Solid Distribution in a Slurry Bubble Column with Two Immiscible Liquid Phases

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Experimental and theoretical studies of axial solid distributions in slurry bubble columns have been carried out extensively (Cova, 1966; Smith et al., 1986; Jean et al., 1989; Reilly et al., 1990). Most of the experimental works performed in this area have characterized the solid concentration profiles using water as the liquid phase. A limited amount of experimental data is available for slurry bubble columns with organic liquids (Bach and Pilhofer, 1970; Bhaga et al., 1971; Brian and Dyer, 1985; Badgujar et al., 1986b) and mixtures of immiscible liquids (Badgujar et al., 1986a).

The properties of the liquid phase are among the most important factors that affect the fluid dynamic behavior of bubble columns. It is a well known fact that mixtures of miscible liquids behave differently from pure liquids in bubble columns when the substances that comprise the mixture have different chemical structure. For instance, Bhaga et al. (1971) noted that the gas holdup of a mixture of two miscible organic liquids in two-phase bubble columns exhibits a maximum with respect to the concentration of one of the liquids. The difference of behavior between mixtures and pure liquids cannot be correlated in terms of the density, viscosity and surface tension. This implies that the empirical correlations available in the literature to predict gas holdup, which have been usually based on experiments with pure liquids, cannot be used for the prediction of gas holdup in bubble columns with liquid mixtures (Badgujar et al., 1986a; Pino et al., 1990). The main reason for this difference is the fact that one of the components in the mixture acts as a surfactant. In this case, the description of dynamic interfacial phenomena at the gas-liquid interface does not depend exclusively on common physical properties (density, viscosity, and surface tension), but also on the way that the surfactant is distributed over the gas-liquid interface. Recent attempts have been made to quantify this effect through measurable properties, such as dynamic surface tensions (Gorowara and Fan, 1990).

The complexities of the fluid dynamic of bubble columns described above become more evident when dealing with immiscible liquids. For instance, in a bubble column with two immiscible liquids, up to three different fluid-fluid interfaces might be present, each of which can be affected by the existence of surface-active substances. Bubble columns with two immiscible liquid phases have not received wide attention in the literature, although they find application in metallurgical and petrochemical processes (Hatzikiriakos et al., 1990). Kato et al. (1984) analyzed the gas holdup in the air-kerosene-water system in a multistage bubble column. They concluded that the addition of kerosene to water reduces the gas holdup, which is the same behavior observed when solid particles are added to gas-liquid systems.

Hatzikiriakos et al. (1990) reached the same conclusion in the experimental study of bubble columns whose liquids were mixtures of water and organic compounds. The reduction in gas holdup has been attributed to the increase in the apparent viscosity of the liquid (taken as a pseudosingle phase) due to the presence of dispersed drops. This is consistent with the explanation given to the reduction in gas holdup due to the addition of solid particles in slurry bubble columns. These observations do not agree with some of the results obtained by Badgujar et al. (1986a). These investigators reported a reduction in the gas holdup when Soltrol-130 was added to water, whereas an increase in gas holdup was obtained when the organic liquid added to water was silicone oil. Badgujar et al. argued that the effect of the presence of two liquid phases should be explained in terms of what happens at the microscopic level on fluid-fluid interfaces.

Studies of solid distributions in slurry bubble columns with two immiscible liquids were performed by Badgujar et al. (1986a). They observed that the solids dispersion coefficient of glass beads (81 μm in diameter) was higher when the liquid was a mixture of 15.6 wt. % silicone oil in water than when the liquid was pure water. Also, the apparent settling velocity of solid particles was lower in the gas-liquid-liquid-solid system.

In all the investigations cited above dealing with immiscible liquid mixtures, liquid phases were water with small to moderate (less than 16 wt. %) additions of organic components; thus, the organic liquid was dispersed in the form of drops in an aqueous continuous phase. In this work we will explore the distribution of a monodisperse solid in a semibatch bubble column with two immiscible liquid phases. In this case, the continuous liquid phase is an organic liquid (kerosene), whereas water will be present in small concentrations as a dispersed liquid phase. Experimental results show that adding small amounts of water to an organic liquid phase in a semibatch

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slurry bubble column has a drastic effect in the solid concentration profile.

Experimental Study

Experiments were carried out in a plexiglas bubble column with an internal diameter of 10 cm. The gas distribution section is a cone with an apex angle of 30° connected to an inlet pipe with 1.5 cm ID. The operating mode was continuous with respect to the gas phase and batch with respect to the slurry phase. The height of the gas-slurry suspension was maintained at approximately 275 cm during operation at steady state. The column was provided with sampling probes consisting of tubes with an internal diameter of 0.64 cm through which slurry samples were withdrawn. We verified that the concentration of solids in the samples was independent of the rate of sample withdrawal. The probes were located throughout the length of the column with a spacing distance of 20 cm. Slurry samples were dried at 60°C, and their solids concentrations (C_s, mass of solid per unit slurry volume) were measured gravimetrically.

All the experiments were conducted at atmospheric pressure and 25°C. The gas used was atmospheric air. The liquid phase was kerosene ($\rho = 808$ kg/m^2, $\mu = 1.6$ mPa·s, $\sigma = 20.4$ mN/m) and mixtures water-kerosene. The solid phase consisted of monodisperse spherical glass beads of diameter equal to 110 μm and density equal to 2,800 kg/m^3.

The following experimental procedure was followed. First, the column was loaded with kerosene and the appropriate amount of solids required to achieve a specified load concentration ($C_{s,L}$). Then, the air flow rate was set and the system was allowed to reach steady-state conditions (approximately two hours). After that, slurry samples were taken at the available sampling ports. After completing the experiment with kerosene as liquid phase, a certain amount of water was added to the column through the top so that a specified water concentration was achieved. The system was again allowed to reach steady-state conditions before taking slurry samples again.

The gas holdup (ϵ_g) was measured by the bed expansion method as the ratio between the volume of gas, determined as the free volume at the top of the column after the complete disengagement of the gas phase at the end of the experiment, and the volume of gas-slurry suspension during operation.

Results and Discussion

At superficial gas velocities that are above the critical gas velocity for solids suspension, the solid distribution in semibatch slurry bubble columns with a single liquid phase exhibits an exponential decay of the solid concentration with' axial distance. These profiles are the consequence of the superposition of a convective mechanism, which represents the sedimentation of the particles, and a dispersive mechanism resulting from the motion of the slurry induced by the gas flow (Smith et al., 1986). In this work we observed this behavior for the air-kerosene-glass beads system.

When small amounts of water are added to the air-kerosene-glass beads system, the solid concentration profiles change drastically. As water is added, the solid particles form clusters within water drops that tend to agglomerate at the bottom of the column. These clusters seem to be the result of the ability of the water to wet the surface of the particles. When a drop of water touches a solid particle, the water spreads over the surface of the particle until it is completely trapped within the drop. This process is repeated until an equilibrium drop size is reached in which a certain amount of solid particles remain in the interior of the drop. In some cases, the drops of water with solids trapped inside were perfect spheres of approximately 0.5 cm in diameter which behaved as independent particles.

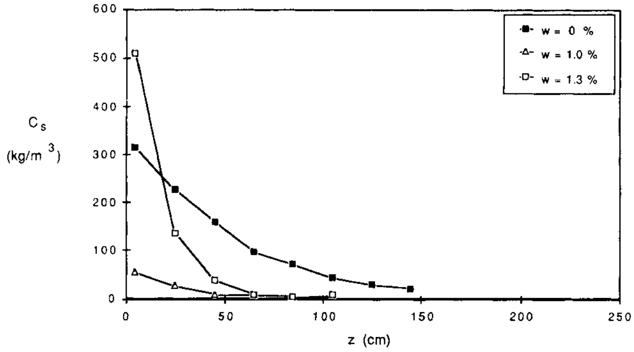

Figure 1. Solid concentration profiles.

$C_{s,L} = 100$ kg/m^3, $V_g = 5.0$ cm/s

Once water is added, the system is allowed to reach steady state. Depending on the operating conditions, three different situations result:

Case 1. Two regions of solids suspension are observed. At the bottom of the column, the solid is suspended in the form of clusters within water drops that are dispersed in the kerosene; we shall term this the four-phase region (gas-water-kerosene-glass beads). The rest of the column has small amounts of suspended solid particles and no water; we shall refer to this zone as the three-phase region.

Case 2. The solid settles forming a compact sedimentation region, which is generally within the cone. In this case, the water occupies part of the interparticle pore space in the sedimentation region. Above this region, the rest of the column resembles the three-phase region described in case 1.

Case 3. The solids suspended again, and the final state is similar to the original steady state before the addition of water.

Figure 1 shows the effect of the presence of water in the solid concentration profile for a superficial gas velocity $V_g = 5$ cm/s and a load solids concentration $C_{s,L} = 100$ kg/m^3. The level $z = 0$ corresponds to the bottom of the cylindrical section of the column. The parameter w represents the volume percent of water in the slurry. The curve corresponding to $w = 0\%$ corresponds to the case in which the liquid phase is pure kerosene. For $w = 1.0\%$, case 1 is observed. The four-phase region exists within the cone; the low values of solids concentration in the column indicate that it is completely a three-phase region as the one previously described. For $w = 1.3\%$, case 1 is also observed but the four-phase region reaches into the lower part of the column. High values of the solids concentration for $z \leq 25$ cm reflect the high level of solids in the four-phase region. It is interesting to note that the presence of water increases the apparent sedimentation velocity of the solid phase, since the solid clusters formed within water drops behave like large particles with an improved ability for sedimentation.

At higher gas superficial velocities ($V_g = 16$ cm/s) and the

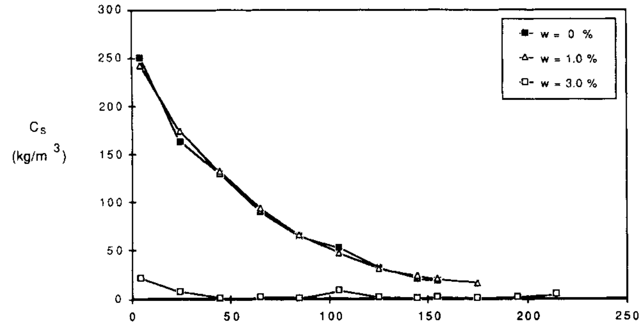

Figure 2. Solid concentration profiles.

$C_{s,L} = 100$ kg/m^3, $V_g = 16.0$ cm/s

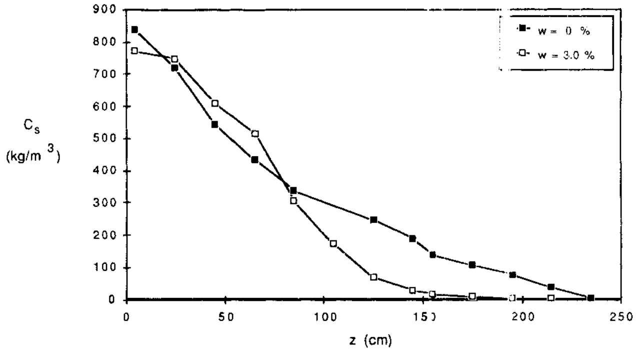

Figure 4. Solid concentration profiles.

$C_{s,L} = 400$ kg/m^3, $V_g = 16.0$ cm/s

same load concentration (Figure 2), a larger amount of water is required to obtain a noticeable effect. For $w = 1.0\%$, the solids suspend again (case 3) to yield the same concentration profile as that obtained with pure kerosene, whereas the same water concentration at $V_g = 5$ cm/s induced the presence of case 1. For $w = 3\%$, the solid settles into the cone (case 2) and once again the column is a three-phase region. The fact that more water is needed at higher gas velocities to form solids agglomeration is due to the rupture of clusters under the high degree of turbulence that exists at high gas velocities.

For high load concentrations ($C_{s,L} = 400$ kg/m^3, Figures 3 and 4), the same behavior is observed. Their main difference with low load concentrations is that, at this level of solids content, when enough water is added the four-phase region occupies part of the column instead of being restricted to the cone. The solids concentration profiles, corresponding to $w = 1\%$ in Figure 3 and $w = 3\%$ in Figure 4, show the variation in solids concentration within that region. In the case of Figure 3 the clusters reach up to $z = 100$ cm, whereas in the results of Figure 4 they reach up to $z = 75$ cm. As a whole, the concentration profiles in these two cases differ from the typical profiles observed in bubble columns with one liquid phase. The global appearance of the curves show an inflection point that marks the transition from the four-phase region to the upper section of the column, where small amounts of solids are present. The difference of solids suspension mechanisms in the four-phase and three-phase regions is evident. In the four-phase region, the solids concentration is more uniform than in the three-phase region. The high values of the solids concentration in the four-phase region suggest that the four-phase suspension is saturated with solids: i.e., a limiting maximum concentration of solids has been achieved. In this region, even though the solids concentration is very high, they do not sediment. Instead, experimental observation shows that they form clusters that move in strong recirculation patterns.

Even though the addition of water has a strong effect over the distribution of the solid phase, the global gas holdup in the column is not sensitive to the presence of water. Table 1 shows the measured gas holdup for the experiments reported here. The changes in this parameter with water concentration are of small magnitude.

Apart from the experiments previously reported, we performed additional qualitative tests to determine if the agglomeration of solids and the formation of the four-phase region occurred under other conditions. The presence of this phenomenon was observed in the following experiments, with water concentrations always less than or equal to 3%: 1. using the air-kerosene-water-sand system with average particle diameter of 80 μm; 2. using the air-hexane-water-glass beads system. Additionally, water was fed to the column through the bottom instead of the top in some experiments, by injecting it to the inlet pipe at the bottom of the cone. In these experiments, solids agglomeration was observed. This indicates that the degree of mixing of the water in the column is sufficiently strong to allow the water to capture the suspended solids regardless of the point of water injection.

The effect observed in the present work has not been reported in the literature to our knowledge. In the experiments per-

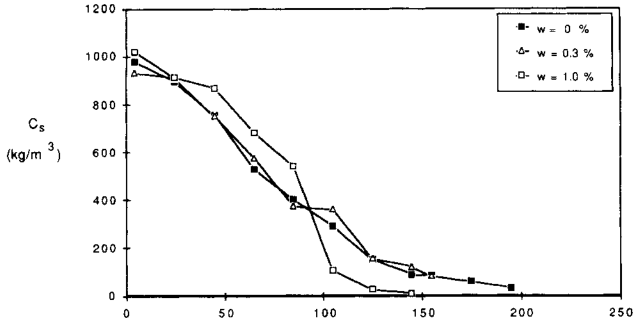

Figure 3. Solid concentration profiles.

$C_{s,L} = 400$ kg/m^3, $V_g = 5.0$ cm/s

Table 1. Gas Holdup

$C_{s,L}$ kg/m^3	V_g cm/s	w %	ϵ_g
100	5.0	0	0.354
	5.0	1.0	0.326
	5.0	1.3	0.333
	16.0	0	0.557
	16.0	1.0	0.557
	16.0	1.3	0.534
400	5.0	0	0.303
	5.0	0.3	0.299
	5.0	1.0	0.254
	16.0	0	0.491
	16.0	3.0	0.526

formed by Badgujar et al. (1986a) with four-phase systems, the organic liquid phase was present in small concentration, which made the aqueous phase continuous. Under these conditions, the phenomenon of solids agglomeration was not reported.

Conclusion

The results obtained in the present work show that small amounts of water in slurry bubble columns with organic liquids can have a strong effect over the solids distribution. This should be taken into account when working with two immiscible liquids in a slurry bubble column or when working with an organic liquid with the possible existence of small quantities of a dispersed aqueous phase. Further work is needed to quantify the effect of the operating conditions and physical properties of the phases on this phenomenon.

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