering, University of Minho, Campus de Gualtar, 4710-057 Braga, Portugal

ARTICLE INFO

Article history:
Received 4 October 2010
Received in revised form
20 January 2011
Accepted 21 January 2011
Available online 31 January 2011

Keywords:
Bubble columns
Hydrodynamics
Regime transition
Spent grains
Suspension
Stability

ABSTRACT

It is well known that two main flow regimes are present in bubble columns, being the evaluation of transition between homogeneous and heterogeneous regimes of crucial importance for reactor design. For air–water systems, several models have been satisfactorily proposed to explain this phenomenon. However when gas–liquid–solids systems are considered, solid particles influence on regime transition is not yet clear, in spite of the amount of research developed over the past years.

The objective of this work is to evaluate the effect of a specific solid phase – spent grains – on homogeneous regime stability and regime transition. Spent grains are cellulose-based particles that have been used to immobilize cells on biotechnology process. These particles are wettable and have a density close to water and its influence on bubble column reactors is particularly important in order to establish the limits were both regimes prevail.

A cylindrical Plexiglax BC of 18 L volume was used with air, water and spent grains at different concentrations (0–20% (wt.$_{\text{WET BASIS}}$/vol.)) as gas, liquid and solid phases. Regime transition was determined according to the drift-flux and slip speed concept.

It was found that at studied concentrations of spent grains, critical gas hold-up decreases as solids concentration increases. At the highest solids concentration and lowest gas flow rates no fluidization of the solid phase was observed. It is believed that the critical hold-up decrease was mainly due to bubble coalescence, as larger bubbles were observed when heterogeneous regime was present. This coalescence may be caused by the non-uniform distribution of solid phase on the column and the interaction of spent grains with bubbles in the liquid–gas interface

1. Introduction

Over the last decades hydrodynamics of gas–liquid–solid systems has been intensively study due to their applications in several industrial fields such as petrochemical, chemical, biochemical and biotechnology processes. The study of the hydrodynamics on three-phase systems presents a challenge to several research communities, which are dealing with bubble columns, airlift reactors, flotation columns, bubbly flow and fluidized beds. Most of the recent work dealing with bubble columns (BC) is focused on the stability of its flow regime (Zahradnik et al., 1997; Ruzicka et al., 2001a, 2003; Mena et al., 2005a)

In BC two main flow regimes occur: the homogeneous regime and the heterogeneous regime, that may be identified by varying gas input (Zahradnik et al., 1997; Ruzicka et al., 2001a). The homogeneous regime (HoR) is characterized by a uniform bubble rise through the column. Bubbles usually have similar size, are spherical, small and rise almost vertical. There is any large-scale liquid circulation and other phenomena as coalescence and break-up are negligible (Ruzicka et al., 2001a). In contrast, the heterogeneous regime (HeR) is characterized by a large bubble size distribution. Macro-scale liquid circulation, coalescence and a parabolic/non-uniform radial profile of hold-up with a maximum at center are typical on this regime (Mena et al., 2005a). The transition starts when the HoR loses its stability and gradual process occurs, where there are an increasing number of coherent structures (circulations) with increasing size and intensity (Zahradnik et al., 1997; Ruzicka et al., 2001a; Mena et al., 2005a).

Due to their characteristics, HoR and HeR have a different hydrodynamic behavior. This results in different mass, heat and momentum transfer properties. Consequently, it is important to clarify how operating parameters (reactor geometry, gas and liquid flow rates, and properties of the contacting phases) act on flow regime properties and transition (Zahradnik et al., 1997).

Parameters such as superficial gas velocity, column diameter, liquid and gas phase properties and distributor geometry acts simultaneously on regime transition(Shaikh and Al-Dahhan, 2007). The selection of the correct distributor is required to study regime transition, being perforated or porous plates the most commonly applied at laboratory and industrial scale.(Zahradnik et al., 1997). Zahradnik et al. (1997) demonstrated that perforated plates with holes inferior to 1 mm and porous plates are adequate

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* Corresponding author. Tel.: +351253601972
E-mail address: amota@deb.uminho.pt (A. Mota).

0009-2509/$ - see front matter © 2011 Published by Elsevier Ltd.
doi:10.1016/j.ces.2011.01.042

to characterize regime transition. Vijayan et al. (2007) studied the influence of sparger geometry in the regime transition, evaluating the influence of the ratio between area of sparger and area of column cross section. They found that if this ratio is increased the critical and global values of gas hold-up also increases (Vijayan et al., 2007).

Generally, regime evolution is observed by increasing gas flow rate input and determining the correspondent gas hold-up. There are several techniques to determine local or global gas hold-up such as: bed expansion (Zahradnik et al., 1997; Ruzicka et al., 2001a,b, 2003; Mena et al., 2005a), pressure drop, dynamic gas disengagement (Schumpe and Grund, 1986; Yang et al., 2010), conductivity (Ohkawa et al., 1997) and optical fibers (Cartellier, 1990). A complete review of the techniques used for hold-up measurement in two and three phase systems has been published by Boyer et al. (2002).

The analysis of the gas hold-up vs superficial gas velocity graph shows that HoR appears as a convex line while HeR follows a rational function (concave line). These lines are connected by a transition zone for intermediate gas velocity values (Ruzicka et al., 2001a). Regime transition identification is possible by applying the drift-flux concept initially proposed by Wallis (1969). In this method, the drift flux, j_{gl} (the volumetric flux of either phase relative to a surface moving at the volumetric average velocity) is plotted against the gas hold-up. The change in the slope of the curve represents the transition from homogeneous to heterogeneous flow. This concept has been used and modified by several authors for determining regime transition (Vial et al., 2000; Ruzicka et al., 2001a,b ; Mena et al., 2005a; Krishna et al., 1999).

Several models for regime transition have been proposed based on: (1) bubble drag force (Riquarts, 1979), (2) gas phase slip velocity (Joshi and Lali, 1984), (3) energy balance of the two flow mixture (Gharat and Joshi, 1992), (4) bubble size (Krishna, 1991; Hyndman et al., 1997), and (5) coupling between phases (Ruzicka et al., 2001a). The model proposed by Ruzicka et al. (2001a) based on the concept of the Darwinian drift of bubbles was able to describe with good accuracy the transition between HoR and HeR in a two-phase system. However some factors that affect regime stability namely, column dimensions, liquid phase properties and solids presence are not explicitly involved in the proposed model and experiments have been done over the last years to validate particular aspects of the stability criteria (Ruzicka et al., 2001a, 2008; Mena et al., 2005a).

In three-phase bubble column reactor, the effect of solids on gas hold-up has been the focus of several studies (Banisi et al., 1995a,b; Gandhi et al., 1999; Mena et al., 2005a,b, 2008). These systems can be classified as liquid-gas flow with the presence of solids or as liquid–solid fluidized beds with the presence of gas bubbles. However comparison between the different studies is difficult and results are often contradictory, due to differences in column design, operating parameters (mainly liquid throughput) and solids properties. Properties of the solid particles can be quite different depending on the size, shape, density, wettability, hydrophobic and surface properties. Having this in mind, their effect in gas hold-up and flow regime transition is far from being totally explained despite several attempts. Banisi et al. (1995a,b) reported that the presence of solids decreased gas hold-up while a dual effect was observed by other authors (Mena et al., 2005a; Xie et al., 2003). The presence of solids on bubble columns affects the gas–liquid mixture in several ways: bubble formation (Yoo et al., 1997), bubble rise, axial and radial (Gandhi et al., 1999; Warsito et al., 1997) profiles, mixing and dispersion, mass transfer (Mena et al., 2005b), gas hold-up and flow regimes (Mena et al., 2005a).

Generally gas hold-up decreases with solids concentration. There are several possible explanations for this kind of effect such as increased coalescence (Lu et al., 1995; Gandhi et al., 1999) and reduction of bubble breakup (Gandhi et al., 1999), increased apparent viscosity, and steric effect.

In what concerns the effect of apparent viscosity, some authors consider the solid and liquid phases as a ‘‘pseudo-homogeneous’’ phase, this requiring the need to define an apparent viscosity due to solid presence in liquid (Lu et al., 1995; Freitas et al., 1999). In a similar way to what has been reported for two-phase flows where a higher viscosity decreases gas hold-up, while at low viscosities the opposite occurs (Eissa and Schugerl, 1975; Ruzicka et al., 2003), a identical behavior might be expected for the effect of solids concentration on gas-hold-up. Nevertheless, recent studies in regime transition studying the effect of liquid viscosity reported that even at low viscosity values the global hold-up decreases (Yang et al., 2010). In general, an increase of viscosity is related with a decrease on gas hold-up. When solids are present, some authors consider the solid and liquid phases as a ‘‘pseudo-homogeneous’’ phase. This leads to the need of defining an apparent viscosity due to the solid presence in the liquid that would affect liquid phase properties as density and viscosity (Lu et al., 1995; Freitas et al., 1999). As far as we are aware some relations to determine viscosity are reported in literature being the most common ones by Oliver et al. (1961) (cited by Lu et al., 1995); Thomas (1965) (cited by Yoo et al., 1997); Barnea and Mizrahi (1973) (cited by Gandhi et al., 1999) and Metzner (1985) (cited by Yoo et al., 1997).

The change in viscosity promoted by solids in the liquid-phase reveals that possible relations/interactions between solids and viscosity are likely to occur on their effect on flow regime destabilization of g–l–s systems. Mena et al. (2005a) noticed that only for a high solid content the viscosity had an important contribution for this; however, this cannot be generalized as the solid effect on the apparent viscosity of the mixture depends on the properties of the solids applied. It seems that the BC design and size also play an important role when all these aspects are considered (Yang et al., 2010; Ruzicka et al., 2001b, 2003).

The dual effect that has been reported in the recent years lead to a discussion of what is the real effect of solids in BC. It seems that size, concentration and wettability play an important role on this effect. Accordingly to Banisi et al. (1995a,b) fine particles in small amount (suppressing coalescence) and large particles in high amount (promoting breakup) tend to increase hold-up, while moderate concentrations and sizes seem to decrease gas hold-up. Concerning the influence of the wettability, its real effect on gas hold-up remains unclear. Jamialahmadi and Muller-Steinhagen (1991) report that wettable particles increased hold-up while non-wettable particles have the opposite effect. However, Mena et al. (2005a) worked with alginate beads (low density and completely wettable soilds) and found that a dual effect is present—for low solid content ($<3.0\%$) solids enhanced the HoR regime stabilization and global hold-up increased while for higher solids content ($>3.0\%$) the opposite effect was observed. (Mena et al., 2005a).

From a critical point of view the actual knowledge of the flow regime transition in three-phase system remains scarce. The main reason is related not only with the correct data interpretation in terms of physical mechanism but also with the difficulty of relating all results reported in literature.

The aim of this study is to contribute to this subject and examine the effect of spent grains on the flow regime transition in a BC. These solids are flat, cellulose-based, completely wettable, low size and low density particles (Brányik et al., 2001). The choice of this solid phase corresponds to our interest in three-phase airlift reactors with immobilized biomass for continuous production of alcohol-free beer. Moreover, the calibration method to determine gas hold-up in three-phase airlift was also tested.

2. Methods

2.1. Apparatus and measurements

Measurements were performed in a cylindrical Plexiglas bubble column with internal diameter of 0.142 m. The distributor was a ceramic porous plate with 0.09 m of diameter and an approximate porosity of 38% (vol.). It ensures the three regimes: homogeneous, transition and heterogeneous. Compressed filtered air was the gas phase and water the liquid phase ($T=20$ °C). Spent grains, almost flat particles, with equivalent diameter $d_{EQ} < 2.1$ mm and density $\rho = 1037$ $\text{kg}_{\text{WET BASIS}}/\text{m}^3$ were the solid phase. The size distribution of the particles was determined by sieving into fractions using a portable sieve shaker (Model Analysette, Fritsch, Germany). With the obtained data, the equivalent diameter was calculated. The solids are completely wettable with a water adsortion index (WAI) of 8,12 $\text{g}_{\text{WET}}/\text{g}_{\text{DRY}}$. The following five solid loadings were used: 0 (water), 4%, 8%, 12%, and 20% ($\text{wt.}_{\text{WET BASIS}}$/vol.). The clear liquid height was $H_{l0} = 1.09$ m for all experiments (no liquid throughput). The dependence of the voidage (ε) on the gas flow rate (q) was measured three times and then averaged. The gas superficial velocity varied in the range $q=0$–0.027 m/s (0–0.43 dm^3/s), covering the homogeneous and part of the transition regime. The gas flow was measured with a Mass Flow Controler (Alicat Scientific, Inc., Tucson, AZ, USA) and variations in gas superficial velocity close to the transition point were within 2 mm/s (0.033 dm^3/s).

2.2. Gas hold-up

Gas hold-up was measured using two techniques: (1) bed expansion and (2) water column based differential pressure.

2.2.1. Bed expansion

In each experimental run, the gas flow was set, the bed height was recorded after the time (never less than five minutes) required to reach a steady value was achieved, and then the gas flow was increased. Each of the eight runs was repeated three times and the voidage values were averaged. Gas hold-up was determined according to (Deckwer, 1992; Zahradnik et al., 1997; Gandhi et al., 1999; Ruzicka et al., 2001a,b, 2003, 2008; Mena et al., 2005a; Yang et al., 2010):

$$\varepsilon_g = (H_{g+1} - H_l)/H_{g+1} \tag{1}$$

2.2.2. Water columns differential pressure

The gas hold-up was determined by measuring static pressure difference between two heights in the column using water columns. Our interest in using this technique is related with future tests to be performed in a three-phase airlift. Moreover, it is reported that pressure difference *per se* is not enough when three phase systems are applied (Boyer et al., 2002) and an additional technique should be applied to determine solids hold-up. Differential pressure was measured by the difference in water columns height. For each set of experiments, pressure differences (H1–H2) were measured at least three times during 5 min (after a gassing time of no less than 5 min). The mean value was then used to determine gas hold-up using the following equation (Freitas and Teixeira, 1998a,b):

$$\varepsilon_g = (H_1 - H_2)/d - (\rho_l - \rho_s)\varepsilon_s/\rho_l \tag{2}$$

2.3. Solids hold-up

Solids hold-up and distribution were determined using the method developed by Freitas et al. (1997). Briefly, a sampler adapted to retain spent grains with 60 mL of volume is used for collecting solids in the bubble column. This sampler consists of a cylinder with two valves in each end. The sampler is introduced into the BC between the two points where the pressure is measured, with the valves open in the flow direction and then these are closed simultaneously for sample collection. In order to achieve higher accuracy the retained solids were then filtrated and dried at 105 °C for 12 h before weighting. Then solids volume was determined and solids hold-up calculated accordingly (Freitas et al., 1997):

$$e_{si} = V_s/V_{spl} \tag{3}$$

2.4. Measurements errors

The relative error for bed expansion method is considered to be less than 5%. On the homogeneous and in the beginning of transition regime the layer is uniform and the interface is easy to locate with a 1 mm precision (precision of scale—millimeter paper). This resolution was considered adequate due to the height of the column used (1090 mm). However when transition starts to occur and waves appear and the determination of the height of the bubble column is difficult. To minimize this effect, the obtained value corresponds to the mean of the values measured during several oscillations. At the end of transition and in the beginning of heterogeneous regime oscillations were at maximum 30 mm around the mean value. Having in mind the increase in height column for these superficial gas velocities, it was possible to have an experimental error not exceeding 5%.

The resolution obtained for the water column method was the same as for bed expansion, for the highest flow rates. However, for the low gas flow rates, the error is larger (up to 10%), considering the measured differences of the height in water columns (10–120 mm). The measurement error associated with solids hold-up determination is considered to be not more than 10% (Freitas and Teixeira, 1998b).

Overall, the combined error for the determination of gas hold-up using is at its maximum 15%.

2.5. Evaluation of critical gas hold-up and critical gas velocity

Considering the primary data obtained (ε vs q), the critical point could be determined as the inflexion point of the data graph. However, its direct determination in the graph is difficult and inaccurate. Consequently, the data were re-plotted according to the drift-flux model and the inflexion point determined from the deviation of the data from the theoretical line of the uniform regime. This is a standard procedure. The theoretical line $j = j(e_g)$ is defined as:

$$j_t = \varepsilon_g(1 - \varepsilon_g)u \tag{4}$$

where u is the hindered bubble speed. Bubble speed (u) is the mean slip speed in case of no liquid flux through the column. The concept of the Darwinian drift was used to determine the bubble mean slip speed (Ruzicka et al., 2001a). Thus:

$$u_{theo} = u_0(1 - a\varepsilon_g/(1 - \varepsilon_g)) \tag{5}$$

where u_0 is the bubble terminal speed, and a is the bubble drift coefficient.

For each data line $\varepsilon_{g\ EXP}(q)$, they are obtained by linearization of Eq. (5), using the basic relation:

$$\varepsilon_{g\ EXP} = q_{exp}/u_{exp} \tag{6}$$

The experimental drift-flux is obtained from Eq. (4) together with Eq. (6),

$$j_{exp} = (1 - \varepsilon_g)q \tag{7}$$

The transition begins where Eq. (7) separates from Eq. (4): it is the critical point [q_C,ε_C], the instability threshold. The values of q_C and ε_C are the quantitative measures of the homogeneous regime stability. The evaluation procedure is an iterative process. The homogeneous data range is initially assessed, then is used for the linearization, till the correlation coefficient of the linearization is sufficiently close to unity.

The regime transition was also found using the slip-speed concept, where, at the critical point, the slip speed data u_{EXP}, departs from the u_{THEO} obtained from the model line.

The first criteria of the drift flux model is based on the coupling of phases, i e., on the mass conservation of the phases. The slip speed concept is based on the fact that, in HoR, the bubble speed decreases with the increase of hindrance caused by the increase of bubble concentration.

The obtained results are the average of these two methods. Since these two methods are equivalent, only different co-ordinates are used, the results should be similar. This was the test of correctness (Mena et al., 2005a; Ruzicka et al., 2008a).

In the literature, the stability criteria normally used on GLS systems are scarce and several principles have been applied. Initially, they have been based on correlations obtained from experimental data but they lack in terms of universal application due to their specificity (Krishna, 1991; Wilkinson et al., 1992; Reilly et al., 1994). Theoretical criteria based on theoretical concepts are more accurate and may be applied *a priori* (Ruzicka and Thomas, 2003; Shnip et al., 1992) or *a posteriori* as the slip speed concept and the drift-flux model (used in this work).

3. Results and discussion

3.1. Bed expansion vs water columns differential pressure for gas hold-up determination

For the system considered, the determination of gas hold-up using both techniques showed similar results. The measuring of gas hold-up by water columns combined with the modified method for solid hold-up determination used by Freitas et al. (1997) appears to be a suitable method to determine gas hold-up in three-phase systems where spent grains are the solid phase. In fact and considering the solid distribution in the entire BC the difference between the experimental and theoretical values of solid load determined by this technique was 2.97%; 3.32%; 3.98% and 6.94% for 4%; 8%; 12%; 20% (wt.$_{\text{WET BASIS}}$/vol.), respectively. The obtained errors are in the same range of the ones obtained by other authors (Freitas et al., 1997; Freitas and Teixeira, 1998a,b). Having in mind that these values have been calculated with the experimental values for gas hold-up, it is clear that the applied technique may be applied with a good accuracy.

Moreover, the results presented in Fig. 1show the experimental errors for the two techniques confirm this conclusion. In Fig. 1A, the deviations for all range of solids loads used are shown while in Fig. 1B only the first three solids loads are considered (4%, 8% and 12% (wt.$_{\text{WET BASIS}}$/vol.)).

The maximum and mean deviations between the results obtained with the two different techniques are, respectively, 26% and 7%, with most of the measured values with errors in the range ±8% an acceptable result having in mind the applied techniques. Nevertheless the highest errors occur at the lowest gas flow rates and high solids content. In fact, for the maximum solids content and due to the non-homogeneity of solids in the column, especially at low gas flows, the combined method for solids and gas hold-up determination is not suitable. It seems that, for these type of solids, only solids load values below 12% (wt.$_{\text{WET BASIS}}$/vol.) are accurate enough to perform a correct evaluation of the data in terms of regime transition. Thus, for 20% (wt.$_{\text{WET BASIS}}$/vol.) solids load the results were not used for the determination of regime transition. Anyway no regime transition was observed at this solids load. For higher gas flow rates the agreement between the data was always below 4%. Therefore both techniques were considered for the determination of critical values (ε_C and q_C) for regime transition.

3.2. Assessment of primary data

In Fig. 2 are plotted the curve $\varepsilon(q)$ obtained for each concentration of solids (0–20% (wt.$_{\text{WET BASIS}}$/vol.)).

Fig. 2 shows that, in this column, regime transition occur at lower gas flows than the ones obtained by other authors in BC (Ruzicka et al., 2001a,b; Mena et al., 2005a). However the values of gas hold-up are in the same range. There are two possible explanations for this: bubble column size and the effect of the distributor.

It is reported that column size (diameter and height) influences the global hold-up. Also, sparger influence over the column axis can go up to four times the column diameter (Gandhi et al., 1999; Lu et al., 1995).For the same gas flow, Ruzicka et al. (2001b) observed that gas hold-up values are usually lower when BC diameter increases and/or column height increases. Thus the critical values (ε_C vs q_C) for regime transition also diminish (Ruzicka et al., 2001b). Generally gas spargers in BC have a diameter that occupies all cross-section of the BC. As in our case the sparger corresponds to 2/3 of

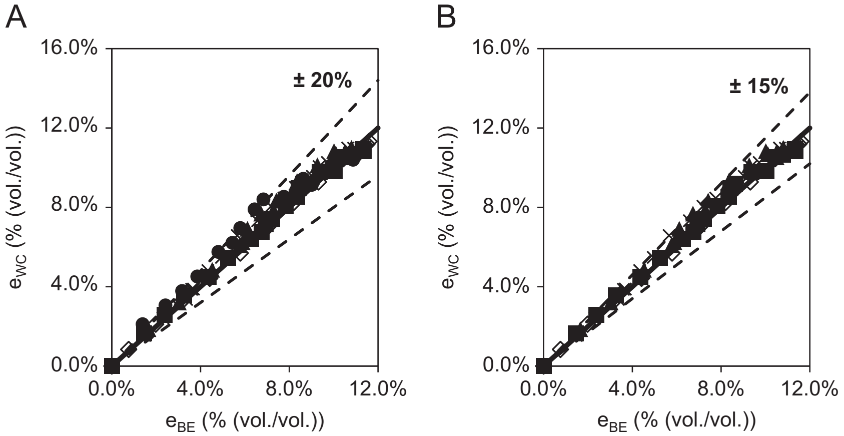

Fig. 1. Comparison between gas hold-up obtained by water columns (e_{WC}) and bed expansion (e_{BE}): (A) solids load up to 20% (wt.$_{\text{WET BASIS}}$/vol.); (B) solids load up to 12% (wt.$_{\text{WET BASIS}}$/vol.). Legend: ◆—water; ■—C=4% (wt.$_{\text{WET BASIS}}$/vol.); ▲—C=8% (wt.$_{\text{WET BASIS}}$/vol.);+—12% (wt.$_{\text{WET BASIS}}$/vol.); ●—20% (wt.$_{\text{WET BASIS}}$/vol.).

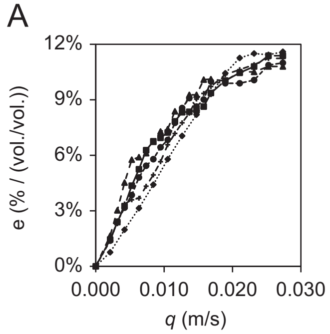

Fig. 2. Primary data obtained from different gas hold-up methods: (A) bed expansion; (B) water columns. Legend: ◆—water; ■—C=4% (wt.$_{\text{WET BASIS}}$/vol.); ▲—C=8% (wt.$_{\text{WET BASIS}}$//vol.); +—12% (wt.$_{\text{WET BASIS}}$/vol.); ●—20% (wt.$_{\text{WET BASIS}}$/vol.).

the bubble diameter, this characteristic is responsible for a higher height until gas flow stabilizes and for local liquid circulation near the sparger. This means that the height of the column necessary for the gas to achieve a flat profile is higher as there is a region at the bottom of the column where liquid circulation occurs (less visible at low flows), which difficults a rapid stabilization of the gas profiles as typically occurs in HoR. The liquid circulation is a consequence of the higher amount of bubbles in the center of the column immediately above the distributor, resulting in "local" lower hold-up values. The influence of sparger geometry on gas hold-up values has also been studied by Vijayan et al. (2007). As the gas hold-up values obtained in this work are in the same range values as those reported by these authors when similar HD/D and A_S/A_C ratios are considered, our experimental values may be considered to be within the expected range (Vijayan et al., 2007).

The selection of this distributor is related with our interest on the study of the three-phase hydrodynamics in an internal airlift reactor, as in these systems the distributor cross section is of the same order of magnitude of the airlift riser cross section and always smaller than the total column cross section. The applied distributor allowed to reach the objective of solids fluidization at low gas flow rates (except for the maximum solids concentration).

Even if this system would behave differently from those reported in literature, which is not the case, it would still possible to conclude on the effect of the solid phase on flow regime transition, as experiences were done for all cases in the same BC. In addition, the presence of both regime flow (HoR and HeR) in air–water systems was clearly identified.

From Fig. 2, it is also possible to observe, at low gas flows, that the presence of the solid phase causes an increase in gas hold-up. This may be attributed to a stabilization effect of the bubbles at low gas flows, being the opposite effect observed as gas flow is increased. This "stabilization" effect causes an increase in gas residence time and consequently hold-up is higher. This is also related with a decrease of the height that the gas needs to achieve the flat profile, typical for HoR.

The effects associated with the use of a sparger not occupying the entire cross section of the column may also contribute to the observed increase in gas hold-up. As due to their sedimentation properties an increase in solids concentration near the distributor occurs (specially at low gas flow and high solids load), a higher interaction between bubbles and particles occurs. There are also the steric effect (presence of solids) of spent grains as well as its surface properties that can have an important effect on the interactions between solids and bubbles. This effect reduces bubble rise velocity leading to a slight increase on hold-up (Lu et al., 1995; Mena et al., 2005a). In fact, when solids were present, a larger amount of smaller bubbles near the wall was observed in comparison with air–water systems.

Spent grains wettability is another factor that may contribute to the obtained results. Also, it has been reported that the influence of the physical properties of solids is higher when solids have a small size compared with bubble size, as is the case (Mena et al., 2005a). A higher drag force on the bubble surface (gas–liquid interface) is created by the presence of small size solids with a consequent reduction on bubble rise velocity and gas hold-up increase (Mena et al., 2005a). The combination of the above mentioned effects contributes for the observed increase in gas hold-up at lower gas flows when solids are present. This is in agreement with Jamialahmadi and Muller-Steinhagen (1991) that concluded that wettable particles increased hold-up by suppressing coalescence while non-wettable particles had the opposite effect. It may also be noted that the resistance promoted by solids sedimentation characteristics was evident when maximum solid concentration was tested (20% (wt.$_{\text{WET BASIS}}$/vol.)), as no fluidization of solid particles was observed at the lowest gas flow rates.

In Fig. 3A, it is possible to verify that when solids are present, at low gas flows, global gas hold-up tends to increase with a maximum around 8% (vol./vol.) of solids. In HoR, this result is a consequence of particle–bubble interactions that result in bubble break-up and dispersion, reduced bubble rise velocity and consequent increase in hold-up.

On the other hand, Fig. 3B shows that in HeR at high flows the opposite effect is present revealing a dual effect of the particles. At these values of gas flow (HeR) liquid circulation is higher and more pronounced, the effect of particle–bubble interaction being lower. Bubble–bubble interactions increased and coalescence occurred. At this stage, coalescence does not seem to be significantly affected by the presence of the solid phase. However, with the obtained data, it is impossible to identify/evaluate the importance of the physical mechanisms related with solid properties (wettability, size, and density). At high gas flows, larger bubbles were visually observed revealing coalescence phenomena at these stages. The absence of 20% (wt.$_{\text{WET BASIS}}$/vol.) solids data is related with the fact that HoR was not achieved under this solids load condition (see Fig. 4).

3.3. *Assessment of secondary data*

In Fig. 4 are plotted the Drift-flux results for the data obtained by the bed expansion method. This method allows for an accurate determination of the critical point (point at which HoR stability disappears). The critical point, which corresponds to the inflexion points on Fig. 2, is now clearly identified on Fig. 4 (open symbols) for the different experimental conditions. As previously said, this point corresponds to the beginning of separation of the plots of the experimental and calculated data.

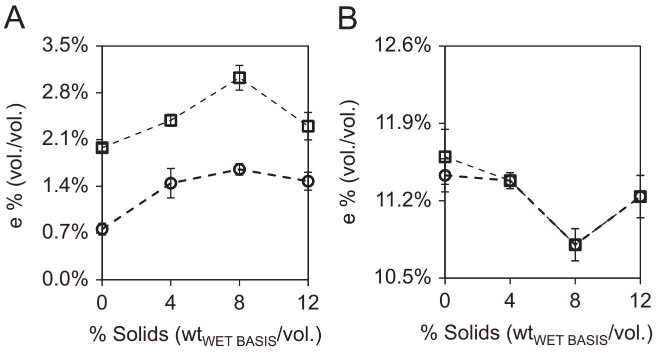

Fig. 3. (A) Solids influence at low gas flow rates in gas hold-up (bed expansion). Legend: ○—q=0.2 cm/s; □—0.4 cm/s; (B) solids influence at high gas flow rates in gas hold-up (bed expansion). Legend: ○—q=2.5 cm/s; □—2.7 cm/s.

From the results plotted in Fig. 4, a decrease of critical gas hold-up and critical gas flow with solids load is clearly observed. Critical gas hold-up decrease presents a linear behavior, while critical gas flow has an exponential trend. This suggests that above 12% (wt.$_{\text{WET BASIS}}$/vol.) of spent grains loading the critical gas flow is similar to the minimum gas fluidization flow for this system. In fact, as said before, when the highest solids load was applied, the value for minimum fluidization gas velocity was only 20% inferior to the critical gas flow for 12% (wt.$_{\text{WET BASIS}}$/vol.) loading. This indicates that, under the tested solids load, 12% (wt.$_{\text{WET BASIS}}$/vol.) solids load was the minimum concentration where HoR could be established and only for a small range of gas velocities ($0.2\ \text{cm/s} < q_G < 0.5\ \text{cm/s}$). Concerning the critical gas hold-up, the following correlation can be found to describe its dependence on solids load (Eq. 8):

$$\varepsilon_c = 0.104 - 0.558\delta_s \quad R^2 = 0.94; \quad R_{xy} = 0.97 \tag{8}$$

This equation indicates the way spent grains influence regime transition on the studied bubble column. Eq. (8) intercepts y-axis at a value lower than the ones found in the literature (Mena et al., 2005a), this being related with the above addressed specific issues of this BC. When comparing the slope of Eq. 8 with the one reported by Mena et al. (2005a) for alginate beads as the solid phase, the obtained value with spent grains is two times larger. This allows to conclude that spent grains have a more pronounced effect on regime transition than alginate beads. This is not unexpected as the properties of both solids are different, mainly size, shape and wettability.

Presented results indicate that a reduction in HoR regime stability is observed when solids are present (Figs. 4 and 5). It was visually verified that when gas flow was increased big bubbles start to appear, especially in the column center, due to coalescence. In fact, at maximum solids load where no HoR was established, bubble coalescence was observed even at the lowest flows. At the highest flow, slug regime was present, a typical situation for BCs with diameters inferior to 20 cm (Deckwer, 1992). This result contradicts the one obtained by Jamialahmadi and Muller-Steinhagen (1991) that observed that wettable particles increase gas hold-up by suppressing coalescence. In fact this occurs at low gas flows (Fig. 3A), but not at higher ones (Fig. 3B). As above said, this dual effect of spent grains is attributed to the BC design. The increase in gas hold-up by the action of spent grains means that more bubbles are present and interactions between bubbles are higher. This increased rate of collisions promotes coalescence. With the gas hold-up increase in the HoR by the presence of the solid phase, non-uniformities are formed in the gas phase and the stability of HoR is reduced. Thus the HeR is achieved earlier and the critical values (ε_C vs q_C) are lower when solids are present (Fig. 5).

It is also possible to observe that either bed expansion and water columns techniques show similar results (Fig. 5). This indicates/confirms that, for solids loads inferior to 12% (wt.$_{\text{WET BASIS}}$/vol.), the combination of solid hold-up method and water columns appear to be a suitable technique to evaluate gas hold-up in different reactors configurations.

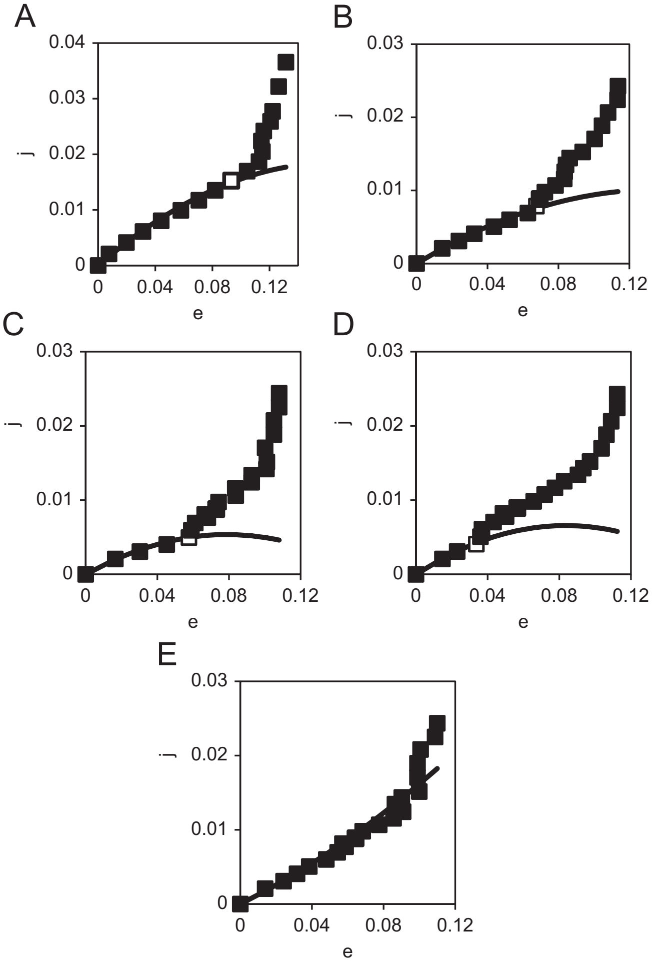

Fig. 4. Drift-flux plot: Drift-flux—j (m/s) vs Gas hold-up (e). Legend: — (smooth line): j_t by Eq. (4); ■ (Data points): j_{EXP} by Eq. (6); □ (blank data): Critical point. A, B, C, D, and E: Different solid loads (0%, 4%, 8%,12% (wt.$_{\text{WET BASIS}}$/vol.))

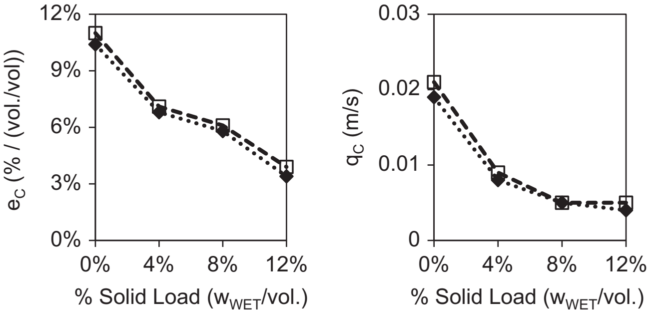

Fig. 5. Evaluation of critical gas hold-up and critical gas flow with solid load. Legend: ◆—bed expansion; □—water columns.

It was suggested that solids effects can be similar to what occurs in a low viscosity liquid on bubble columns (Mena et al., 2005a). In our case, critical values (ε_C vs q_C) decrease with solids load, which is in agreement with published results on viscosity effect on flow regime transition (Yang et al., 2010). Ruzicka et al. (2003) reported a dual effect of liquid viscosity when low viscosity (<2.1 mPa s) and high viscosity liquids are present. Meanwhile, recent studies published by Yang et al. (2010) identified that at low viscosity liquids there is a fast decrease of critical values (gas hold-up and gas flow) followed by a small decrease. The critical viscosity where this change occurs was around 3 mPa s. The behavior of critical gas flow vs viscosity was (Yang et al., 2010):

$$q_{C1} = -0.019\ln\mu_l + 0.025 \text{ and } q_{C2} = -0.018\ln\mu_l + 0.056 \text{ for } \mu_l < 2.8\,\text{mPa s} \tag{9}$$

These authors considered that the first value for the critical gas flow – q_{C1} – corresponds to the end of the homogeneous regime when the first big bubbles are formed and the second one – q_{C2} – is obtained when full heterogeneous regime is established (JH Yang et al., 2010). Accordingly, they suggest that the values obtained for critical values of transition correspond to the value when homogeneous regimes loses stability (q_{C1}).

Oliver et al. (1961) cited by Lu et al. (1995) suggested the determination of a pseudo-homogeneous viscosity for a solid–liquid mixture according to the following equation:

$$\mu_{s-l} = \mu_l [f(\delta_s)]^{-1}, \qquad (10)$$

where

$$f(\delta_s) = (1-0.75\delta_s^{1/3})(1-2.15\delta_s)/(1-\delta_s)^2. \qquad (11)$$

The application of this equation to our experimental data was based on the fact that the solid phase used – alginate beads – by Lu et al. (1995) had similar properties – density and size –to the solids used in this work. In our case, the following empirical curve relating critical gas flow with the pseudo-homogeneous viscosity (1 mPa s $< \mu_{S-L} <$ 1.7 mPa s) was obtained:

$$q_{C1} = -0.032 \ln \mu_l + 0.02 \quad (R^2 = 0.98). \qquad (12)$$

This relation was obtained for solids load 0%, 4%, 8% and 12% (wt.$_{\text{WET BASIS}}$/vol.) as only in these situations transition regime was observed. The obtained values are in the same range as those of Eq. (9) (JH Yang et al., 2010).

These results demonstrate that the influence of spent grains load in the critical value corresponding to the end of HoR (q_C) follows the same pattern of low viscosity liquids. It seems that, depending on the column size (diameter and height), type of distributor and type of solids, the dual effect of solids and viscosity suggested by some authors (Ruzicka et al., 2003; Mena et al., 2005a) may or may not occur (Yang et al., 2010) as in this case. It is important to notice that Eq. (12) is limited to this particular system (air–water–spent grains), being similar evaluations possible and desirable for other gas–liquid–solid systems in order to have a better understanding of its hydrodynamics.

Although the presented information aims at contributing to the effect of a solid phase on the hydrodynamics of g–l–s systems, further work is required to clarify how solids influence regime transition and the magnitude of this influence. Obtained results allowed to confirm that that solid influence is related with the low viscosity of the "pseudo-homogeneous" liquid phase formed and that other parameters such as steric effect, wettability, BC design and distributor geometry play also an important role in flow regime transition.

4. Conclusion

The effect of spent grains particles on homogeneous regime stability and regime transition in a three-phase bubble column was investigated experimentally. The stability was expressed by the critical values of gas holdup and gas flow rate. The experiments showed that the solids promoted stabilization on HoR for low gas flow rate. Moreover, it was demonstrated that spent grains decrease the critical values where the HoR prevails. This influence was demonstrated to have similar effect to the one found for low viscosity liquids on regime transition. In addition, it was possible to conclude on the importance of the steric effect of solids as well as their specific properties (wettability) on regime transition.

Despite this, the mechanisms by which solids affect the regime transition is far from being understood. Furthermore the influence of column size and type of solids makes difficult a real assessment when all results available in literature are compared.

Nomenclature

H	column height, bed expansion, (mm)
H_1	column height, water column nr 1, (mm)
H_2	column height water column nr 2, (mm)
d	distance between water columns points (mm)
V_s	volume of solids collected (mL)
V_{slp}	volume of sample (mL)
j	drift flux (m^3/m^2 s)
u	mean bubble rise speed, mean velocity of gas in column, (m/s)
u_0	Terminal bubble speed (m/s)
a	coefficient of the Darwinian drift (dimensionless)
q	gas flow rate (m/s)
A_S/A_C	ratio between area of sparger and area of cross section (dimensionless)

Greek symbols

ε	hold-up (dimensionless)
ρ	density (kg/m^3)
δ	solid load, %, (vol./vol.)
μ	viscosity (mPa s)

Sub/superscript

g	gas
l	liquid
s	solid
theo	theorical
exp	experimental
C	critical

Acknowledgement

The authors gratefully acknowledge the financial support from FCT (Fundacão para a Ciência e Tecnologia, SFRH/BD/37082/2007).

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