ULTRASONIC CHARACTERIZATIONS OF SLURRIES IN BUBBLE COLUMN REACTORS

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INTRODUCTION

For the optimum design and operation of gas-liquid-solid three-phase reactors, the degree of dispersion of the solid (catalyst) in the reactor must be understood and controlled. Recently, a method involving the measurement of ultrasound transmission has been reported in a slurry-phase stirred-tank reactor which offers the possibility of using the ultrasonic technique to measure solid holdup in a three-phase slurry reactor (1-3). The ultrasonic transmission uses measurements of the velocity and attenuation of the sound wave which travels directly through the slurry sample. When an acoustic wave strikes the boundary between two different media (liquid and solid) and the acoustic impedances of the two media are different, some acoustic energy will be reflected, absorbed, and some will be transmitted. The reflected wave travels back through the incident medium (liquid) at the same velocity. The transmitted wave continues to move through the new medium (solid) at the sound velocity of the new medium. When the velocity of sound in a liquid is significantly different from that in a solid, a time shift (a velocity change) 9n the sound wave can be detected when solid particles are present relative to that for the pure liquid. The amplitude of the sound wave is also reduced when a solid particle is present since the wave is partially scattered and absorbed. Therefore, a change in amplitude of the sound wave can also be detected when solid particles are present relative to that for the pure liquid. Okamura et al. (1) and Soong et al., (2-4) used a continuous stirred-tank reactor to correlate the solid holdup to the relative time shift $[(t_a-t_b)/t_o]$. Furthermore, the application of the measurement of ultrasound transmission for gas holdup (5-7) and for gas holdup as well as low concentration of solid (up to 1 wt. %) under limited superficial gas velocities (up to 3 cm/s) in a slurry-bubble-column reactor has been reported (8,9). This leads to the initial study of using the ultrasonic technique for the measurement of solid holdup in a three-phase gas-liquid-solid bubble column reactor over a wide range of superficial gas velocities and solid holdup.

EXPERIMENTAL

A schematic representation of the bubble-column-reactor in which the ultrasonic investigation was conducted is shown in Figure 1. The transparent acrylic bubble-column-reactor has an internal diameter of 8.89 cm and a height of 290 cm. The column has six different axial locations for data collection. The ultrasonic signals are transmitted at 33 cm above the bottom of the gas distributor, which is a perforated-plate gas distributor with 15 x 1 mm diameter holes, along the center of the bubble-column-reactor. Experiments were conducted in batch-mode operation (stationary liquid-water and continuous flow of gas-nitrogen). Nitrogen bubbles were introduced through the gas distributor plate located at the bottom of the reactor. The nitrogen flow was controlled electronically to a maximum of 12 cm/s through a mass-flow controller. Glass beads from Cataphote, Inc., (10-37 μm in diameter with density of 2.46 g/cm^3) were used as the solid in the slurry. The solid holdup (solid weight/total slurry weight) was varied from 5 to 30 wt. % for each nitrogen flow in the reactor. To evaluate the accuracy of the ultrasonic technique for solid holdup measurement, an independent slurry sampling device was installed. The measurement was conducted by inserting a stainless steel tubing (0.775 cm. I.D.) horizontally into the center of the column at 0.635 cm above the path of the ultrasonic transmission. For each sampling, a 10 cm^3 of slurries sample was collected and analyzed for solid holdup characterization. The ultrasonic transmitter/receiver and the solid sampling device are positioned such that both means are measuring approximately the same hydrodynamic phenomena as shown enlarged areas in Figure 1. The detailed information of the ultrasonic unit has been reported elsewhere (2-4). Data were obtained with longitudinal waves at a frequency of 1 MHz using lithium niobate transducers. Both the transmitter and receiver were mounted directly inside the reactor wall at 33 cm above the gas distributor.

RESULTS & DISCUSSION

Figure 2 illustrates the effects of the superficial gas velocity (SGV) on the transit time [an arbitrary first distinct zero crossing time in the ultrasonic signal; the details have been described elsewhere (2)] and on the gas holdup in the reactor. The average gas holdup was determined by visual observations of the expanded bed height versus the static bed height. During this process, we have visually identified the various flow regimes since our bubble column is transparent. Basically, three flow regimes were identified in the bubble column. The homogeneous flow regime was observed when the SGV is 2.4 cm/sec or less. The average gas holdup in this regime was found to increase linearly from 0.015 at a gas velocity of 0.26 cm/sec to 0.093 at a SGV of 2.4 cm/sec. A transition flow regime exists between the SGV of 2.4 and 4 cm./sec. A slug flow regime is established when the velocity is 4 cm/sec or higher. The average gas holdup increased from 0.1 to 0.148 when the flow regimes changed from transition to slug flow. The transit time does not have an apparent correlation with the SGVs. It was approximately 72 μs at all SGVs and all flow regimes. Because what we measured was the signal that not transmitted through the nitrogen. Chang et al. (5) also reported that the amplitude of the transmitted sound pulses depends significantly on the number of bubbles; however, the transit time does not change with the void fraction. Uchida et al. (8) also measured the gas holdup in a bubble column for a gas-liquid system through determining the variation in transit time ratios. More recently, Warsito et al. (9) reported the measurement of gas holdup in a bubble column using the ultrasonic method. A change in transit time of 0.09 μs as the gas holdup increased from 0.05 to 0.1 was reported from their system. In current study, we did not observe such a change in transit time as the gas holdup increased from 0.05 to 0.1 in our system. The discrepancy may be due to different experimental setup or other factors. The small variation in transit time in this study is probably due to the experimental errors rather than the effect of nitrogen flow.

Figure 3 shows the change in the amplitude ratio of the transmitted ultrasonic signals A/A_0 and the local gas holdup in the reactor as a function of the SGV. A and A_0 are the amplitudes of the transmitted signals with and without the presence of nitrogen, respectively. Figure 3 suggests that the amplitude ratio is approximately an inverse exponential function of the SGV when the column is operated in the homogeneous flow regime (SGV of 2.4 cm/sec or less). No discernible relationship between A/A_0 and SGV could be found when the latter is higher than 2.4 cm/sec, i.e., while the column is operating in transition or slug flow regimes. When a large nitrogen bubble (slug) passes across the ultrasonic transmitted path, the transmitted signal will be reduced significantly. The transmitted signal will regain some amplitude immediately after the slug has passed through the transmitted path. Therefore, a large scatter of the A/A_0 ratio is observed at SGV of 2.4 cm/sec or higher. These phenomena could be observed while the column was operated in the transition or slug flow regimes. Chang et al. (5) measured void fractions up to 20% in bubbly air-water two phase flow using an ultrasonic transmission technique. Their results also showed that the A/A_0 ratio has exponential relationship with the void fraction and a function dependent on the bubble diameter. The effect of air bubble diameters on A/A_0 ratio was found to be significant where A/A_0 decreased with increasing bubble size. Bensler et al.(6) also conducted the measurement of interfacial area in bubbly flows in air-water systems by means of an ultrasonic technique. Their observations suggest that the A/A_0 ratio has an exponential relationship with the interfacial area and the scattering cross section, which depends on the bubble radius (a) and the wave number (k) of the ultrasonic wave surrounding the bubble. Our observations of A/A_0 in the nitrogen/water system are in qualitative agreement with those reported by Chang et al. (5) and Bensler et al.(6).

Figure 4 shows the effect of gas velocity (SGV) on amplitude ratio (A/A_0), transit time, and average gas holdup in three-phase systems (30 wt. % of glass bead/nitrogen/water) in the bubble column reactor. A and A_0 are the transmitted signals with and without the presence of solids, respectively. The similar flow regimes' patterns were also observed in the three-phase system (Figure 4) as those in a two-phase system (Figures 2 and 3). The fluctuation patterns of the A/A_0 along with different flow regimes in Figure 4 are similar to that in Figure 3. Unlike the constant transit time observed in Figure 2 for a two-phase system, the transit time in a three-phase system is related to the flow regimes operated and the presence of solids. The transit times were 70.56 and 70.52 μs, respectively, for superficial gas velocities of 0.537 and 1.611 cm/sec when the column is operated in the homogeneous flow regime. In the transition flow regime, the transit time increased from 70.8 to 70.88 μs as the SGV changed from 0.537 to 12.05 cm/sec suggests that there was variation in the concentration of solids in the ultrasonic path. For example, partial sedimentation occurred when the SGV was 2 cm/sec or less. Thus the concentration of solids should be high under these conditions. The detected constant transit time of approximately 70.88 μs in the slug flow regime suggests that there is a complete suspension of solids when the SGV is 4 cm/sec or higher. Kölbel and Realek (10) indicate that sedimentation will occur when the bubble column is operated under 2 cm/sec or less and the complete suspension of the solid will be established when the column is operated at 4 cm/sec or higher. Our ultrasonic observations are in good agreement with this finding.

Figure 5 illustrates the effects of solid holdup on the transit time measured at 33.65 cm above the gas distributor in the glass beads/nitrogen/water system at different SGVs. In this experiment, the SGV was systematically varied at any given initial solid holdup of 5, 10, 20, and 30 wt. % in the bubble column reactor. In general, the transit time varies with the variation of the superficial gas velocity for the SGV of 4 cm/sec or less at any given initial constant solid holdup loading in the reactor. The transit time was relative constant when the SGV is 4 cm/sec or higher. The transit times are around 71.96, 71.6, 71.12 and 70.88 μs for the solid holdup of 5, 10, 20, and 30 wt. % respectively, when the SGV is 4 cm/sec or higher. Therefore, the transit time can be utilized to determine the solid holdup when the column is operated in a complete suspension mode. The fluctuation of the transit time when the SGVs is 4 cm/sec or less may attribute to the both partial sedimentation and other factors which is under investigation

The fractional change of transit time [□ $t/t_o=(t_a-t_b)/t_o$] can be calculated on each individual transit time in Figure 5. From the fractional change of transit time, the solid holdup can be determined from the previous calibrated curve obtained from a stirred tank reactor [Figure 11. in (2)]. The determined solids holdup from these procedures and the solid holdup determined by the direct sampling are illustrated in Figure 6. The solid holdup measurements by the ultrasonic technique compared reasonably will with results obtained by the direct sampling techniques. Some discrepancies observed between these two techniques are probably due to the nature of these techniques. The ultrasonic technique measures the average solid holdup in the ultrasound path while direct sampling determines the collected local solid holdup.

CONCLUSIONS

An ultrasonic transmission technique has been developed to measure solid holdup in a gas-liquid-solid bubble column reactor. The results presented in this study show that the transit time of an ultrasonic signal is influenced by the variation of solid holdup and the operating conditions in the bubble column. The transit time can be correlated to the solid holdup. The variation of nitrogen flow has little influence on the observed transit time within the two-phase flow conditions studied. The ultrasonic technique is potentially applicable for solid holdup measurements in slurry-bubble-column reactors.

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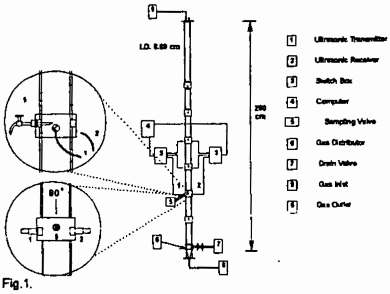

Fig.1.

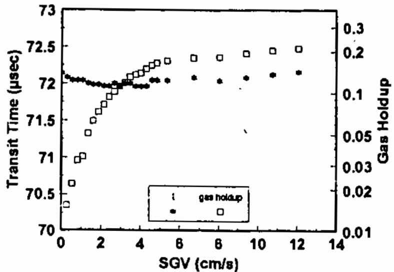

Fig. 2. Effect of superficial gas velocity (SGV) on transit time and gas holdup in nitrogen-water system.

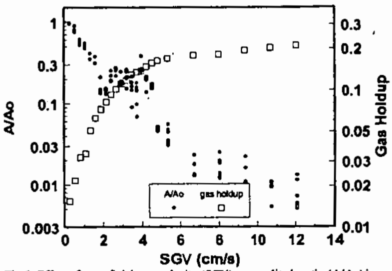

Fig. 3. Effect of superficial gas velocity (SGV) on amplitude ratio (A/Ao) in nitrogen-water system.

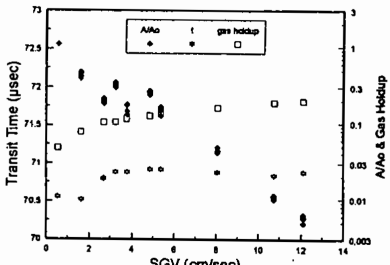

Fig. 4. Effect of superficial gas velocity (SGV) on amplitude ratio (A/Ao), transit time, and gas holdup in glass beads (30 wt.%)/nitrogen/water system.

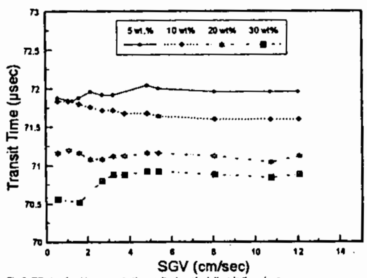

Fig 5. Effects of solids concentration on the transient time in the glass beads/nitrogen/water system.

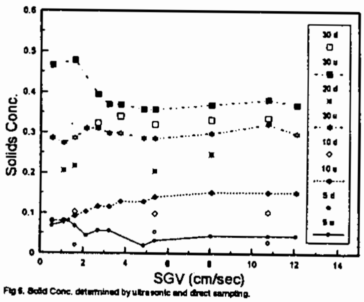

Fig 6. Solid Conc. determined by ultrasonic and direct sampling.