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Hydrodynamics of Three-Phase Slurry Fischer-Tropsch Bubble Column Reactors

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Quarterly Technical Progress Report for the Period 1 July 1989 - 30 September 1989

Dragomir B. Bukur, James G. Daly and Snehal A. Patel

Texas A&M University Department of Chemical Engineering College Station, TX 77843

September 25, 1989

Prepared for the Pittsburgh Energy Technology Center, the United States Department of Energy Under Contract No. DE-AC22-86PC90012 George Cinquegrane, Project Manager (PETC) John Shen, Program Manager (DOE/FE) Hydrodynamics of Three-Phase Slurry Fischer-Tropsch Bubble Column Reactors

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I. <u>Abstract</u>

During the past quarter we concentrated our efforts towards experiments in the large diameter (0.21 m ID) stainless steel column. The large column circuit was made ready for hot flow experiments. The feed section had to be modified to accommodate the new pump. Shakedown runs with water were conducted following instrument calibration. After the system performed satisfactorily with water, hot flow experiments were initiated. A total of seventeen runs were conducted during the quarter, three with water (no solids) and the remaining fourteen with Sasol wax (five without solids and 12 with 0-5 μ m iron oxide) as the liquid medium. No operational problems were encountered during these runs.

Two-phase runs with water and with Sasol showed a slight decrease in average gas hold-up when liquid circulation was introduced. When the bubble-cap distributor was used, instead of the perforated plate distributor, gas hold-ups increased for Sasol wax, and remained unchanged for water. There was good agreement between hold-up values obtained by DP cells and those obtained using the nuclear density gauge apparatus.

Prior to the initiation of the three-phase hot flow studies, slurry samples containing known amounts of solids in Sasol wax were analyzed using the Archimedean Principal for calibration purposes. There was good agreement between the measured and actual values of wt.% solids. Results from the three-phase experiments with the perforated plate distributor indicate an increase in average gas hold-up with an increase in solids concentration, and also with an increase in liquid circulation velocity. Hold-up values with the bubble-cap distributor were consistently higher than those obtained with the perforated plate distributor. No effect of liquid circulation on gas hold-up was observed when the bubble-cap distributor was used. The nuclear density gauge apparatus was used during these runs, and wall pressure fluctuation data were acquired, but have not yet been analyzed.

II. Objective and Scope of Work

The overall objective of this contract is to determine the effects of bubble column diameter, solids loading and particle size, and operating conditions (temperature, gas and liquid flow rates) on hydrodynamics of slurry bubble columns for Fischer-Tropsch synthesis, using a molten wax as the liquid medium. To accomplish these objectives, the following specific tasks will be undertaken.

Task 1 – Project Work Plan

The objective of this task is to establish a detailed project work plan covering the entire period of performance of the contract, including a detailed program schedule, analytical procedures, and estimated costs and manhours expended by month for each task.

Task 2 – Design and Construction of the Experimental Apparatus

The existing glass and stainless steel columns (0.051 m and 0.229 m in diameter, 3 m tall) that were constructed under our previous DOE contract (DE-AC22-84PC70027), will be modified and additions made in order to study the effect of continuous upward liquid flow. After the procurement of equipment and instrumentation, and construction of the unit is completed, a shakedown of test facilities will be made to verify achievement of planned operating conditions.

Task 3 – Measurement of Hydrodynamic Parameters by Conventional Techniques

In this task, the effects of operating conditions (liquid and gas superficial velocities, temperature), gas distributor, column diameter, and solids concentrations and particle size on hydrodynamic parameters in the glass and stainless steel columns will be determined. All experiments will be conducted using nitrogen at atmospheric pressure.

The hydrodynamic parameters that will be determined as a function of the independent variables mentioned above are: average gas hold-up, axial solids distribution, axial gas hold-up, flow regime characterization, and qualitative information on bubble size distribution.

<u>Task 4 – Application of a Gamma Radiation Density Gauge for Determining</u> Hydrodynamic Parameters

The objective of this task is to determine hydrodynamic parameters for the threephase system using a nuclear density gauge apparatus. A movable assembly mechanism and positioning racks for the two nuclear density gauges and detectors will be designed and constructed. Following the interfacing of the apparatus with an on-line microprocessor, the gauges will be calibrated using pure components (liquid wax and solid particles), and with known proportions of liquid and solid. After calibration, the following parameters will be obtained from experiments in the large stainless steel column: axial gas hold-up, axial concentration of solids, and qualitative information on flow regimes and bubble size distributions.

III. Summary of Progress

The large diameter column (0.21 m ID) was made ready during the first part of the quarter. The slurry withdrawal system was installed, and the feed section was modified to accommodate the new lobe type positive displacement pump from Tuthill Corporation (Chicago). The system was heat traced and instruments were calibrated before shakedown runs with water were conducted. No major problems were encountered during the shakedown runs and the subsequent hot flow runs. There was a minor problem with the stirrer assembly; which has been eliminated by installing a more rigid support for the stirrer. Also, the density gauge apparatus, associated electronics, and the movable assembly mechanism performed without any problems.

A total of seventeen runs were conducted during the quarter. Of these, the first three were conducted with water. These served as shakedown runs, and the various components of the system were tested out. The remaining fourteen runs were conducted using Sasol wax at 265°C. Two different batches of Sasol wax were used to complete the fourteen runs, and the system was cleaned out after completion of runs with a given batch. Solids were not used in the first five runs with Sasol, while 0-5 μ m iron oxide particles in varying concentrations were used in the remaining seven runs.

Slurry samples containing known weight fractions of solids (iron oxide or silica) in Sasol wax were used to check for the error associated with using the Archimedean Principal for estimating solids concentration. Results from these measurements showed good agreement between actual and measured values of solids concentration. However, when samples of used Sasol wax (that has been on stream for 72-144 h) were checked for their density, it was found that the density of this wax increased initially with time on stream (up to about 72 hours) and then reached a constant value. Since results from this procedure are very sensitive to wax density, we took corrective measures to prevent the change in density from affecting our results.

Experiments with water showed little or no effect of liquid circulation or of distributor type on average gas hold-up. For experiments conducted with Sasol wax as the liquid medium, the average gas hold-up increased with solids concentration (for both batch and continuous modes of operation). Average gas hold-up also showed a slight increase with liquid circulation rate, which was opposite of what was observed during earlier runs with FT-300 wax in the 0.05 m ID column. Experiments with Sasol wax in the 0.05 m ID column. Experiments with Sasol wax in the 0.05 m ID column showed little or no effect of liquid circulation on average gas hold-up. The bubble-cap distributor (with 7 caps, each having three 2 mm holes) also produced higher average hold-ups than did the 19 X 2 mm perforated plate distributor.

Liquid circulation velocity had a very small effect on axial gas hold-up profiles. The profiles show a gradient at low gas velocities, and become uniform at a gas velocity of 0.12 m/s. There was no obvious effect of the presence of solids on the axial gas hold-up profiles. Also, a change in liquid circulation velocity did not affect the axial gas hold-up profile. The axial gas hold-up values for the bubble-cap distributor were higher than those obtained with the perforated plate distributor. The axial solids distribution profiles were uniform for all experiments conducted with the 0-5 μ m iron oxide particles, irrespective of the gas or liquid velocity used.

Pressure fluctuation measurements were made in selected runs, but have not yet been analyzed. Hold-up measurements were also made with the nuclear density gauges. Results from experiments conducted with water and two-phase runs with Sasol wax have been analyzed. These results show that the two density gauges give comparable gas hold-up values, and these values are consistently higher than those obtained using pressure measurements (the relative difference was as high as 28% for tap water). Radial gas hold-up profiles were also obtained using the density gauges. These profiles are fairly uniform at low gas velocities, but show a maximum at the center of the column at higher gas velocites. The profiles were similar at different heights along the column.

IV. Detailed Description of Technical Progress

A. Task 3 - Measurement of Hydrodynamic Parameters by Conventional Techniques

A.1. Overview of Bubble Column Operations

Once experiments in the small diameter (0.05 m ID) column were completed, we removed the lines, values and pneumatic actuators associated with the slurry sample withdrawal system, and moved them to the 0.21 m ID column circuit. The slurry feed section (i.e., the section between the storage tank and the column) was modified to accommodate the new lobe type positive displacement pump from Tuthill Corporation (Chicago). This included the installation of larger diameter (2 inch OD) inlet and outlet pipes for the pump. Following hardware modifications, the system was heat traced, and electrical connections for the pump, controllers and heaters were completed and tested. The system was then insulated.

An additional pressure transducer readout was procured and installed. With the additional readout it is now possible to monitor and record pressures at three axial locations simultaneously. Once the column was ready, the instruments were calibrated. The mass flow meter was calibrated using nitrogen. The pressure transducers were calibrated using different heights of water in the column. The actual height of water in the column was measured using the density gauge (which was already installed on the movable assembly mechanism). The density gauges were also calibrated and readings were taken at various combinations of axial and radial locations with the column empty and with the column filled with the liquid being used (water and wax).

No modifications to the large diameter column circuit were necessary during the course of the experiments. There were no problems encountered with settling of solids in process lines during the runs. The only change made was to the stirrer support system on the storage tank. The bearing assembly on the stirrer showed signs of wear and tear after the first few experiments. We replaced the bearings with pillow blocks which gave more stability to stirrer. Once these modifications were made, the stirrer performed with no additional problems. The pump operated satisfactorily during the continuous mode of operation. We had to remove the insulation from the pump to prevent it from overheating, and there was some leakage of wax from the seals on the drive shafts, which is expected. For the most part we were able to control slurry flow rate without any problem, and the flow rate remained fairly steady for a given gas flow rate. Some adjustments to the flow rate were necessary after the gas velocity was changed, probably due to the change in liquid head in the column (different void fractions at different gas velocities).

The movable assembly mechanism performed flawlessly during the experiments. The secondary electronics used to process the signals from the two density gauges showed little or no drift over extended time periods. This was checked by scanning the empty column after every alternate run.

During the hot flow experiments (batch and continuous modes of operation) temperature control was very good. We were able to maintain all temperatures within $\pm 3^{\circ}$ C of the desired values. Some damping of the pressure signal from the wall mounted transducers was observed when slurries with high solids concentrations were used, probably because of partial blockage of the short tube connecting the column to the transducers with settling solids. We therefore had to clear the tube with nitrogen backpressure whenever this happened.

A.2. Calibration of Slurry Samples using the Archimedean Principal

Samples with known weights of solids in fresh Sasol wax were prepared. Two grades of solids were used to prepare the samples, i.e., 0-5 μ m iron oxide, and 20-44 μ m silica. Eight samples were prepared with each solids type, containing solids concentrations in the range 3-35 wt.%. The Archimedean Principal was described earlier (Quarterly Technical Progress Report for April-June 1988), and requires the weight of the slurry sample and the weight loss when the sample is immersed in acetone. Using these values along with the densities of wax, solids and acetone, the weight fraction of solids is determined using

$$w_{s} = \frac{\rho_{s} - \left(\frac{\rho_{w}\rho_{s}}{\rho_{acet}}\right)\left(\frac{m_{1} - m_{2}}{m_{s\ell}}\right)}{\rho_{s} - \rho_{w}}$$
(1)

The density of fresh Sasol wax (solidified) was first measured using the Archimedean Principal and found to be 0.9303 g/cc. Table 1 shows results for slurry samples containing 0-5 μ m iron oxide. The percent error between actual solids concentrations and those obtained by the Archimedean Principal range between -1.02% (for the sample containing a nominal solids concentration of 20 wt.%) and 0.77% (for the sample containing 10 wt.% solids). The mean error for the eight samples is -0.28% and the standard deviation for the error is 0.62%. The density of wax was adjusted to minimize the mean error for the samples, and a value of 0.9305 g/cc gave the best results. This compares very well with the value of 0.9303 g/cc obtained for pure Sasol wax. Results for slurry samples containing 20-44 μ m silica particles are shown in Table 2. The percent error for these samples ranged between -0.83% (at a nominal solids concentration of 15 wt.%) and 0.60% (at a solids concentration of 10 wt.%), and the mean error was -0.11% with a standard deviation of 0.48. The density of Sasol wax that gave the minimum mean error was 0.9306 g/cc, which also is in excellent agreement with the value of 0.9303 g/cc for pure Sasol wax.

The above results indicate that the Archimedean Principal can be used to estimate solids concentration in slurries of solids in Sasol wax with good accuracy. In the above analysis we have assumed that the density of Sasol wax remains constant for the duration of experiments with a given batch of wax; however, it is likely that some change in wax composition would take place with time on stream. This is based on our observation that Sasol wax contains an unusually higher fraction of light components when compared to FT-300 wax, and these light components evaporate when the slurry is held at 265°C for extended periods of time (during multiple runs with a given batch of wax). We took two samples of used Sasol from the first batch of wax used in our experiments (i.e., wax that was on stream for approximately 72 and 144 hours), and measured the densities of these samples. The values were 0.9412 and 0.9405 g/cc, respectively, for the two samples, indicating that once the light components have left the system wax density approaches a constant value. Since Sasol wax undergoes this change in density, we performed sensitivity analysis to assess the impact this had on solids concentrations estimated using the Archimedean Principal.

Tables 3 and 4 show results from the sensitivity analysis calculations for the 0-5 μ m iron oxide and 20-44 μ m silica, respectively. Estimated solids concentrations were obtained using different values for wax density in Eq. 1. These results clearly indicate that the estimates are very sensitive to wax density, particularly for samples with low solids concentrations. The mean error when the density of used Sasol wax is used is as high as 13%. To alleviate this problem we have decided to use the higher density for Sasol wax once the wax has been on stream for over 72 hours, and use the lower density prior to that. The accuracy of the weight percent solids determination procedure can indirectly be checked by the wax and solids inventories that are performed during and after each run. Any underestimation of solids content would inflate the amount of wax present in the system. We were able to monitor the solids inventory in the system fairly well during the nine experiments with small iron oxide in Sasol wax.

A.3. Experimental Results

A total of seventeen experiments were conducted in the 0.21 m ID stainless steel column during the reporting period. The conditions used in these experiments are summarized in Table 5. The three experiments with tap water served as shakedown runs for the bubble column apparatus. The next series of experiments were at 265°C with Sasol wax, and following the successful completion of these two-phase runs, a fresh batch of Sasol wax was charged and three-phase experiments were initiated. Superficial gas velocities of 0.02, 0.04, 0.06 and 0.09 m/s were used (in the increasing order) for the experiments with water and those from the first batch of Sasol wax. For experiments using the second batch of wax, measurements were also made at 0.12 m/s, and decreasing order of gas velocities were used, i.e., the run was started at 0.12 m/s and gas velocity was then changed to 0.09 m/s and so on.

Average Gas Hold – ups

Figure 1 shows hold-up values for the three runs conducted using tap water. The first two runs were conducted using the 19 X 2 mm perforated plate (batch mode and using u_{ℓ} =0.005 m/s, respectively), and the third run was conducted using the bubble-cap distributor (batch mode). Hold-up values from the three runs are very similar, with no effect of distributor type and very little effect of liquid circulation. Hold-ups in the continuous mode of operation (u_{ℓ} =0.005 m/s) are slightly lower than those obtained in the batch mode of operation.

Hold-up values from two-phase experiments with Sasol wax are shown in Figure 2. Results from the three runs with the 19 X 2 mm perforated plate distributor ($u_{\ell} = 0, 0.005$, and 0.02 m/s, respectively), and single run with the bubble-cap distributor ($u_{\ell}=0.005$ m/s), are shown in the figure. There appears to be no effect of liquid circulation on gas hold-up for Sasol wax, with similar values obtained from all three runs with the perforated plate distributor. Unlike FT-300 wax, Sasol wax does not produce foam and is coalescing in nature, therefore the introduction of liquid circulation did not promote the coalescence process as was the case with the FT-300 wax runs in the 0.05 m ID column (Quarterly Technical Progress Report for April-June 1988). This could possibly explain the absence of any effect due to liquid circulation. The bubble-cap distributor resulted in hold-ups that were significantly higher than those obtained with the perforated plate under similar conditions. At this point it is not clear as to why this distributor increased the gas hold-up. It is possible that the wax composition changed somewhat with time on stream. The run with the bubble-cap distributor was the last run with this batch of wax, and the higher hold-ups could be due to aging effects. We plan to repeat this experiment with a fresh batch of Sasol wax to ascertain this behavior.

The effect of solids concentration on average gas hold-up for experiments conducted in the batch mode of operation using the 0-5 μ m iron oxide particles is show in Figure 3. The lowest gas hold-ups were obtained during the run with no solids, and consistently higher hold-ups were obtained in the presence of solids. There is no definitive trend in hold-up behavior with the amount of solids present, and the highest gas hold-ups were obtained when a slurry containing 20 wt.% solids was used. At a gas velocity of 0.12 m/s, hold-up values for all cases are similar. The increase in gas hold-up with the addition of solids was also observed in experiments conducted in the small diameter (0.05 m ID) column (Quarterly Technical Progress Report for April-June 1988). Figure 4 shows the effect of solids concentration on gas hold-up for experiments conducted in the continuous mode of operation using a superficial liquid velocity of 0.005 m/s. Hold-up values for the run without solids were similar to those for the slurry with 20 wt.% solids, while values for the slurry containing 30 wt.% solids were substantially higher. When a superficial liquid velocity of 0.02 m/s was used, hold-up values from the two runs (0 and 20 wt.% solids) were fairly similar.

The effect of liquid circulation on average gas hold-ups in the presence of solids is shown in Figures 5 and 6. Figure 5 shows results from three runs conducted using superficial liquid velocities of 0, 0.005 and 0.02 m/s, respectively, with a slurry containing 20 wt.% of solids. The effect of liquid circulation on gas hold-up is minimal, with slightly higher hold-ups observed when liquid circulation is introduced. Figure 6 compares gas hold-ups for two runs conducted using superficial liquid velocities of 0

and 0.005 m/s, respectively, using a slurry containing 30 wt.% solids. In this case the hold-ups increased significantly when liquid circulation was introduced. This is surprising since previous experiments in the small diameter (0.05 m ID) column with FT-300 wax showed the opposite trend. For FT-300 wax, liquid circulation always decreased the average gas hold-up. Experiments with a slurry of 20 wt.% 20-44 μ m iron oxide particles in Sasol wax did not show any significant effect of liquid circulation during runs in the 0.05 m ID column. The opposite trend obtained with Sasol wax could be attributed to differences in the foaming capacity of the two media (i.e., FT-300 wax has a tendency to foam, whereas Sasol wax is a coalescing medium). It would be premature to attribute these differences to the effect of column diameter, until additional data from experiments with FT-300 wax in the large column are available.

The effect of distributor type on gas hold-up is illustrated in Figures 7 and 8 for batch mode and continuous mode experiments, respectively. In the batch mode of operation (Figure 7), a slurry containing 20 wt.% of solids showed little effect of distributor type, and hold-ups from the experiment with the bubble-cap distributor were only slightly higher than those observed when the 19 X 2 mm perforated plate distributor was used. The difference between results from experiments with the two distributors was more apparent when a superficial liquid velocity of 0.005 m/s was used (Figure 8). In this case, gas hold-ups with the bubble-cap distributor were somewhat higher than those obtained with the perforated plate distributor.

In summary, average gas hold-ups increase with solids concentration (for both batch and continuous mode of operations). Liquid circulation also increases the gas hold-up; however, the increase is only slight. Finally, the bubble-cap distributor gives higher gas hold-ups than those obtained using the perforated plate distributor.

Axial Gas Hold – ups and Solids Distribution Profiles

Figure 9 shows axial gas hold-ups for the batch mode run with Sasol wax without

solids. There is some non-uniformity in the axial hold-up profile at low gas velocities (0.02 and 0.04 m/s), while at 0.12 m/s the profile is fairly uniform. The behavior is similar for three-phase batch mode runs as well (i.e., in the presence of 0-5 μ m iron oxide particles with different solids concentrations) as shown in Figure 10a for a slurry containing 20 wt.% of 0-5 μ m iron oxide. For the batch mode run using the bubble-cap distributor, axial gas hold-up profiles showed slightly greater gradients (Figure 11a) than did profiles from experiments conducted using the 19 X 2 mm perforated plate distributor. These may be responsible for the slightly higher hold-ups observed with this distributor. Axial solids concentration profiles for all runs conducted using the 0-5 μ m iron oxide particles (batch and continuous mode) were fairly uniform at all gas velocities (e.g., Figures 10b and 11b). This is similar to the observations made in the 0.05 m ID column with this solids type and size.

The effect of liquid circulation velocity on axial gas hold-up is shown in Figure 12 for a slurry containing 20 wt.% of 0-5 μ m iron oxide. Results for superficial liquid velocities of 0, 0.005 and 0.02 m/s are very similar, with slightly higher values at the higher liquid velocities. This follows from the average gas hold-up values shown in Figure 5 for these runs, where hold-ups at u_l=0.02 m/s were only slightly higher than values at u_l=0 m/s.

Pressure Fluctuation Measurements

Pressure fluctuations were measured at different axial locations during the hot flow runs. The analysis of these results will be presented in a future report.

B. <u>Task 4 – Application of a Gamma Radiation Density Gauge for Determining</u> <u>Hydrodynamic Parameters</u>

The nuclear density gauge was used to obtain axial and radial slurry density profiles during the runs conducted in the 0.21 m ID column. We are in the process of analyzing data from these measurements; however, some of the results from the two-phase experiments with water and with Sasol are presented here. Calibration data for the nuclear density gauge apparatus with solids and additional results, as they become available, will be included in future reports.

For the two-phase experiments, data from measurements with a single density gauge are sufficient to determine volume fractions of gas and liquid. Nevertheless, we acquired data with both density gauges and compared hold-ups from these measurements with those obtained from conventional measurements (i.e., pressure measurements). The gas hold-up at a given height in the column was determined using a weighted average of the six point measurements of hold-up made at different radial locations at that height. The weights used in the averaging process were proportional to the fraction of the column cross-sectional area traversed by the beam when positioned at the various radial locations (i.e., product of the beam path length through the column and the spacing between adjacent radial locations). The average gas hold-up for the entire column was obtained by taking the arithmetic mean of the hold-ups at the different heights.

Table 6 summarizes results obtained from density gauge measurements for the batch mode run conducted with tap water. Comparisons with results obtained using pressure measurements are also given. The run was conducted in the batch mode of operation, and density gauge measurements were made at heights of 0.9 and 1.5 m above the distributor for gas velocities of 0.02, 0.04, 0.06 and 0.09 m/s. Hold-up values obtained from density gauges are higher than those obtained from pressure measurements for most cases, with differences as high as 28% at a height of 1.5 m for the Cesium-137 source at a gas velocity of 0.04 m/s. Results from the two density gauges are similar to each other at both heights.

Figure 13 shows radial hold-up profiles obtained at a height of 0.9 above the distributor using the nuclear density gauges at various gas velocities for the two-phase batch run with Sasol wax. Measurements were made at six radial locations at each gas velocity. For each case results from the two density gauges are presented along with the average values. Once again the density gauges gave very similar results. The radial hold-up profile is fairly uniform at a gas velocity of 0.02 m/s, which is expected since flow is in the homogeneous bubbly regime at these velocities. However, as gas velocity increases, the profile becomes slightly **non-uniform with** higher hold-ups in the center of the column. This corresponds to an increase in the number of large bubbles present in the system, which have a tendency to move along the center of the column. The trends at a height of 1.5 m above the distributor were very similar to those observed at 0.9 m above the distributor.

Average gas hold-up values obtained using the various methods are compared in Figure 14. The solid symbols represent hold-up values from pressure measurements (solid circles) and the average of values from the two density gauges (solid squares). Also shown are hold-up values for the individual density gauges, as well as values obtained when the density gauge was used to measure the expanded and static heights. There is very good agreement between the different sets of values, with values from pressure measurements being consistently lower than those from the density gauges.

Figure 14 compares average gas hold-up values obtained using pressure measurements with those obtained with the density gauges. There is very good agreement between the different sets of values, with values from pressure measurements being consistently lower than those from the density gauge using the Cs-137 source and comparable to those obtained with the density gauge using the Co-60 source.

V. <u>Nomenclature</u>

- d_p particle diameter, cm
- d_c column diameter, m
- m₁ weight of slurry sample and support (sample in air), g
- m₁ weight of slurry sample and support (sample in acetone), g
- $m_{s\ell}$ weight of slurry sample, g
- t time, s
- ug superficial gas velocity, m/s
- u_{ℓ} superficial liquid velocity, m/s
- ws weight fraction solids in slurry

Greek letters

ϵ_{g}	gas phase hold-up					
$ ho_{\sf acet}$	acetone density (g/cm^3)					
$ ho_{\sf s}$	solids density (g/cm ³)					
ρw	wax density (g/cm ³)					

Nominal wt.%	Sample wt., m _{sl} (g)	m ₁ (g)	m ₂ (g)	Actual wt.%	Meas. wt.% w _s	% Error ^b	
3	24.232	463.925	443.809	3.03	3.02	-0.34	
7	25.128	464.824	444.614	6.78	6.74	-0.65	
10	26.912	466.603	445.548	9.81	9.89	0.77	
15	28.254	467.944	446.842	14.94	14.99	0.31	
20	31.731	471.421	448.841	20.26	20.05	-1.02	
25	31.968	471.664	449.894	24.63	24.45	-0.72	
30	32.834	472.528	451.217	28.95	29.04	0.32	
35	32.295	471.986	452.340	35.21	34.90	-0.89	
$\begin{array}{rcl} \text{mean error} = & -0.28\\ \text{std. dev.} = & 0.62 \end{array}$							

Table 1. Weight Fraction Determination using the Archimedean Principal^a (0-5 μ m Iron Oxide in Sasol)

 $^{\rm a}$ densities used: $\rho_{\rm acet}$ = 0.792 g/cc, $\rho_{\rm w}$ = 0.9305 g/cc, $\rho_{\rm s}$ = 5.1 g/cc

^b %Error = 100(Actual wt.% - Meas. wt.%)/Actual wt.%

Nominal wt.%	Sample wt., m _{sl} (g)	m ₁ (g)	m ₂ (g)	Actual wt.%	Meas. wt.% w _s	% Error ^b	
3	22.814	462.505	443.463	2.97	2.97	0.00	
7	23.556	463.246	444.105	7.02	6.97	-0.71	
10	25.628	465.323	444.927	9.94	10.00	0.60	
15	27.815	467.505	446.122	15.03	14.91	-0.83	
20	29.947	469.645	447.442	19.94	19.86	-0.41	
25	31.528	471.226	448.730	24.81	24.91	0:39	
30	31.973	471.664	449.738	29.88	29.88	0.18	
35	31.538	471.233	450.502	35.12	35.08	-0.10	
$\begin{array}{rcl} \text{mean error} = & -0.11\\ \text{std. dev.} = & 0.48 \end{array}$							

Table 2. Weight Fraction Determination using the Archimedean Principal^a (20-44 μ m Silica in Sasol)

^a densities used: $\rho_{acet} = 0.792$ g/cc, $\rho_{w} = 0.9306$ g/cc, $\rho_{s} = 2.65$ g/cc

^b %Error = 100(Actual wt.% - Meas. wt.%)/Actual wt.%

ρ _w (g/cc)	Fresh: 0.9305		Used (72h): 0.9412		Used (144h): 0.9405	
Nominal	Meas. wt.%	% Error	Meas. wt.%	% Error	Meas. wt.%	%Error
3	3.02	-0.34	1.65	-45.47	1.74	-42.51
7	6.74	-0.65	5.42	-20.05	5.51	-18.77
10	9.89	0.77	8.61	-12.18	8.70	-11.33
15	14.99	0.31	13.79	-7.71	13.87	-7.18
20	20.05	-1.02	18.93	-6.58	19.00	-6.22
25	24.45	-0.72	23.39	-5.04	23.46	-4.76
30	29.04	0.32	28.04	-3.13	28.11	-2.91
35	34.90	-0.89	33.98	-3.50	34.04	-3.33
mean err std. dev.	-0.28 0.62		-12.96 13.35		-12.13 12.47	

Table 3. Sensitivity of Results from Archimedean Procedure to Wax Density^a (0-5 μ m Iron Oxide in Sasol)

 $^{\rm a}$ densities used: $\rho_{\rm acet}$ = 0.792 g/cc, $\rho_{\rm s}$ = 5.1 g/cc

$\rho_{\rm W} ({\rm g/cc})$	Fresh: 0.9305		Used (72h): 0.9412		Used (144h): 0.9405	
Nominal	Meas. wt.%	% Error	Meas. wt.%	% Error	Meas. wt.%	%Error
3	2.97	0.00	1.26	-57.71	1.37	-53.87
7	6.97	-0.71	5.33	-24.12	5.44	-22.56
10	10.00	0.60	8.41	-15.40	8.52	-14.34
15	14.91	-0.83	13.40	-10.83	13.50	-10.17
20	19.86	-0.41	18.44	-7.51	18.54	-7.04
25	24.91	0.39	23.58	-4.95	23.67	-4.60
30	29. 9 3	0.18	28.70	-3.96	28.78	-3.68
35	35.08	-0.10	33.94	-3.37	34.01	-3.15
mean err std. dev.	-0.11 0.48		-15.98 17.07		-14.93 15.94	

Table 4. Sensitivity of Results from Archimedean Procedure to Wax Density^a (20-44 μ m Silica in Sasol)

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 $^{\rm a}$ densities used: $\rho_{\rm acet}$ = 0.792 g/cc, $\rho_{\rm s}$ = 2.65 g/cc $^\circ$

Run No.	Liquid Medium	Solids type	Solids size (µm)	Solids conc. (wt.%)	u _ℓ b (m/s)
W110-00000-9S	Water	none	-	-	0
W125-00000-9S	21	none	-	_	0.005
W130-00000-9S ^c	37	none	-	-	0
S110-00000-9S	Sasol	none	_	_	0
S125-00000-9S	"	none	-		0.005
S132-00000-9S	"	none	-	-	0.02
S145-00000-9Sc	25	none	-	-	0.005
S210-00000-9S	Sasol	none	-	_	0
S220-FES10-9S	11	lron oxide	0-5	10	0
S230-FES20-9S	"	"	27	20	0
S245-FES20-9S	22	n	**	n .	0.005
S252-FES20-9S	n	. 17		22	0.02
S260-FES20-9S°	n	**	***	"	0
S275-FES20-9S°	n	**	22	11	0.005
S280-FES20-9S	"	"	21	22	0
S290-FES30-9S	n	11	11	30	0
S2105-FES30-9S	27	**	27	30	0.005

Table 5. Conditions used during runs in the 0.23 m ID column^a

 ${}^{a}u_{g}=0.02,\,0.04,\,0.06$ and 0.09 in first 7 runs, and 0.02, 0.04, 0.06, 0.09, and 0.12 m/s in remaining 10 runs ^bsuperficial liquid velocity

^cbubble cap distributor used instead of the perforated plate

ug (m/s)	DP Cells	Co-60		Co-60 Cs-		s-137		
	€g	€g	% dev. ^b	€g	% dev. ^b			
Hold-ups at	Hold-ups at 0.9 m above distributor							
0.02	0.047	0.042	-10.6	0.043	-8.5			
0.04	0.075	0.075	0.0	0.062	-17.3			
0.06	0.100	0.109	9.0	0.100	0.0			
0.09	0.132	0.142	7.6	0.136	3.0			
Hold-ups at	Hold-ups at 1.5 m above distributor							
0.02	0.046	0.053	15.2	0.050	8.7			
0.04	0.076	0.087	14.5	0.097	27.6			
0.06	0.102	0.117	14.7	0.123	20.6			
0.09	0.122	0.126	3.3	0.144	18.0			
Average hold-ups for whole column								
0.02	0.040	0.047	17.5	0.047	17.5			
0.04	0.069	0.081	17.4	0.080	15.9			
0.06	0.096	0.113	17.7	0.112	16.7			
0.09	0.123	0.134	8.9	0.140	13.8			

Table 6. Comparison of Hold-up Results for Water: DP vs. NDG^a

^aDP: using pressure measurements; NDG: nuclear density gauges ^b% dev. = $100(\epsilon_{gDP} - \epsilon_{gNDG}) / \epsilon_{gDP}$



Figure 1. Effect of superficial gas velocity and distributor type on average gas holdup.



Figure 2. Effect of superficial gas velocity and distributor type on average gas holdup.



Figure 3. Effect of superficial gas velocity and solids concentration on average gas holdup. (batch mode of operation)



Figure 4. Effect of superficial gas velocity and solids concentration on average gas holdup. (continuous mode of operation)



Figure 5. Effect of superficial gas velocity and liquid velocity on average gas holdup. (20 wt.% solids)



Figure 6. Effect of superficial gas velocity and liquid velocity on average gas holdup. (30 wt.% solids)



Figure 7. Effect of superficial gas velocity and distributor type on average gas holdup. (batch mode of operation)



Figure 8. Effect of superficial gas velocity and distributor type on average gas holdup. (continuous mode of operation)



Figure 9. Effect of superficial gas velocity on axial gas holdup.



Figure 10. Effect of superficial gas velocity on axial gas hold-up and solids distribution. (19 X 2 mm perforated plate distributor)



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Figure 11. Effect of superficial gas velocity on axial gas hold-up and solids distribution. (Bubble-cap distributor)



Figure 13. Effect of superficial gas velocity on radial gas hold-up.

Figure 14. Comparison of average gas hold-ups from different techniques.