

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

Quarterly Technical Progress Report
for the Period 1 July 1988 - 30 September 1988

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

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

October 25, 1988

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)

DOE/PC/90012-5

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

Quarterly Technical Progress Report
for the Period 1 July 1988 - 30 September 1988

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

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

October 25, 1988

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)

NOTICE

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States nor any agency thereof, nor any of their employees, makes any warranty, expressed or implied, or assumes any legal liability or responsibility for any third party's results of such use of any information, apparatus, product or process disclosed in this report, or represents that its use by such a third party would not infringe privately owned rights.

PATENT STATUS

U.S./DOE Patent Clearance is not required prior to the publication of this document.

TECHNICAL STATUS

This technical report is being transmitted in advance of DOE review and no further dissemination or publication shall be made of the report without prior approval of the DOE Project/Program Manager.

TABLE OF CONTENTS

I.	Abstract	1
II.	Objective and Scope of Work	3
III.	Summary of Progress	5
IV.	Detailed Description of Technical Progress	8
	A. Task 3 - Measurement of Hydrodynamic Parameters by Conventional Techniques	8
	A.1. Physical Property Measurements	8
	A.2. Overview of Bubble Column Operations	9
	A.3. Experimental Results	14
	B. Task 4 - Application of a Gamma Radiation Density Gauge for Determining Hydrodynamic Parameters	21
V.	References	23
	Tables and Figures	24
	APPENDIX A - Axial Gas Hold-up and Solids Distribution Profiles	36

I. Abstract

During the past quarter, we sieved approximately 35 pounds of large iron oxide particles (35 – 44 μm) for experiments in the small stainless steel column. Due to the excessive amount of time needed to obtain the particles in the desired range (i.e. took approximately 8 hours to obtain 4 - 5 pounds of the desired product), we decided to have Vortec Product Co. of Long Beach, California grind/ classify the particles for us. The solids were classified in the range 20 – 44 μm as opposed to 35 – 44 μm to reduce the cost.

A new stirrer was built for the large storage tank. It was tested using large iron oxide particles with 60 % of the particles greater than 44 μm . Results from the test indicate that we are able to suspend the solid particles.

Work on Task 3 (Measurement of Hydrodynamic Properties by Conventional Techniques) continued during the past quarter. A total of nine experiments with FT-300 wax and solids were conducted. Five experiments were conducted with 0 – 5 μm silica particles, three with 35 – 44 μm iron oxide particles and one with 20 – 44 μm silica particles. Six experiments were conducted in the continuous mode of operation and three experiments were conducted in the batch mode of operation.

Results from these experiments indicate that for a given system, the superficial liquid velocity has a greater effect on the average gas hold-up, compared to the effect of solids concentration. For experiments conducted in the batch mode of operation the axial solids distributions decreased with an increase in height above the distributor; whereas, for experiments conducted in the continuous mode of operation the distributions remained essentially uniform. The axial gas hold-ups increased with height above the distributor for all runs. For the batch experiment with large silica particles, which was conducted in a decreasing order of gas velocities, no foam was produced; whereas, for all other experiments conducted in the batch mode of operation (all with increasing

gas velocities), a substantial amount of foam was produced at gas velocities greater than 0.02 m/s. Some problems with solids settling were encountered during these runs. Settling of solids in the storage tank was reduced by the adding an additional propeller to the stirrer. We are currently modifying the expansion unit and calibration unit to minimize settling in these units. Viscosities and surface tensions were obtained for some of the samples.

Operational problems with the pump were encountered following the last run with small silica. At the beginning of the next run (large silica), a flowrate of 0.005 m/s could not be maintained. Inspection of the pump indicated that cooling water was leaking into the slurry chamber causing the pump to cavitate. The pump was cleaned, rebuilt and two experiments with large iron oxide were conducted before the pump failed (worn idler gear, wear plates, and bearings).

Work on Task 4 was continued during the past quarter. The movable assembly mechanism which will be used to transport the nuclear density gauges along the column has been built and tested. We received our license from the Radiological Safety Office (RSO) for the use of radioactive materials. The radioactive sources were installed in the apparatus by the RSO and the laboratory was surveyed. The radiation levels in the laboratory were acceptable.

The data acquisition system was installed and used to collect data from the Validyne DP cells during some of the experiments. A low pass filter was purchased which will be used in conjunction with the Sensotron DP cell. The ratemeters were connected to the data acquisition system and calibrated using a constant frequency generator.

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.

Task 4 – Application of a Gamma Radiation Density Gauge for Determining Hydrodynamic Parameters

The objective of this task is to determine hydrodynamic parameters for the three-phase 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

During the past quarter, we sieved 35 pounds of large iron oxide particles (35 – 44 μm) for experiments in the small stainless steel column. We were able to obtain approximately 4 – 5 pounds of particles within the desired range over an eight hour period. Initially, we were planning to sieve all particles ourselves, but due to the amount of time taken to obtain the iron oxide particles for experiments in the small stainless steel column, we decided to have Vortec Product Co. of Long Beach, California sieve all silica particles and iron oxide particles for experiments in the large column. In order to reduce the cost of sieving, we had the solids classified in the size range 20 – 44 μm . According to Vortec, there could be as much as 10 % of the particles greater than 44 μm and 10 % of the particles less than 20 μm . Samples of the particles sieved by Vortec were taken to Kansas State University, and analyzed to determine the size range of the particles. Approximately 70 % of the iron oxide particles were in the specified range (i.e. 20 – 44 μm). Approximately 70 % of the silica particles were in the size range 10 – 44 μm . All particles were less than 70 μm .

The original stirrer purchased for the large storage tank was tested. Results from the test indicated that it would not be capable of suspending the large iron oxide particles. A new stirrer was designed comprised of a 3/4 HP motor and an 8 inch diameter propeller (8 degree blade pitch). The new stirrer was tested and was able to maintain suspension of the large iron oxide particles.

Work on Task 3 (Measurement of Hydrodynamic Parameters by Conventional Techniques) was continued during the past quarter. We received the new idle gear for the pump and rebuilt the pump. A total of nine experiments were conducted before the pump became inoperable. Five of the experiments were conducted with small silica particles (0 – 5 μm), three with large iron oxide particles (35 – 44 μm), and one with large silica particles (20 – 44 μm). All experiments were conducted using FT-300 wax

as the liquid medium at 265 °C. All experiments were conducted in an increasing order of gas velocities except for the experiment with large silica particles. It was conducted in decreasing order of gas velocities. The gas velocities employed for all experiments were 0.02, 0.04, 0.06, and 0.09 (and/or 0.12) m/s. Four of the five experiments conducted with small silica particles and two of the three experiments conducted with large iron oxide particles were performed in the continuous mode of operation. All other experiments were performed in the batch mode of operation. Viscosities and surface tensions of some of the samples withdrawn during our experiments were obtained.

Regardless of the system studied, the slurry velocity had a greater effect on the gas hold-up than the solids concentration. Increasing the superficial slurry velocity from 0.0 m/s to 0.005 m/s caused a significant decrease in the gas hold-up. Increasing the slurry velocity to 0.02 m/s had little effect on the gas hold-up compared to experiments conducted using a slurry velocity of 0.005 m/s. Experiments with 10, 20 and 30 wt % silica particles at a slurry velocity of 0.005 m/s showed a slight decrease in gas hold-up with an increase in solids concentration.

Axial gas hold-ups increased with both an increase in the superficial gas velocity and an increase in height above the distributor. The increase in the axial gas hold-up with an increase in height above the distributor was most pronounced for batch experiments at gas velocities greater than 0.02 m/s. Foam was produced in all batch experiments except for the experiment with large silica particles which was conducted in a decreasing order of gas velocities.

Solids concentrations in the column remained uniform for experiments conducted in the continuous mode of operation. However, for experiments conducted in the batch mode of operation, the axial solids concentration decreased with an increase in height above the distributor. The gradient was most pronounced for experiments conducted with large particles.

Following the experiments with small silica particles, an additional propeller was added to the small storage tank stirrer to try and improve the suspension of solids in the storage tank. A significant amount of solids settled in the system during the experiments with large iron oxide particles. Examination of the system following the completion of the experiments conducted in the continuous mode of operation with large iron oxide particles revealed that a significant amount of solids were settling in the expansion unit and calibration unit. Modification of these units are underway to try to minimize the amount of solids which settle in these units.

Operational problems with the pump were encountered following the last run with small silica. At the beginning of the next run (large silica), a flowrate of 0.005 m/s could not be maintained. Inspection of the pump indicated that cooling water was leaking into the slurry chamber causing the pump to cavitate. The pump was cleaned, rebuilt and two experiments with large iron oxide were conducted before the pump failed (worn idler gear, wear plates, and bearings).

Work on Task 4 was continued during the past quarter. The movable assembly mechanism which will be used to transport the nuclear density gauges along the column has been built and tested. We received our license from the Radiological Safety Office (RSO) for the use of radioactive materials. A plexiglass chamber was constructed around the large stainless steel column and movable assembly mechanism to prevent anyone from coming too close to the radioactive sources. The radioactive sources were installed in the apparatus by the RSO and the laboratory was surveyed. The radiation levels in the laboratory were acceptable.

The data acquisition system was installed and used to collect data from the Validyne DP cells during some of the experiments. A low pass filter was purchased which will be used in conjunction with the Sensotron DP cell. The ratemeters were connected to the data acquisition system and calibrated using a constant frequency generator.

IV. Detailed Description of Technical Progress

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

A.1. Physical Property Measurements

Surface Tension Measurements

Surface tension measurements were made using samples from Runs F415-SIS10-2S, F455-SIS30-2S, and F540-FEL20-2S. Surface tensions were measured at 200 and 260 °C. The surface tension was measured twice at each condition. The difference in surface tension values between the two measurements was less than 0.001 N/m for all cases.

The surface tension was essentially the same for all samples analyzed. At 200 °C the surface tension was 0.020 N/m and at 260 °C it was 0.017 N/m. These values are slightly lower (5 to 7 %, relative) than those obtained previously (Technical Progress Report 4, April - June 1988).

Viscosity Measurements

Viscosity measurements were made at 230 °C using a Brookfield viscometer at 60 rpm equipped with a thermocel at two different times. Viscosities were obtained following our first two series of experiments using fresh FT-300 wax, used FT-300 wax (Run 2-3 from our previous contract DE-AC22-84PC70027), and used FT-300 from Runs F160-FES10-2S and F260-FES30-2S. The second set of viscosity measurements were made using fresh FT-300 wax and used FT-300 wax from runs F415-SIS10-2S and F455-SIS30-2S. The viscometer was calibrated before and after each set of samples were analyzed using solutions of known viscosities. The viscosities of the standard solutions ranged from 0.97 to 8.8 cp. The maximum percent difference (relative) between measured viscosities of the standard solutions was less than 4 percent.

Table 1 contains our results for the various waxes we analyzed as well as results from our previous contract at 230 °C for fresh FT-300 wax. The viscosities measured from our second set of samples were approximately 20 to 30 % (relative) higher than those

from our first set of samples. The viscosity of fresh FT-300 wax differed for the two samples analyzed from 2.3 to 2.8 cp. During our previous contract the viscosity of fresh FT-300 wax was measured at 230 °C. A value of 3.6 cp was obtained for the viscosity of FT-300 wax. The samples analyzed from experiments with 10 and 30 wt % iron oxide and silica did not contain 10 and 30 wt % of solids. The samples were prepared by melting the solidified slurry on a hot plate (approximately 30 ml) and decanting the liquid (8 ml) from the top. The exact weight percent of solids in the 8 ml samples was not known. Due to poor reproducibility (i.e. different values for the viscosity of pure FT-300 wax) and the fact that the exact weight percent of solids in each sample was not known, a direct comparison between viscosities measured for samples containing iron oxide and samples containing silica cannot be made. In general, the viscosity of samples containing some solids was greater than the viscosity of pure FT-300 wax.

A.2. Overview of Bubble Column Operations

During the past quarter, we sieved 35 pounds of large iron oxide particles (35 – 44 μm) for use in the small column using a Sweco vibrating sieve. We were able to obtain approximately 4 – 5 pounds of particles in the desired range over an eight hour period. Due to the amount of time required to obtain these particles, we decided to have Vortec Product Co. of Long Beach, California grind/classify iron oxide and silica particles for use in the large columns. The particles were sized in the range 20 – 44 μm as opposed to 35 – 44 μm , in order to reduce the cost. We have received all of the solids from Vortec Product Co. (approximately 300 pounds of each).

Particle size analysis of the iron oxide and silica particles classified by Vortec was made using a Baush and Lomb Image Analyzer at Kansas State University. Results from the analysis for the large iron oxide particles showed approximately 70 % of the particles to be in the range 20 – 44 μm with 16 % less than 20 μm and 14 % greater than 44

μm . All particles were less than $70 \mu\text{m}$. Thirty six percent of the silica particles were in the range $20 - 44 \mu\text{m}$ with 9 % above $44 \mu\text{m}$ and 55 % below $20 \mu\text{m}$. Approximately 70 % of the silica particles were in the range $10 - 44 \mu\text{m}$.

The stirrer for the large storage tank was tested using a 5 wt % slurry of large iron oxide in water. Approximately 60 % of the iron oxide was greater than $44 \mu\text{m}$. The stirrer was comprised of a 1/8 hp motor and a 3 inch diameter propeller (5 degree blade pitch). The propeller was located approximately 4 inches from the bottom of the tank. Samples were withdrawn from the tank approximately one hour after the slurry had been added to the tank, and the stirrer had been turned on. The weight percent of solids at both the top and bottom of the storage tank was less than one percent. The stirrer remained on overnight, and samples were withdrawn the next day. There was no improvement in mixing.

Thus, a new stirrer was designed and built. The new stirrer is comprised of a 3/4 hp motor and an eight inch diameter propeller with an 8 degree blade pitch located approximately 2 inches from the bottom of the tank. The new stirrer performed satisfactorily. Samples withdrawn from the top and bottom of the storage tank approximately 12 hours after turning on the stirrer were 5.6 and 8.7 percent, respectively. The increase in weight percent over the 5 percent initial loading is due to the evaporation of water from the tank (i.e. the water/iron oxide slurry remained in the tank approximately 2 weeks before the new system was tested), as well as previous sample withdrawals (i.e. samples withdrawn during testing of the original stirrer). Further tests will be conducted using the actual particles we plan to use in order to determine if another propeller will be needed to obtain a uniform suspension.

The new idle gear for the pump was received and the pump was rebuilt. A total of seven experiments were performed, five with $0 - 5 \mu\text{m}$ silica particles and 2 with $35 - 44 \mu\text{m}$ iron oxide particles before the pump became inoperable.

During the first five experiments with silica particles, the stirrer became detached from the stirring rod after the second experiment. By the last experiment (30 wt% silica), the solids concentration in the storage tank dropped to 26.5 wt % and the average solids concentration in the column was 27.5 wt %. The slurry was drained from the storage tank following the last experiment. Once the system cooled down, the storage tank was opened and approximately 2000 g of slurry remained inside the storage tank "caked" on the walls. The slurry was removed and analyzed. It was found to contain approximately 58 wt % solids.

We were able to account for 91.5 % of the solids charged into the system. We believed the remaining solids were probably in the expansion unit. The system was cleaned using hot toluene in order to removed any wax/solids which might be in the system before the next set of experiments were initiated.

An additional propeller was added to the stirring rod and both propellers were silver soldered in place to prevent them from becoming detached in subsequent experiments. Next, two experiments were attempted using large silica (20 – 44 μm) in FT-300 wax; however, we were never able to maintain flow with the pump. We speculated that the problem might be caused by cavitation in the pump. At the time, we believed that the additional stirrer might be too close to the top of the slurry in the storage tank, producing foam. Thus, we removed the additional propeller, but this did not help. The pump was removed and cleaned. Some wax was found between the graphite bushing and the seal seat indicating that trace amounts of cooling water were passing from the cooling water chamber to the wax slurry and vaporizing inside the pump causing cavitation. The pump was cleaned and reinstalled.

Two experiments were conducted with large iron oxide particles (35 – 44 μm , 10 wt %) before the pump became inoperable. The first experiment was conducted using a superficial slurry velocity of 0.005 m/s. During this experiment, there was considerable

settling of solids in the storage tank and the system. The maximum solids concentration observed in the column was only 4 wt % which occurred in the uppermost section of the column at a superficial gas velocity of 0.02 m/s. Throughout the course of the experiment, the solids concentration in the storage tank remained at approximately 2 wt %. In order to achieve better mixing in the storage tank, and increase the concentration of solids in the column, we added an additional propeller. Figure 1 is a schematic of the storage tank with the locations of the two propellers. The propellers are located approximately 2 inches and 6 inches from the bottom of the storage tank. The lower propeller has a blade pitch of approximately 8 degrees and the upper propeller has a blade pitch of approximately 5 degrees. This new design seemed to help. During the next experiment with a superficial slurry velocity of 0.02 m/s, the solids concentration in the storage tank was approximately 5 wt % during the experiment and the maximum solids concentration in the column was 6.5 wt %. After the slurry was transported from the column to the storage tank at the end of the experiment, the solids concentration in the storage tank was 9 wt %. This seemed to indicate that solids might be settling in the bottom of the column or in the bottom of the storage tank.

A third experiment was attempted with 20 wt % iron oxide particles but, we were unable to maintain a slurry velocity of 0.005 m/s. Following this attempt, we drained the slurry from the system and inspected all major components of the system. Upon examination of the various components of the system, a large amount of slurry was recovered in the expansion unit. Samples taken from the expansion unit contained approximately 85 wt % solids. The solids recovered from the expansion unit were a mixture of silica and iron oxide. A total of approximately 3200 g of solids was recovered in the expansion unit. It appears that throughout the experiment, solids continue to settle in the expansion unit, and this is one of the causes of the decrease in solids concentration during the experiment. Also, some solids were recovered in the calibration

unit (approximately 250 g). Since there is a significant amount of settling in these units, we are in the process of modifying both the expansion unit and the calibration unit to minimize solids settling.

Figure 2 is a schematic of the modified expansion unit which is being constructed. The modifications consist of constructing a channel between the overflow from the column and the recirculation line at the bottom of the expansion unit. This will minimize the surface area available for solids settling as well as increase the flowrate of slurry from the column overflow to the recirculation line which will help prevent solids from settling in the expansion unit.

The modification to the calibration unit consists of increasing the slope of the conical section at the bottom of the unit. Previously, the conical section at the bottom of the calibration unit had a slope of 10 degrees. The new conical section will have a slope of 45 degrees. The expansion unit and calibration unit for the large column are also being modified.

Some tests were performed to determine the extent of solids settling in the small storage tank. The iron oxide slurry removed from the storage tank following the last experiment (F535-FEL20-2S) was used for the test. With the stirrer running at 70 % of full power, the solids concentration in the storage tank was 15 wt % at the top and 15.7 wt % at the bottom. After 24 hours, the solids concentration at the top of the tank dropped to 13.6 wt % and at the bottom of the tank it dropped to 14.1 wt %. Upon increasing the stirrer speed to 80 % the solids concentration in the tank rose to 14.9 wt % at the top and 13.4 wt % at the bottom within 4 hours. Next, the stirrer speed was increased to 90 % and the solids concentration rose to 15.4 wt % at the top and 14.3 wt % at the bottom after 3 hours. Solids were added to the tank to increase the concentration to 20 wt %. Approximately five hours after adding the solids, the solids concentration in the tank was 21.7 wt % at the top and 21.1 wt % at the bottom.

Next, a batch experiment was conducted using the 20 wt % (nominal) slurry of iron oxide. The average solids concentration in the column ranged from 21 wt % at a gas velocity of 0.02 m/s to 24 wt % at a gas velocity of 0.12 m/s.

Following the experiment with the iron oxide slurry, a batch experiment was attempted with 20 – 44 μm silica particles (20 wt %). Several of the DP cells became plugged during the experiment and the run had to be aborted. All DP cells were removed from the column, cleaned, and reinstalled. Once the DP cells were recalibrated, we attempted to perform the same experiment. Two of the DP cells became plugged and upon using nitrogen (80 psig) to unplug the DP cells, we forced a significant amount of the slurry into the expansion unit. The DP cells became plugged again, and upon clearing them, more slurry was forced into the expansion unit. We believe the problem with plugging is due to worn fittings which may be leaking. We have ordered new fittings for the DP cells and will rebuild the system when they arrive.

We have contacted Gelber Pumps requesting some form of restitution for the gear pump since it is not capable of performing the job it was supposed to. Gelber Pumps is looking into an alternate material for the idle gear (i.e. not graphite) which may be more suitable for our system. They have contacted us and will send us replacement parts for the worn out parts (i.e. wear plates, bearings, etc) as well as a new drive gear and idle gear at no additional charge.

A.3. Experimental Results

Nine successful experiments were conducted using FT-300 wax at 265°C. In addition to these three more runs were initiated, but had to be aborted because of operational problems. Results from the nine successful runs are discussed here. Three of the experiments were conducted in the batch mode of operation, and the remaining experiments were conducted in the continuous mode of operation using the rebuilt slurry pump for

recycling the slurry.

Five of the nine experiments were conducted using 0 - 5 μm silica particles. One experiment was conducted with a 10 wt% slurry using a superficial slurry velocity of 0.005 m/s. Three experiments were conducted with a 20 wt % slurry using superficial slurry velocities of 0.0, 0.005, and 0.02 m/s. And the last experiment was conducted at a superficial slurry velocity of 0.005 m/s with a 30 wt % slurry.

Two experiments were made with a 10 wt % slurry of 35 - 44 μm iron oxide particles in FT-300 wax using superficial slurry velocities of 0.005 and 0.02 m/s. A third experiment was attempted using a 20 wt% slurry at a velocity of 0.005 m/s but had to be aborted due to operational problems with the pump.

Two batch experiments were conducted using 20 wt % slurries of 35 - 44 μm iron oxide particles and 20 - 44 μm silica particles. The experiment with silica particles was conducted in order of decreasing gas velocity (i.e. from $u_g = 0.12$ to 0.02 m/s). There were some operational problems with the DP cells during the experiment with the silica slurry.

The axial hold-up profiles for the three phases were determined for all experiments. The gas and liquid phases account for most of the dispersion volume and only a small volume is occupied by the solids phase. Therefore, the axial gas hold-up profiles are complementary to the axial liquid hold-up profiles. The axial solids hold-up profiles are mainly determined by the axial weight fraction solids profiles. Therefore, only the axial gas hold-up profiles and the axial weight fraction solids distributions are presented for each of the nine runs. These are included in APPENDIX A. Results from the different runs are categorized in terms of the various effects and are discussed here. Wax and solids inventories were also made for all runs and the discrepancies in these balances and the possible causes are discussed. Also included is a discussion of the effect of solids type and size based on the results obtained this quarter and those presented in

the previous Technical Progress Report (April – June, 1988).

Effect of liquid velocity on average gas hold – up :

Figure 3 shows average gas hold-ups from experiments conducted with 20 wt % 0 – 5 μm silica particles in FT-300 wax. Foam was produced for the batch case with gas hold-up values ranging from 33 to 41 % over superficial gas velocities of 0.04 to 0.12 m/s. For experiments conducted in the continuous mode of operation, the gas hold-up decreases significantly for gas velocities greater than 0.02 m/s. There is essentially no effect of slurry velocity on the average gas hold-up for experiments conducted in the continuous mode of operation. At $u_g = 0.12$ m/s hold-ups from all three runs begin to approach the same value. The foam produced with FT-300 wax, while operating in the batch mode, appears to have dissipated at this gas flow rate, and hold-ups have converged to values similar to those for the continuous mode of operation. Our earlier experiments in the glass column (Bukur et al., 1987) have shown that slug flow dominates for gas velocities in this range, and hold-ups are determined by the size of these slugs.

Figure 4 shows average gas hold-up values from the two runs made with a slurry of 10 wt % 35 – 44 μm iron oxide in FT-300 wax. The trends are similar to those for the experiments conducted in the continuous mode of operation with 0 – 5 μm silica particles (i.e. essentially no effect of liquid velocity for experiments conducted in the continuous mode of operation).

The above results indicate that liquid circulation tends to lower gas hold-ups, however, further increases in liquid flow rates do not have a significant effect on the average gas hold-up values. The significant difference in hold-ups between the batch mode of operation and continuous mode of operation is due to the changes in the foaming characteristics of the medium. For the batch case, the foam accumulates at the top and increases the gas hold-up. However, as slug flow sets in, the foam tends to disperse and

the hold-up profiles tend to flatten out. In the continuous mode of operation, any foam that tends to accumulate at the top is carried back down by the circulating slurry and dispersed. With an increase in the liquid circulation, this rate of dispersion increases, decreasing the average gas hold-up.

Effect of solids concentration on average gas hold – up :

Figure 5 shows the effect of solids concentration on gas hold-up at a superficial slurry velocity of 0.005 m/s. In Figure 5 the hold-up values for the various silica (0 – 5 μm) slurries decrease with an increase in slurry concentration. The highest hold-up values are observed with pure FT-300 wax and the lowest are obtained with a slurry containing 30 wt % solids. The decrease in hold-up with increasing solids concentration is probably due to the increase in slurry viscosity associated with the additional solids. A similar trend was observed in our earlier experiments in the glass column (Bukur et al., 1987) where an increase in the viscosity of the wax (i.e. a decrease in temperature) caused the gas hold-up to decrease.

Axial gas hold – up profiles :

The axial gas hold-up profiles at gas velocities of 0.02, 0.04 and 0.12 (or 0.09) m/s are shown in Figures A-1 through A-9. The three profiles for each run clearly illustrate the effect of superficial gas velocity on the axial gas hold-up. For all runs, the axial gas hold-ups increased with increases in gas velocity, which is expected, since the overall average gas hold-up increases with superficial gas velocity. The results also show that gas hold-up increases with an increase in height above the distributor for all cases. The increases are more significant at higher superficial gas velocities than the increases at lower gas velocities. For experiments conducted in the batch mode of operation, the foam at the top of the dispersion is responsible for the significantly higher hold-ups in the upper section, compared to the values obtained in the continuous mode of operation. In general, the axial gas hold-up profiles are affected by the superficial slurry velocity.

At $u_g=0$ (batch case), the profiles are non-uniform (low hold-ups at the bottom of the column and higher values at the top). However, as u_g is increased to 0.005 m/s, the profiles are less steep and become fairly uniform at $u_g=0.02$ m/s.

Figure A-9 shows results from the batch experiment conducted with 20 – 44 μm silica particles. This experiment was conducted in order of decreasing gas velocities. For this experiment, a substantial amount of foam was not produced. This is reflected in the axial gas hold-up profiles which do not show a substantial increase in the axial gas hold-up for gas velocities of 0.04 and 0.12 m/s. At gas velocities of 0.04 and 0.12 m/s the axial gas hold-up exhibits a slight increase with increasing height above the distributor.

Axial solids distribution profiles :

The axial solids distribution profiles for gas velocities of 0.02, 0.04, and 0.12 (or 0.09) m/s are also shown in Figures A-1 to A-9. For experiments conducted in the continuous mode of operation, the axial solids profiles remained essentially uniform; whereas, for the experiments conducted in the batch mode of operation, there was a decrease in solids concentration with increase in height above the distributor for all solids employed. This concentration gradient was most pronounced for the experiments conducted with the large iron oxide and silica particles (See Figures A-8 and A-9). For the experiment conducted with large silica (Figure A-9), there is a substantial decrease in the solids concentration in the column between gas velocities of 0.12 and 0.04 m/s. This loss in solids concentration is due to the large amount of slurry that was "blown" into the expansion unit between gas velocities of 0.12 and 0.06 m/s in an effort to unplug the DP cells.

For experiments conducted in the continuous mode of operation with large iron oxide (i.e. 35 – 44 μm) particles (see Figures A-6 and A-7), the weight fraction of solids observed in the column was substantially less than the nominal weight percent of

solids charged. This was caused by solids settling in the expansion unit and bottom of the storage tank and column.

Wax and solid inventories :

Wax and solids balances were made for each run. The hold-up profiles in the column, together with the weight fraction and volume information for the slurry in the storage tank, were used to make the balances. The solids inventory showed that approximately 10% of the solids were unaccounted for after the very first run with small silica. Following the second experiment, there was still approximately 10 % of the solids unaccounted for. After this experiment, the stirrer became detached and solids settled in the storage tank. For the third and fourth experiments, approximately 19 and 23 % of the solids were unaccounted for, respectively. Solids were added to the system following the fourth experiment. The percent of solids unaccounted for increased to 30 % during the fifth experiment. After accounting for the solids found in the storage tank, and on the distributor following the last experiment with small silica particles, we were able to account for 91 % of the solids charged. The remaining solids had been deposited in the expansion unit and calibration chamber.

For the experiments conducted in the continuous mode of operation with large iron oxide particles, approximately 60 - 85 % of the solids were unaccounted for. Examination of the calibration chamber and expansion unit after these experiments revealed a substantial amount of solids had deposited in these units (primarily in the expansion unit). A summary of the solids recovered in various parts of the system following the experiments with small silica particles and large iron oxide particles is given in Table 2. The closure for the overall solids balance from the two sets of experiments was 105 %. We probably overestimated either the solids recovered in the expansion unit or the solids drained from the storage tank. Very large quantities of slurry were recovered in these units, and only 20 - 30 % of the slurry recovered was analyzed for solids content. It was

assumed that the solids content obtained from these measurements were representative of the entire slurry recovered from these units.

Wax inventories for the various runs were as expected. With time on stream, the quantity of wax unaccounted for increased. This is because the wax evaporates during the experiments and is carried out with the exit gas.

Effect of Solids Type and Size on Average Gas Hold – up :

Figures 6 – 9 show the effect of solids type and size on average gas hold-ups for all systems studied. In general, for experiments conducted in the continuous mode of operation, the average gas hold-up decreased with the addition of solids. Gas hold-ups were lowest for experiments conducted with silica particles. Since the density of silica is less than iron oxide (2.65 as opposed to 5.1 g/cc), for the same weight fraction slurry, the volume fraction of silica is greater than that of iron oxide. This increase in volume of the solids particles causes an increase in the viscosity of the slurry. This increase in viscosity may cause the hold-ups associated with the silica slurry to be less than the hold-ups associated with the iron oxide slurry. Experiments with large iron oxide particles produced slightly lower hold-ups than experiments conducted with small iron oxide particles. Foam was observed for all batch runs conducted in increasing order of gas velocity. Two interesting trends were observed for experiments conducted in the batch mode of operation (See Figure 10). First, for experiments conducted in increasing order of gas velocities, the gas hold-up for experiments conducted in the presence of solids was greater than the gas hold-ups observed with pure FT-300. Secondly, for the experiment with large silica particles conducted in a decreasing order of gas velocities, no foam was observed and the hold-ups were similar to those obtained for experiments conducted in the continuous mode of operation with small silica particles. A similar trend was observed in our early work (Bukur et al., 1987) for experiments conducted with FT-300 in the glass column in a decreasing order of gas velocities.

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

During the past quarter, we received the remaining material needed to construct the movable assembly mechanism which will be used to transport the nuclear density gauges both axially and radially along the column. The movable assembly mechanism has been constructed and tested.

The data acquisition system from Metrabyte was received and installed during the past quarter. A program for data acquisition from the DP cells (both Validyne and Sensotron) and the nuclear density gauges has been written. The data acquisition system was tested using both the high temperature (Sensotron) DP cell and the low temperature (Validyne) DP cells. Preliminary results indicated that a significant amount of noise is present when using the Sensotron DP cell. The noise is a result of the form of the output from the Sensotron readout. The output signal from the readout is in the form of a sine wave. Thus, when we record data from the Sensotron DP cell, we are recording arbitrary points on the sine wave. The frequency of the sine wave was determined to be 26 kHz using an oscilloscope. We have purchased a low pass filter which should eliminate this problem. It will wipe out all frequencies greater than 1 kHz. Some data were collected from the Validyne DP cells during the experiments in the small stainless steel column, but they have not been analyzed yet.

Our application for a license permitting the use of the high energy (Cesium – 137) and low energy (Americium – 241) sources has been reviewed by the Radiological Safety Office and approved. The Radiological Safety Office installed the sources in the apparatus and surveyed the laboratory. Radiation levels in the lab were within the acceptable limits.

The large stainless steel column and movable assembly mechanism have been enclosed in a plexiglass chamber to prevent people from coming too close to the radioactive

sources. Also, additional 3/8 inch steel plates have been mounted around the source holders to help minimize the amount of radiation in the laboratory. We have received radiation badges from the radiological safety office which will be used by personnel working in the laboratory.

The ratemeters were connected to the data acquisition system and calibrated using a constant frequency generator.

V. REFERENCES

Bukur, D.B., Daly J.G., Patel, S.A., Raphael, M.L. and Tatterson, G.B., "Hydrodynamics of Fischer-Tropsch Synthesis in Slurry Bubble Column Reactors," Final Report to the Department of Energy for Contract No. DE-AC22-84PC70027 (1987).

Table 1. Viscosity of waxes.

LIQUID	SET	RUN NUMBER	230 °C
FT-300 (Fresh)	1	N/A	2.3
FT-300 (Used)	1	F160-FES10-2S	2.8
FT-300 (Used)	1	F260-FES30-2S	3.1
FT-300 (Used)	1	2-3 Two-Phase	2.6
FT-300 (Fresh)	2	N/A	2.8
FT-300 (Used)	2	F415-SIS10-2S	3.9
FT-300 (Used)	2	F455-SIS30-2S	4.1
FT-300 (Fresh)	N/A	Previous Measurement (DE-AC22-84PC70027)	3.6

All values are in centipoise

N/A: Not Applicable

Table 2. Summary of solids recovered.

LOCATION	SILICA (g)	IRON OXIDE (g)
Added	7925	5397
Drained from Storage Tank	4825	3051
Recovered in Storage Tank	1175	0
Recovered from Distributor	65	211
Expansion Unit	-	3183
Calibration Unit	-	246
Removed During Experiment	908	71
Lines (Estimated)	250	-
Total Recovered	7223	6762

Total Charged: 13322

Total Recovered: 13985

Percent Accounted For: 105%

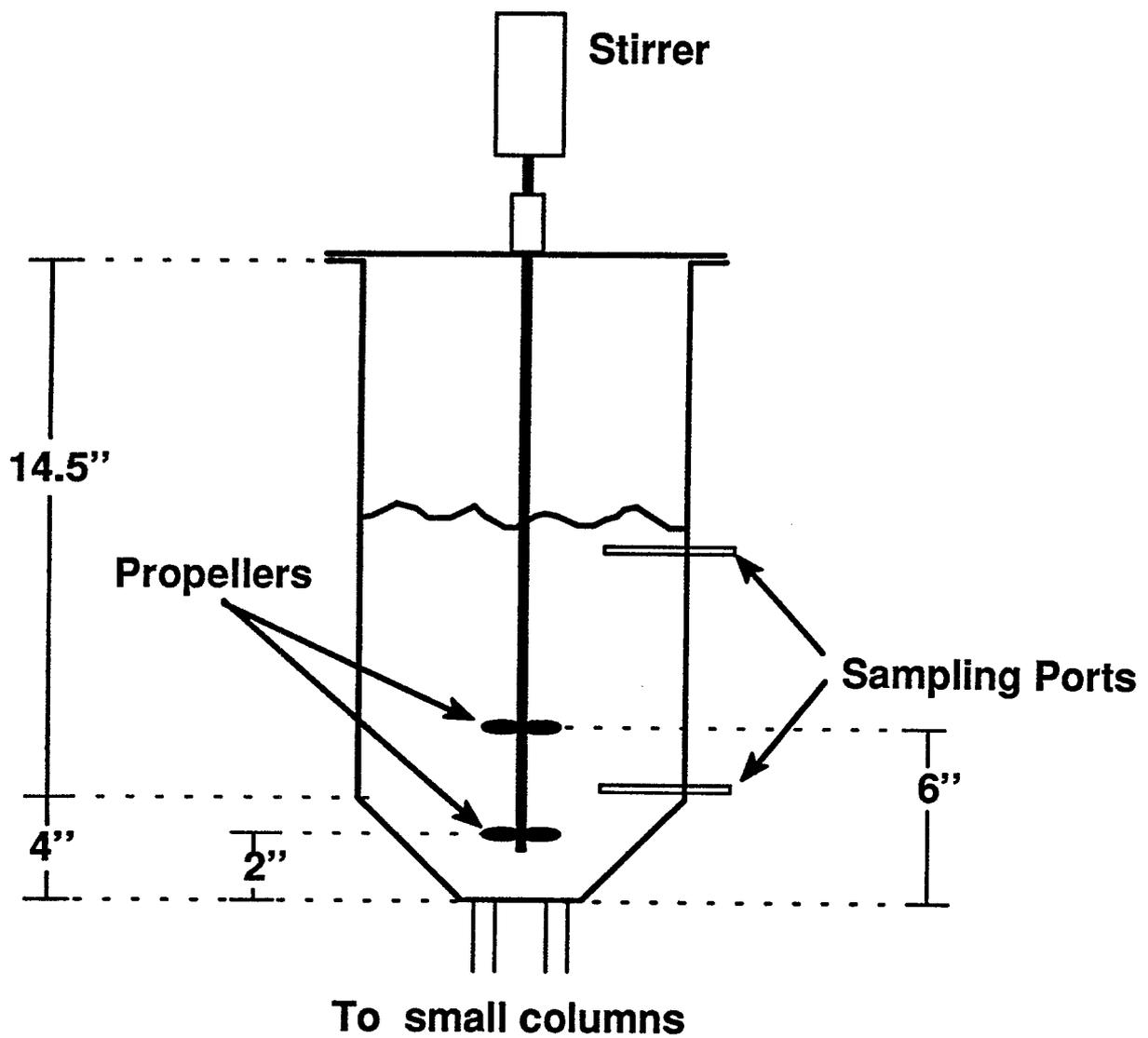


Figure 1. Schematic of stirrer for the small storage tank.

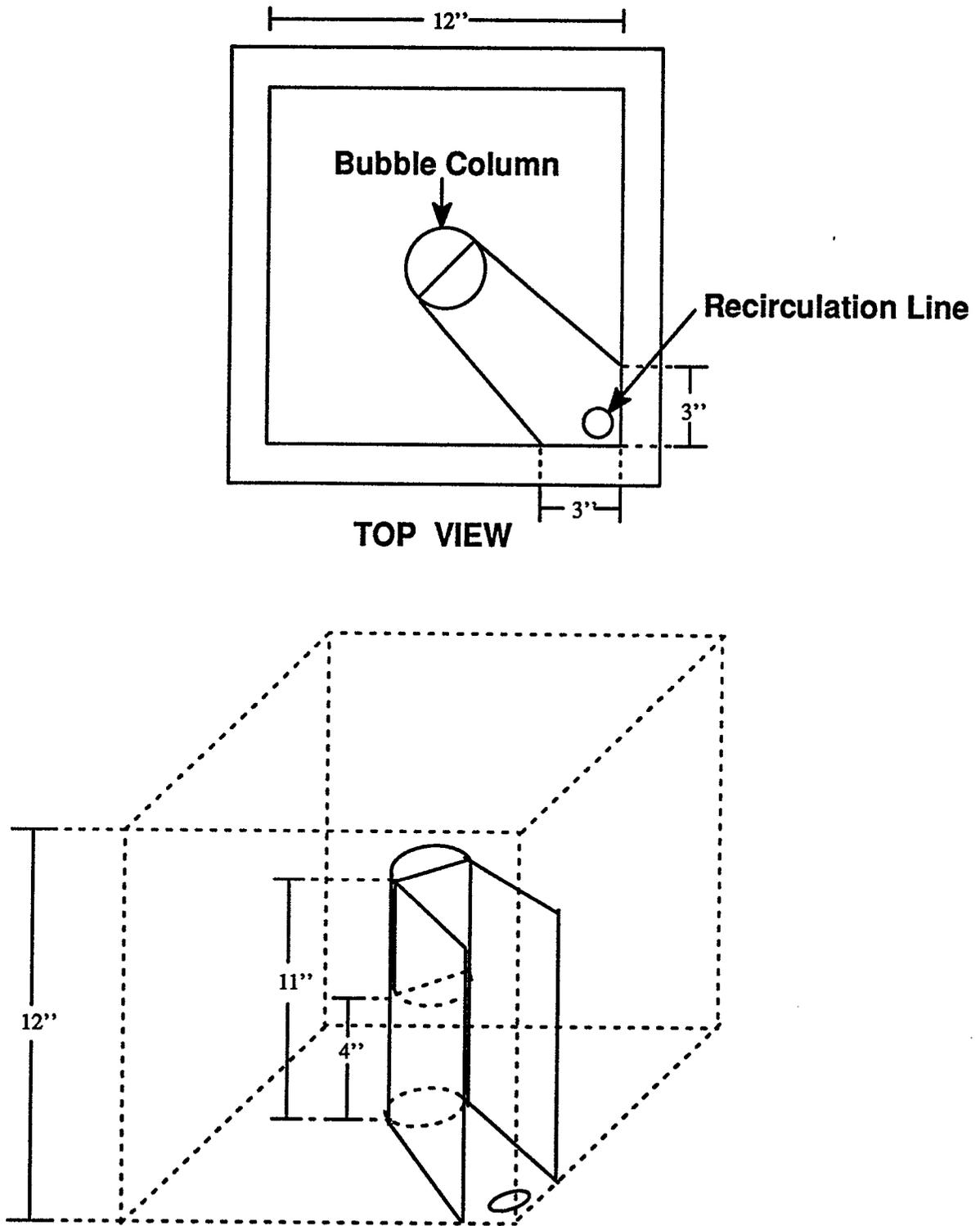


FIGURE 2. Schematic of modified expansion unit.

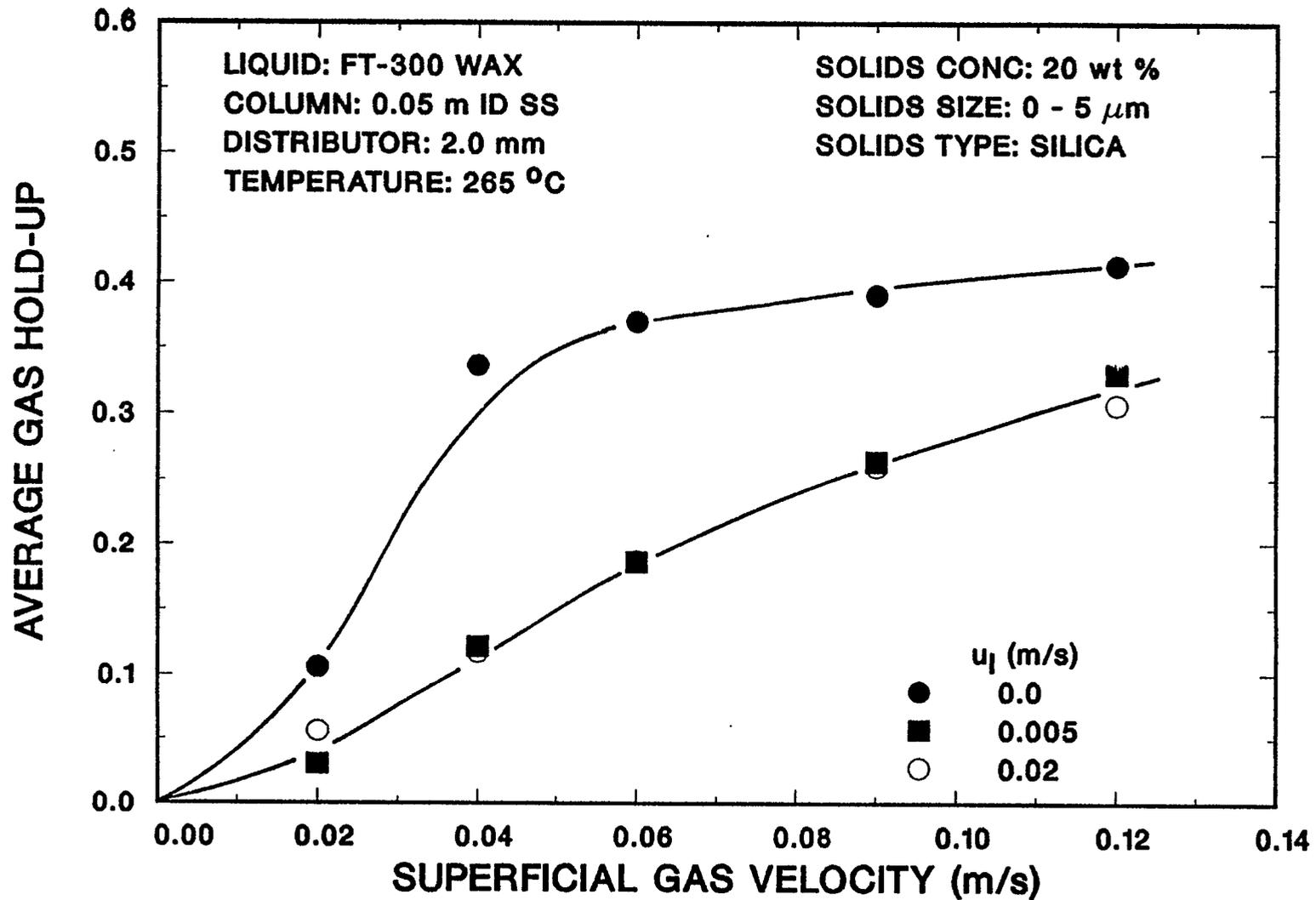


Figure 3. Effect of liquid velocity on average gas hold-up.

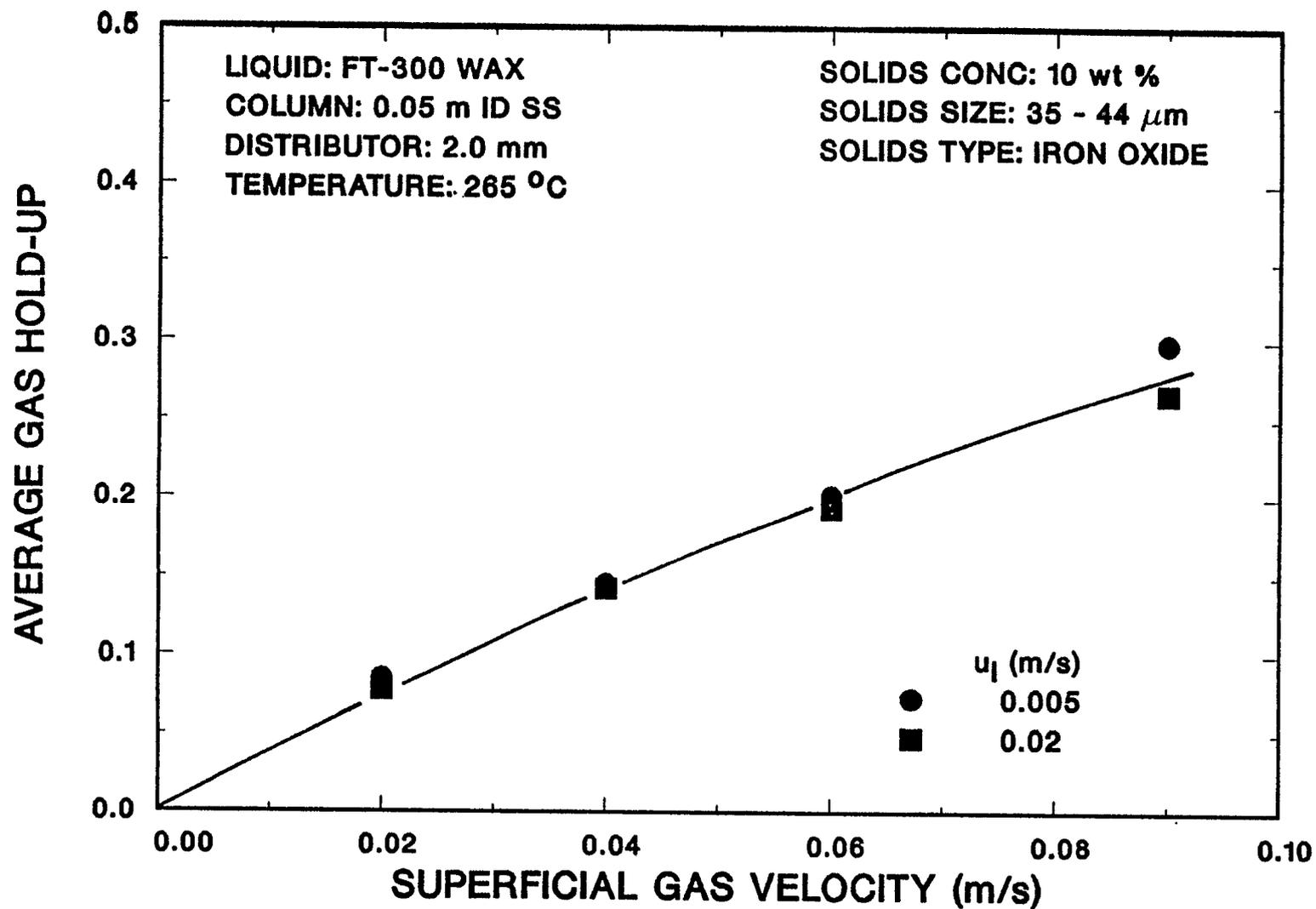


Figure 4. Effect of liquid velocity on average gas hold-up.

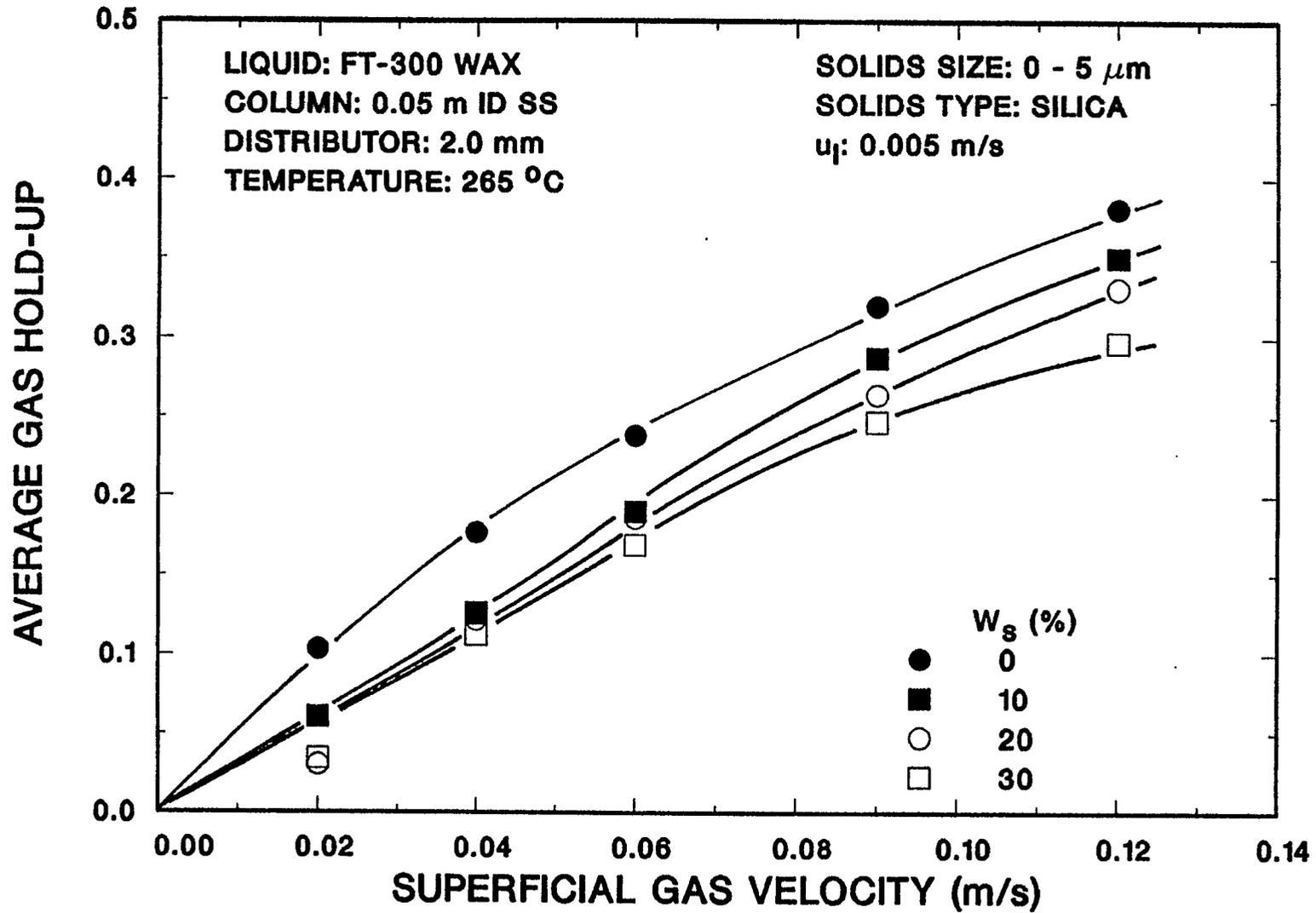


Figure 5. Effect of solids concentration on average gas hold-up.

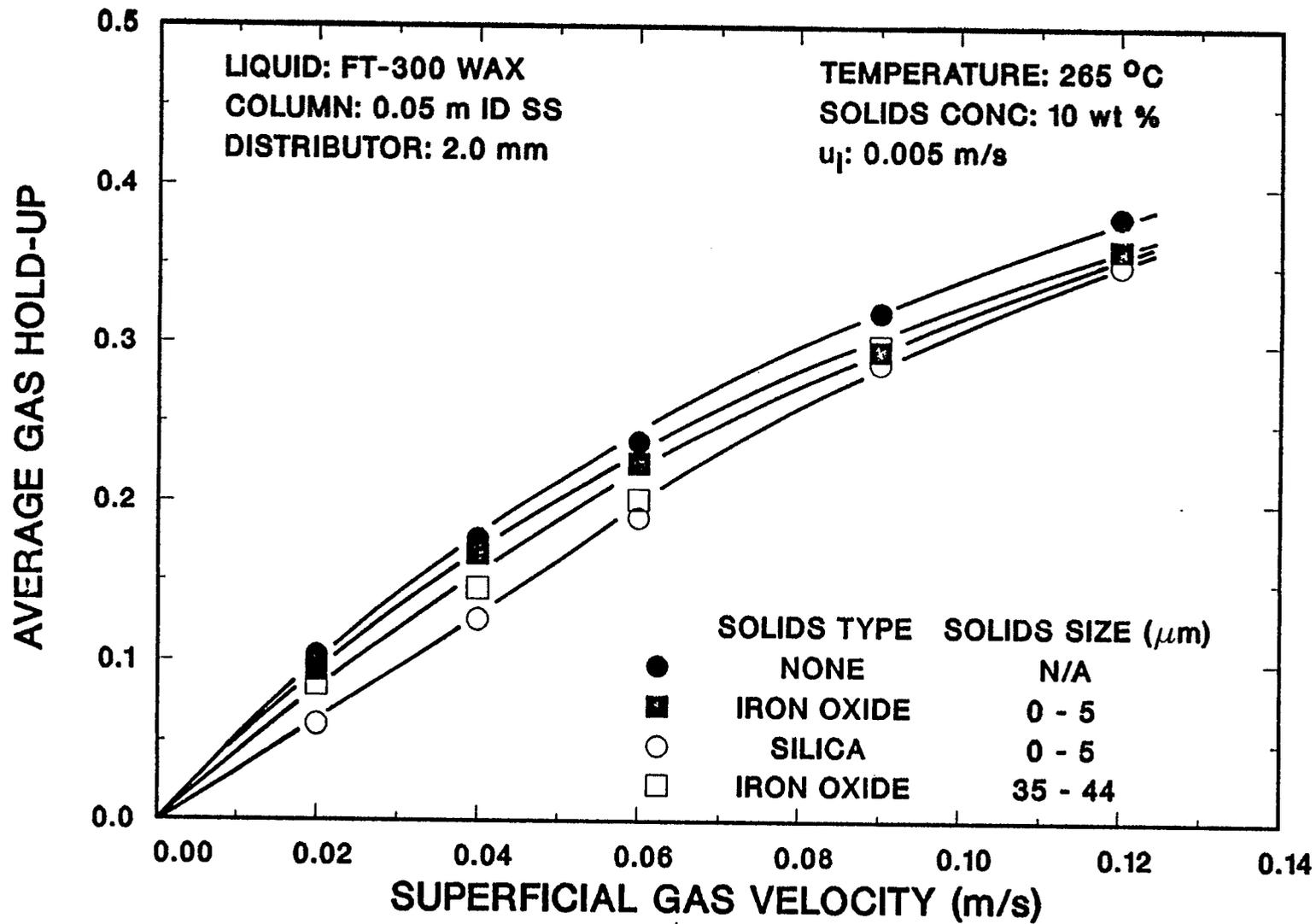


Figure 6. Effect of solids type and size on average gas hold-up.

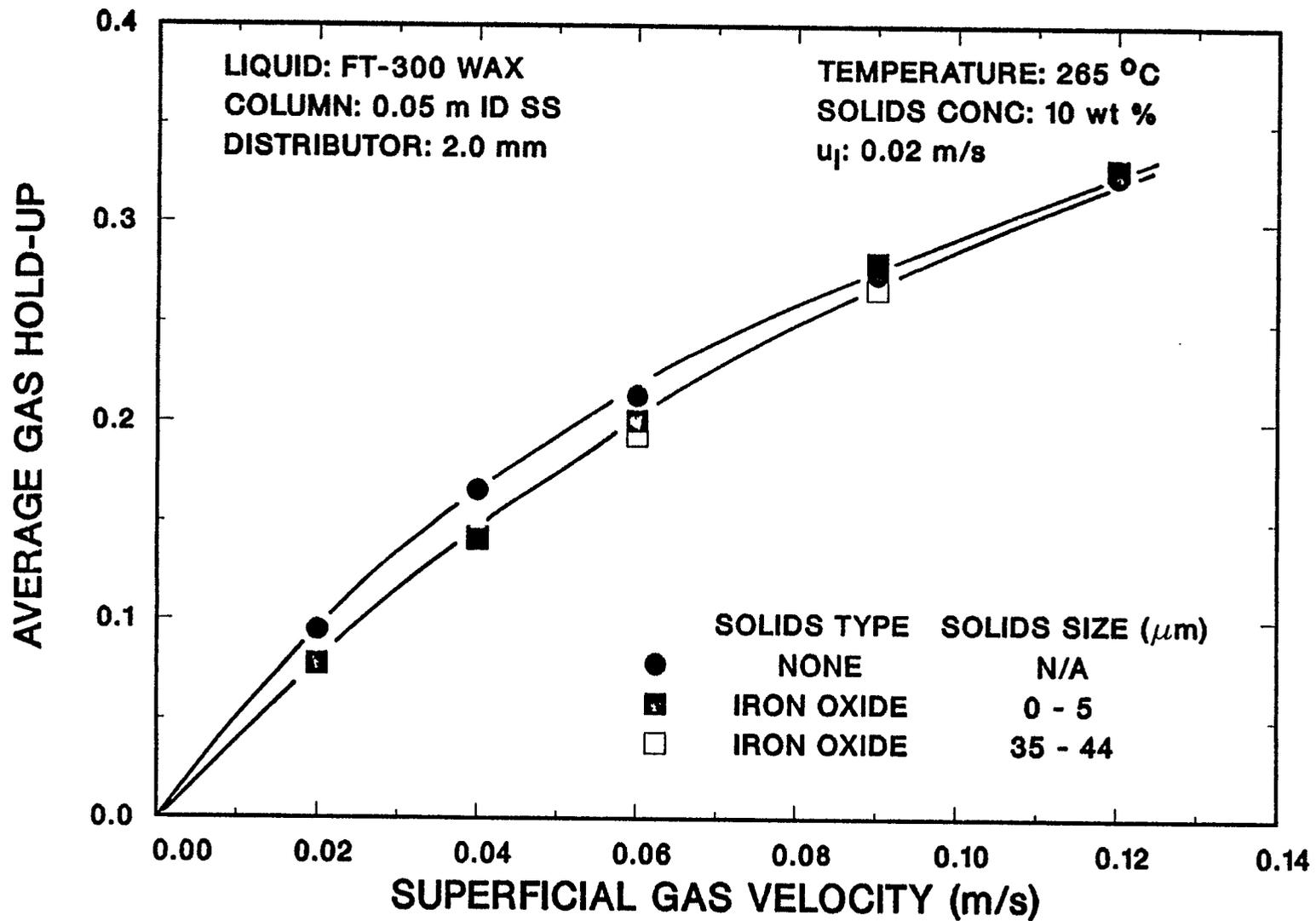


Figure 7. Effect of solids type and size on average gas hold-up.

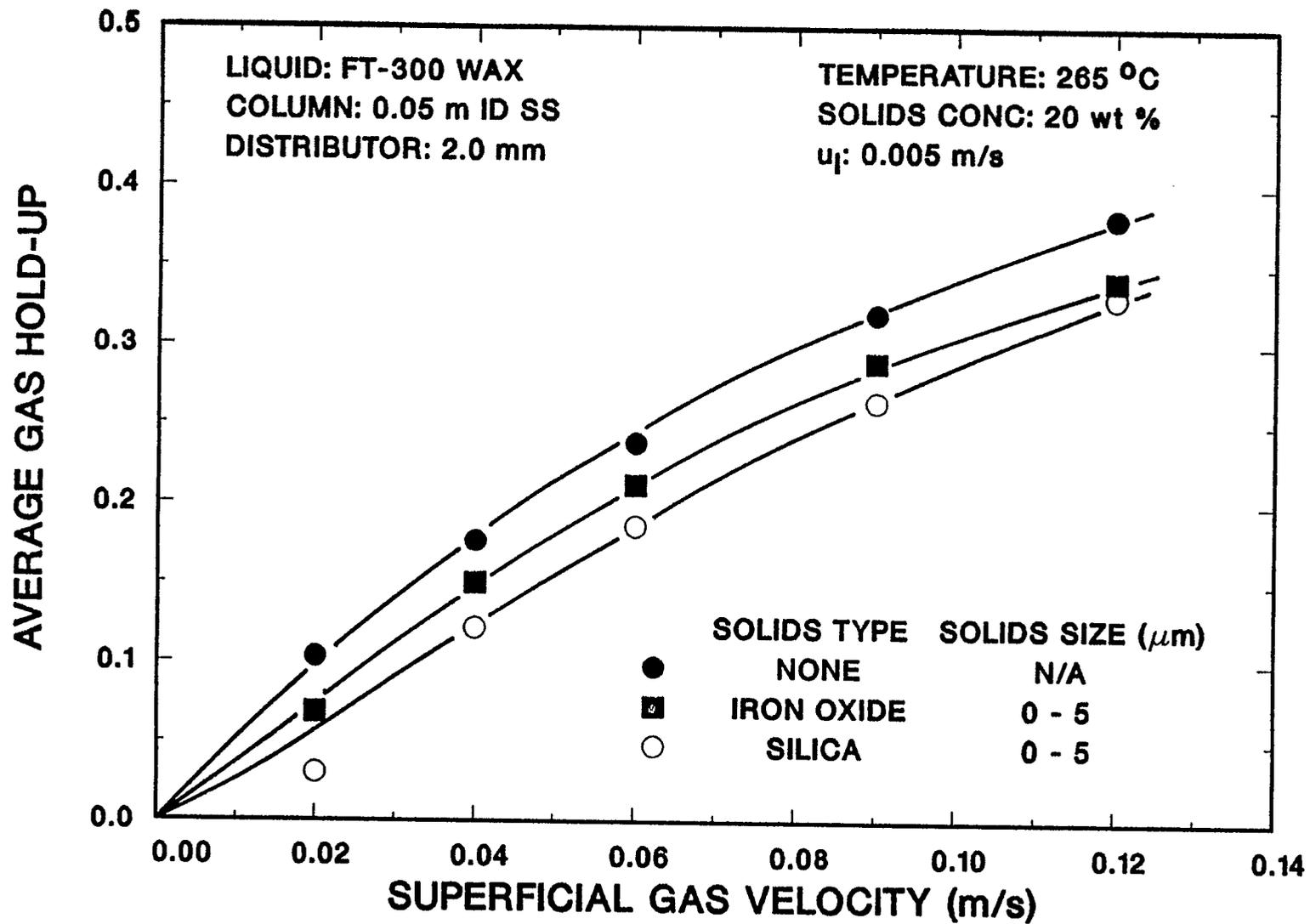


Figure 8. Effect of solids type and size on average gas hold-up.

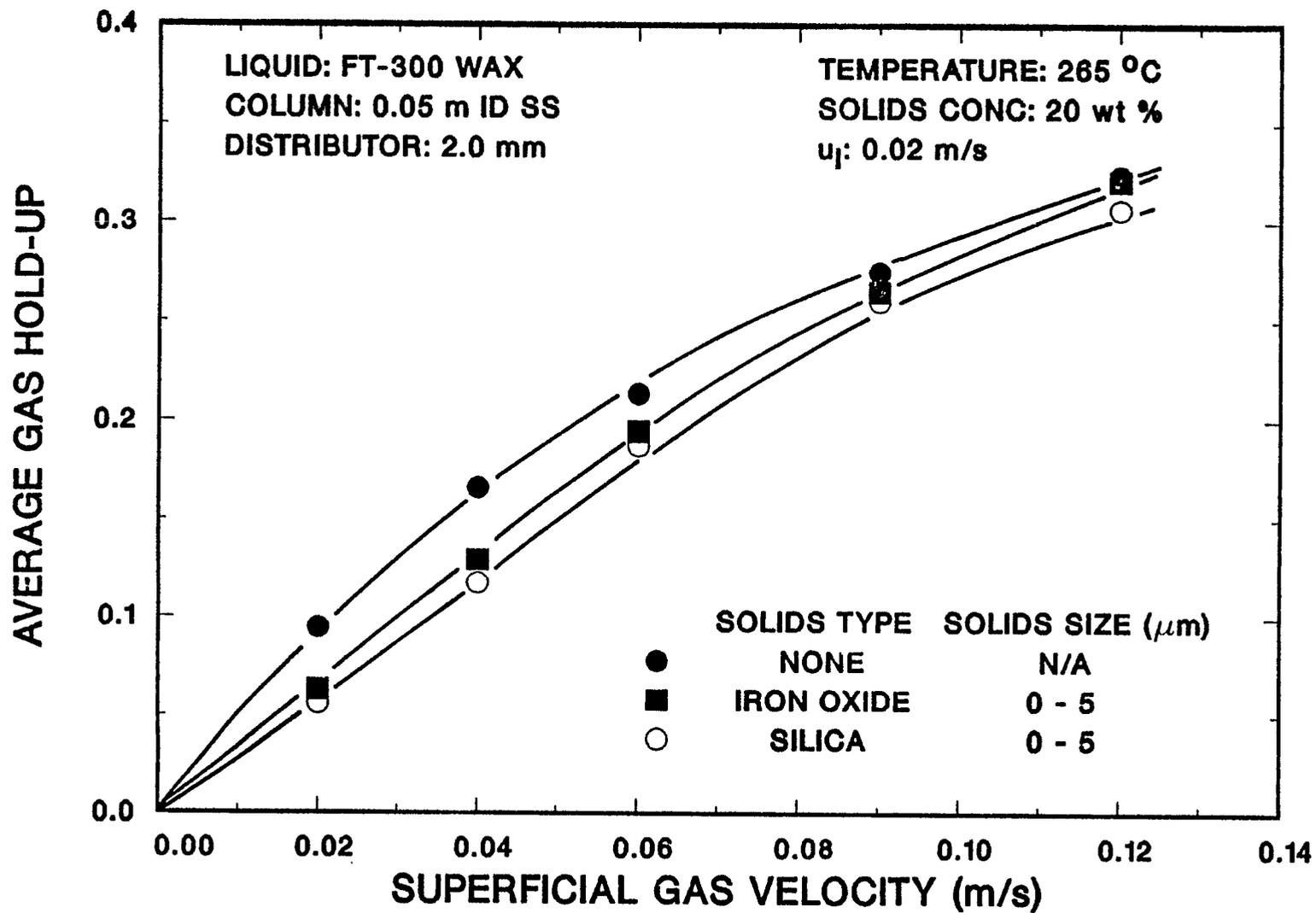


Figure 9. Effect of solids type and size on average gas hold-up.

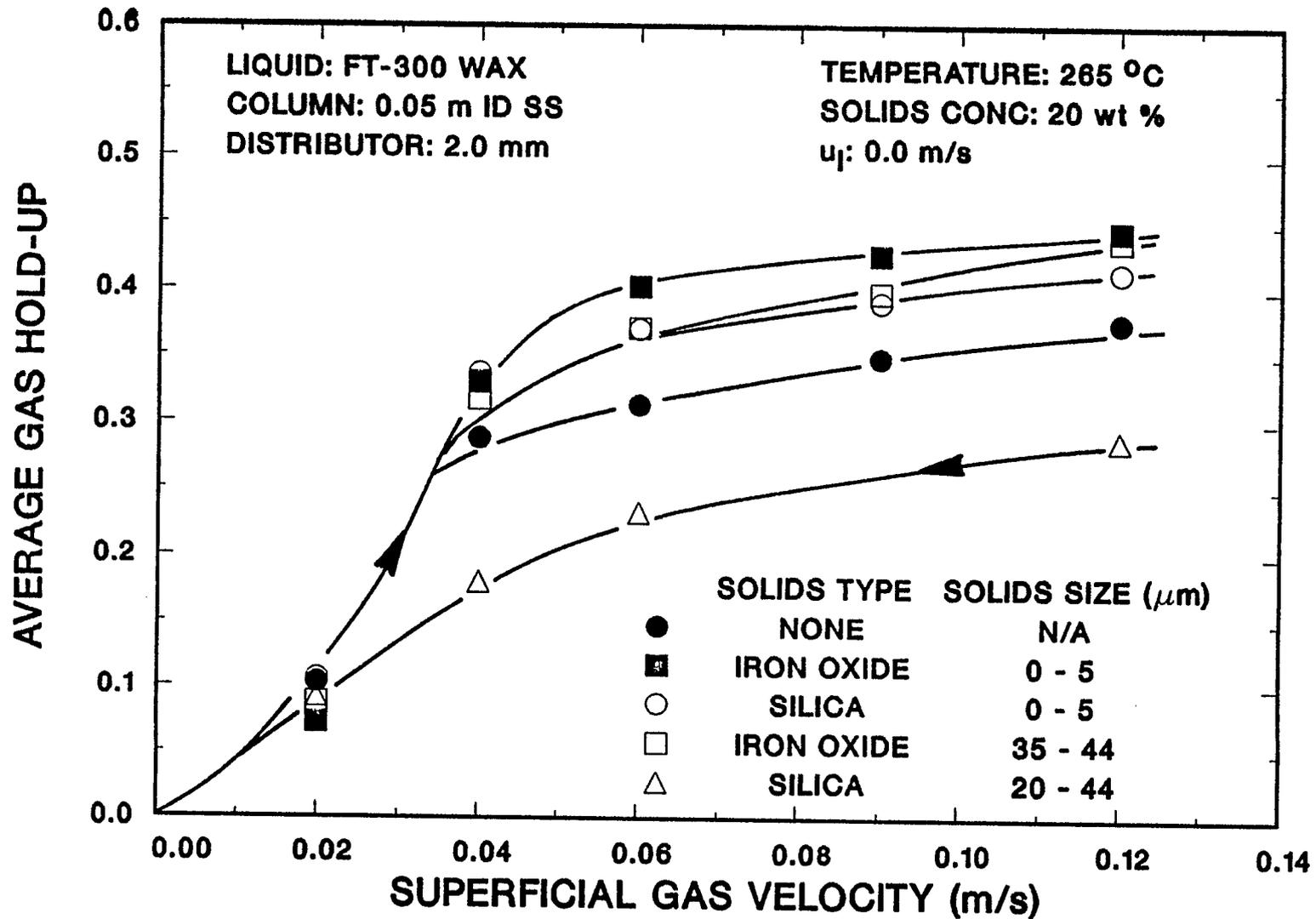


Figure 10. Effect of solids type and size on average gas hold-up.

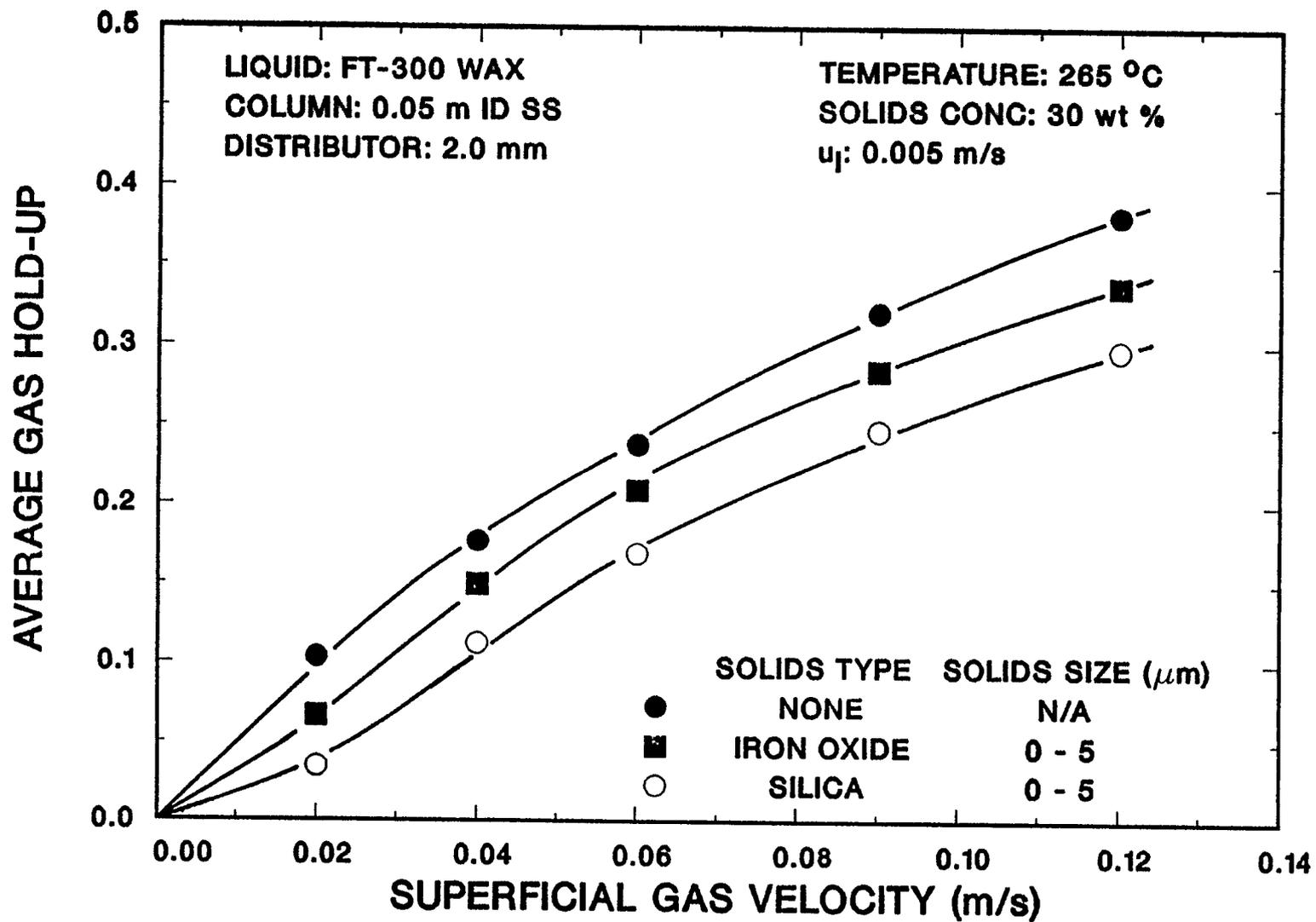


Figure 11. Effect of solids type and size on average gas hold-up.

APPENDIX A – AXIAL GAS HOLD-UP AND SOLIDS DISTRIBUTION PROFILES

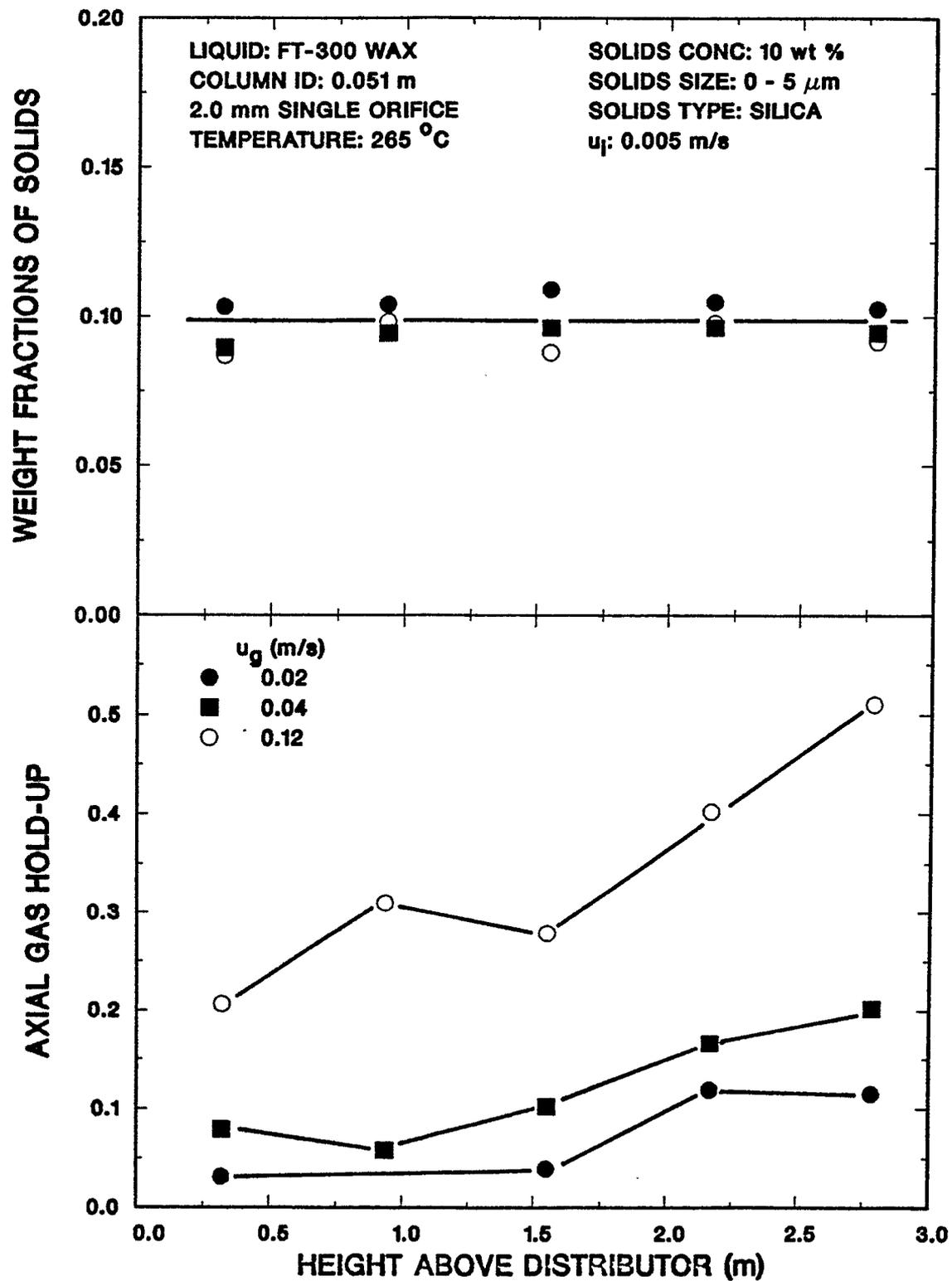


Figure A-1. Effect of superficial gas velocity on axial solids distribution (Top) and on axial gas hold-up (Bottom).

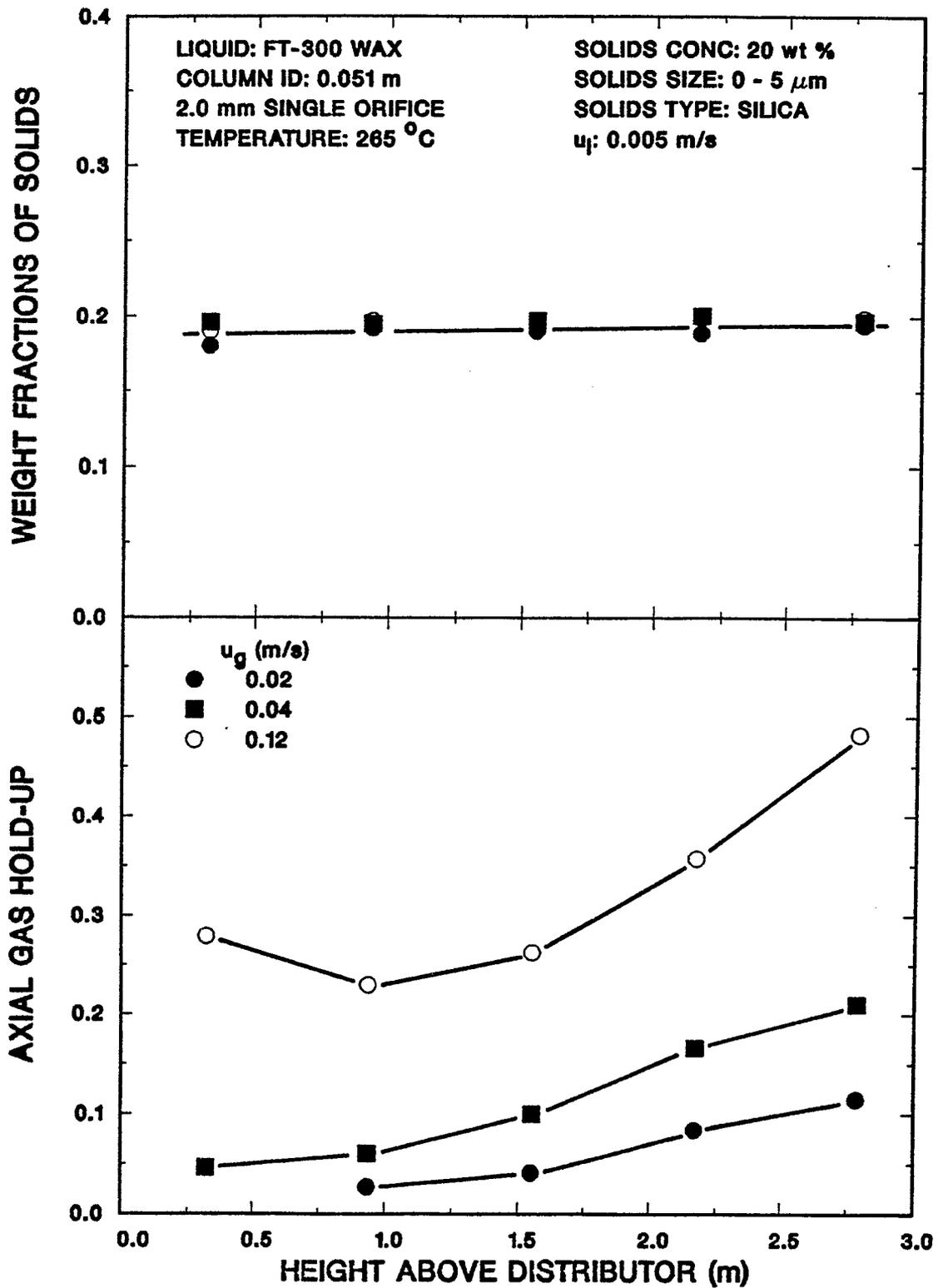


Figure A-2. Effect of superficial gas velocity on axial solids distribution (Top) and on axial gas hold-up (Bottom).

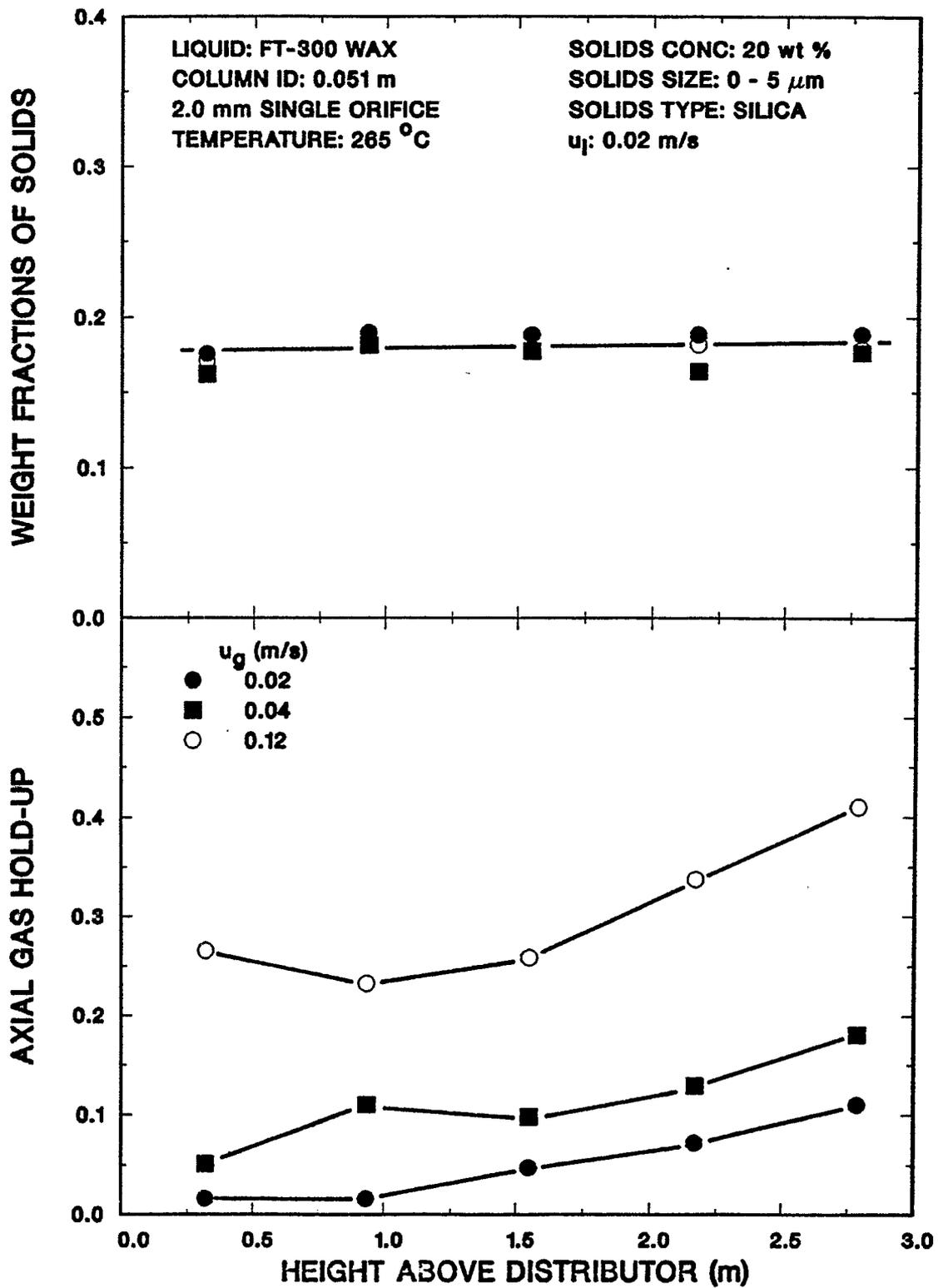


Figure A-3. Effect of superficial gas velocity on axial solids distribution (Top) and on axial gas hold-up (Bottom).

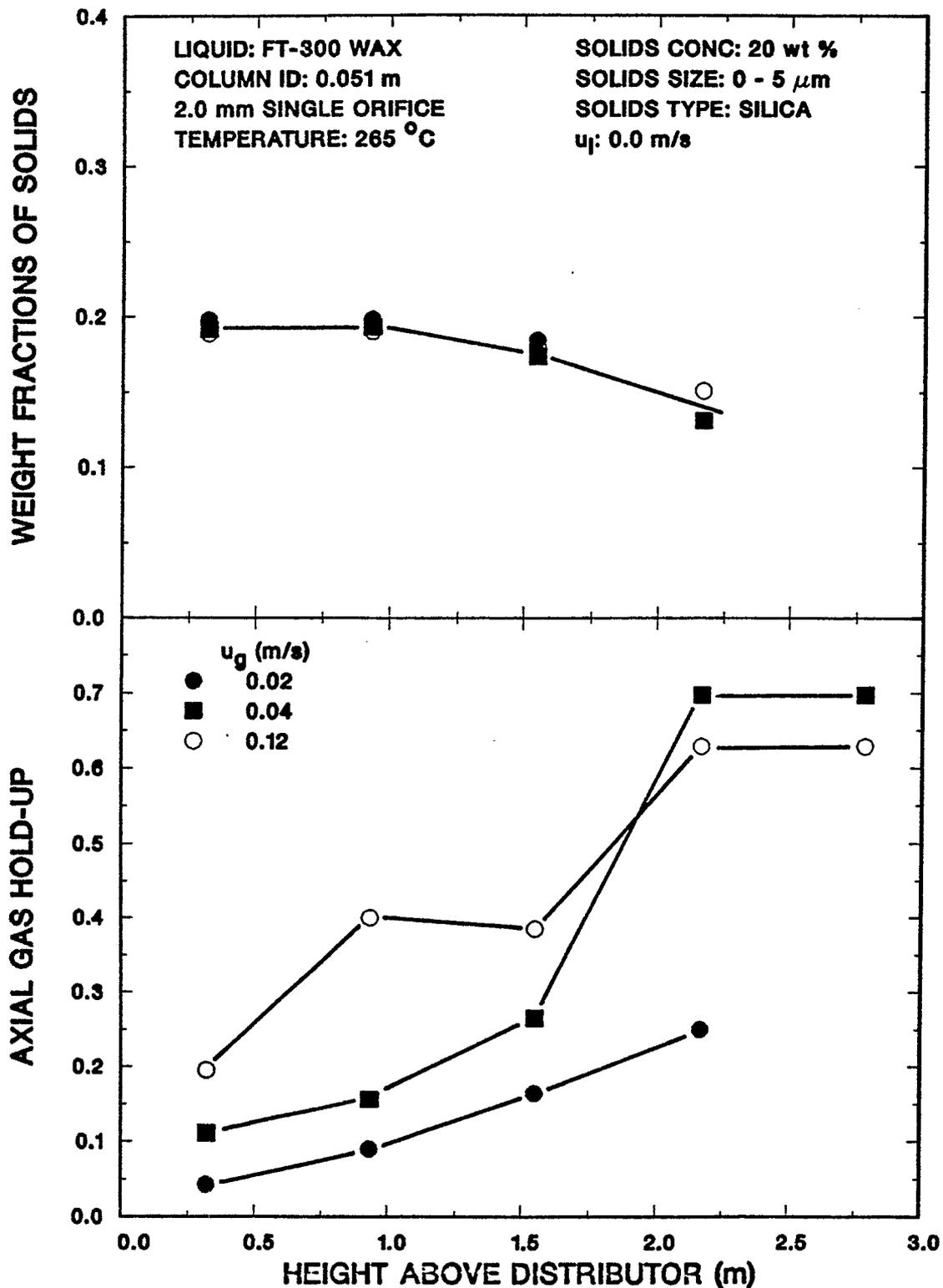


Figure A-4. Effect of superficial gas velocity on axial solids distribution (Top) and on axial gas hold-up (Bottom).

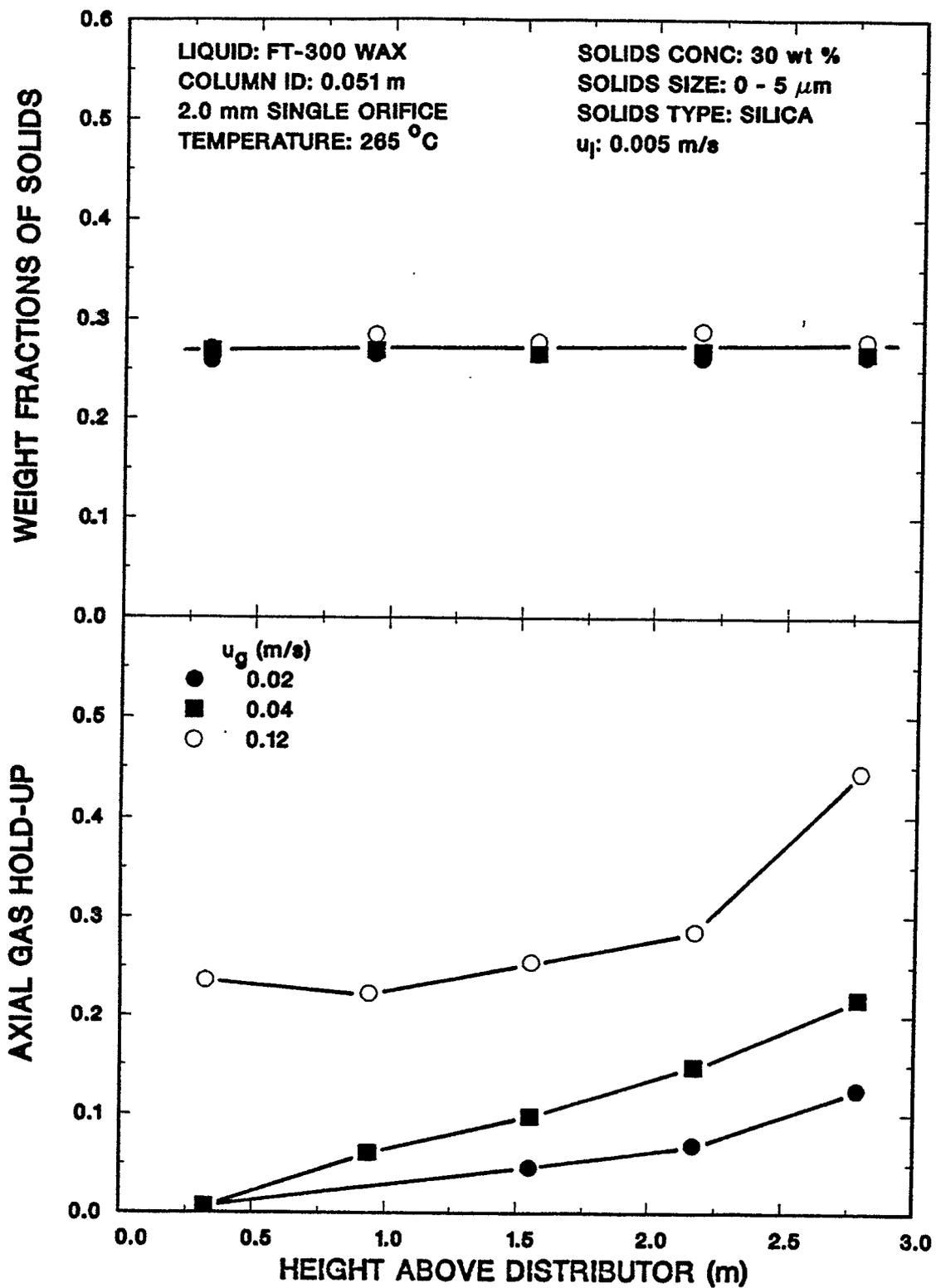


Figure A-5. Effect of superficial gas velocity on axial solids distribution (Top) and on axial gas hold-up (Bottom).

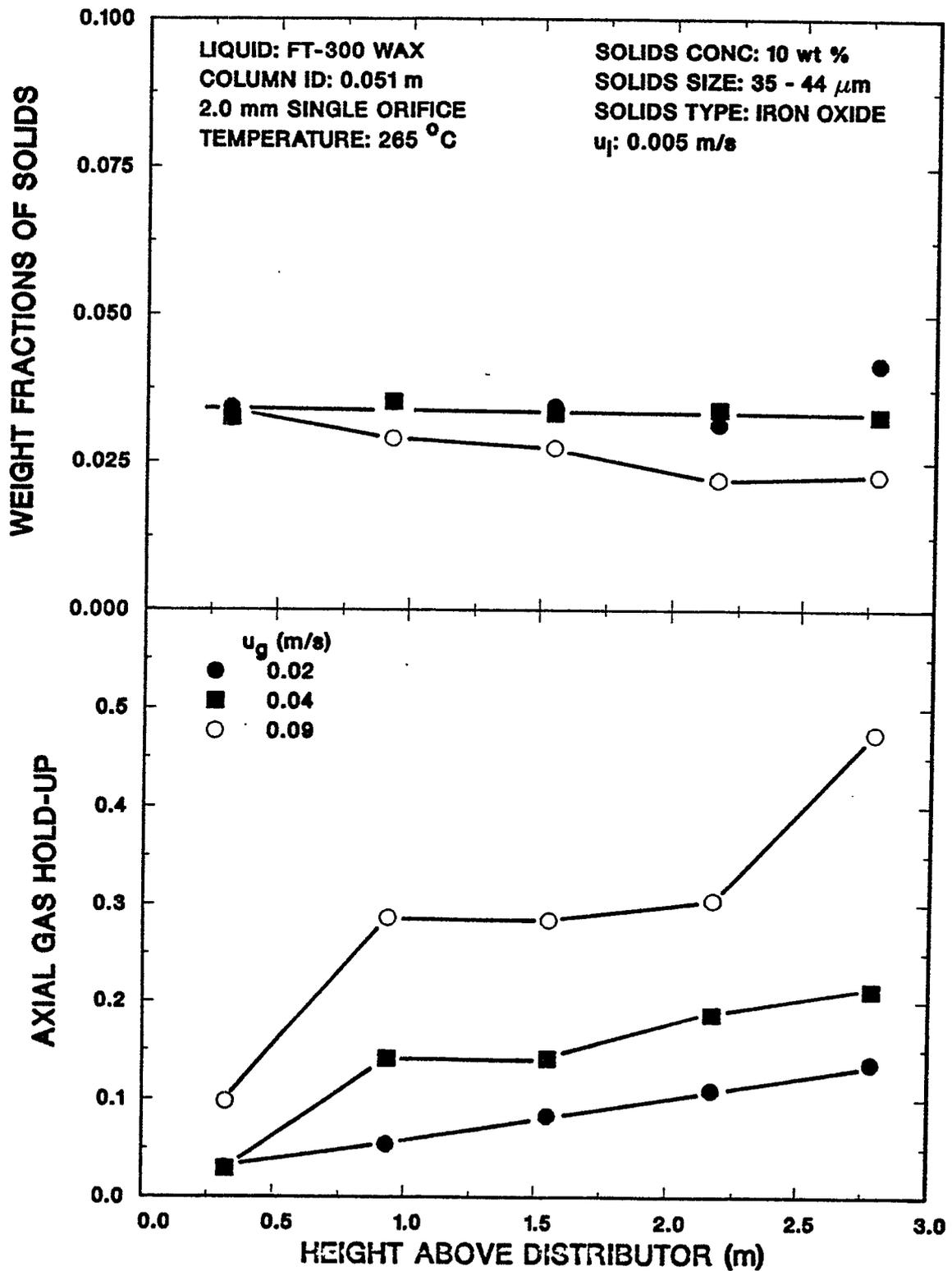


Figure A-6. Effect of superficial gas velocity on axial solids distribution (Top) and on axial gas hold-up (Bottom).

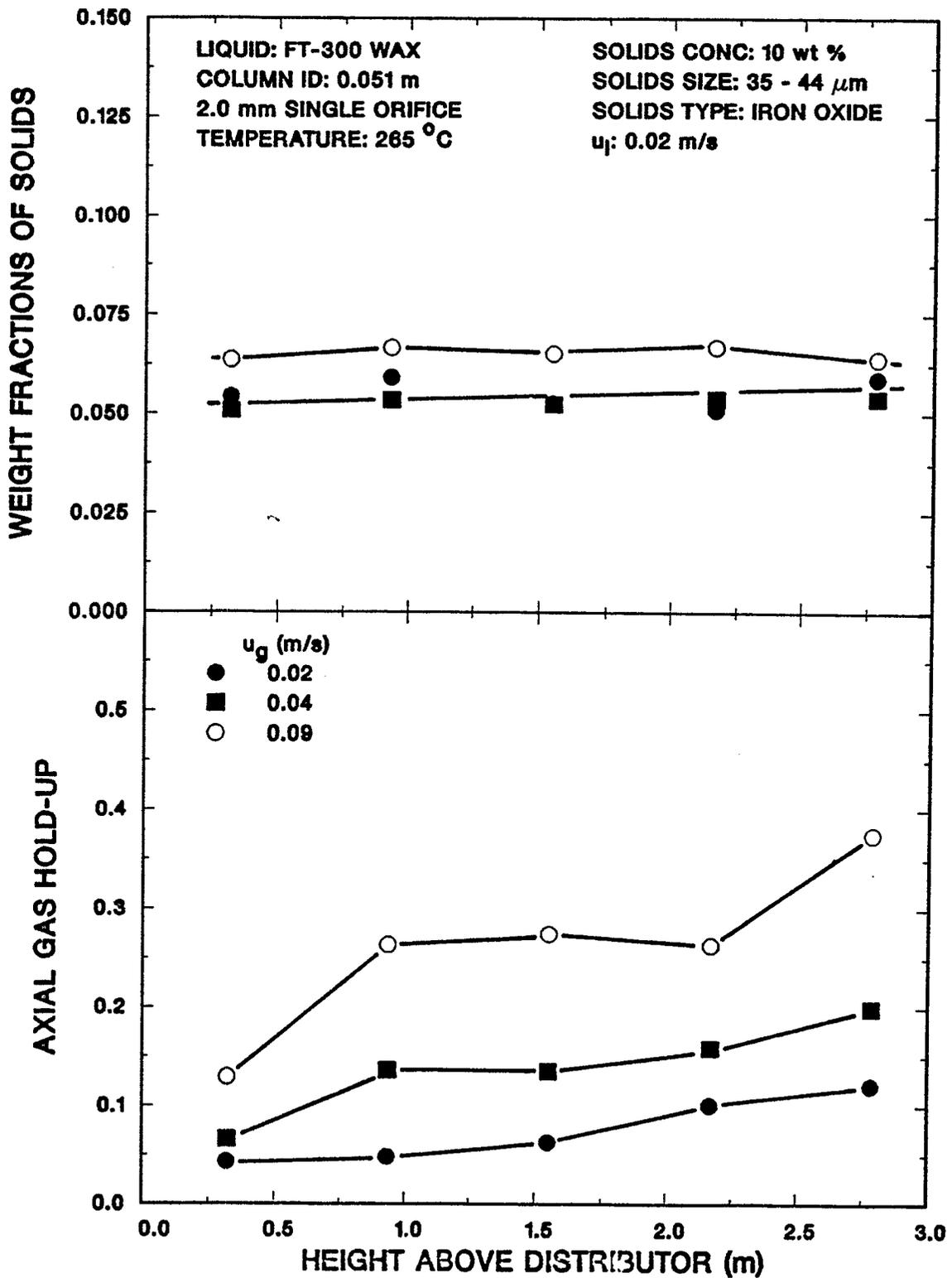


Figure A-7. Effect of superficial gas velocity on axial solids distribution (Top) and on axial gas hold-up (Bottom).

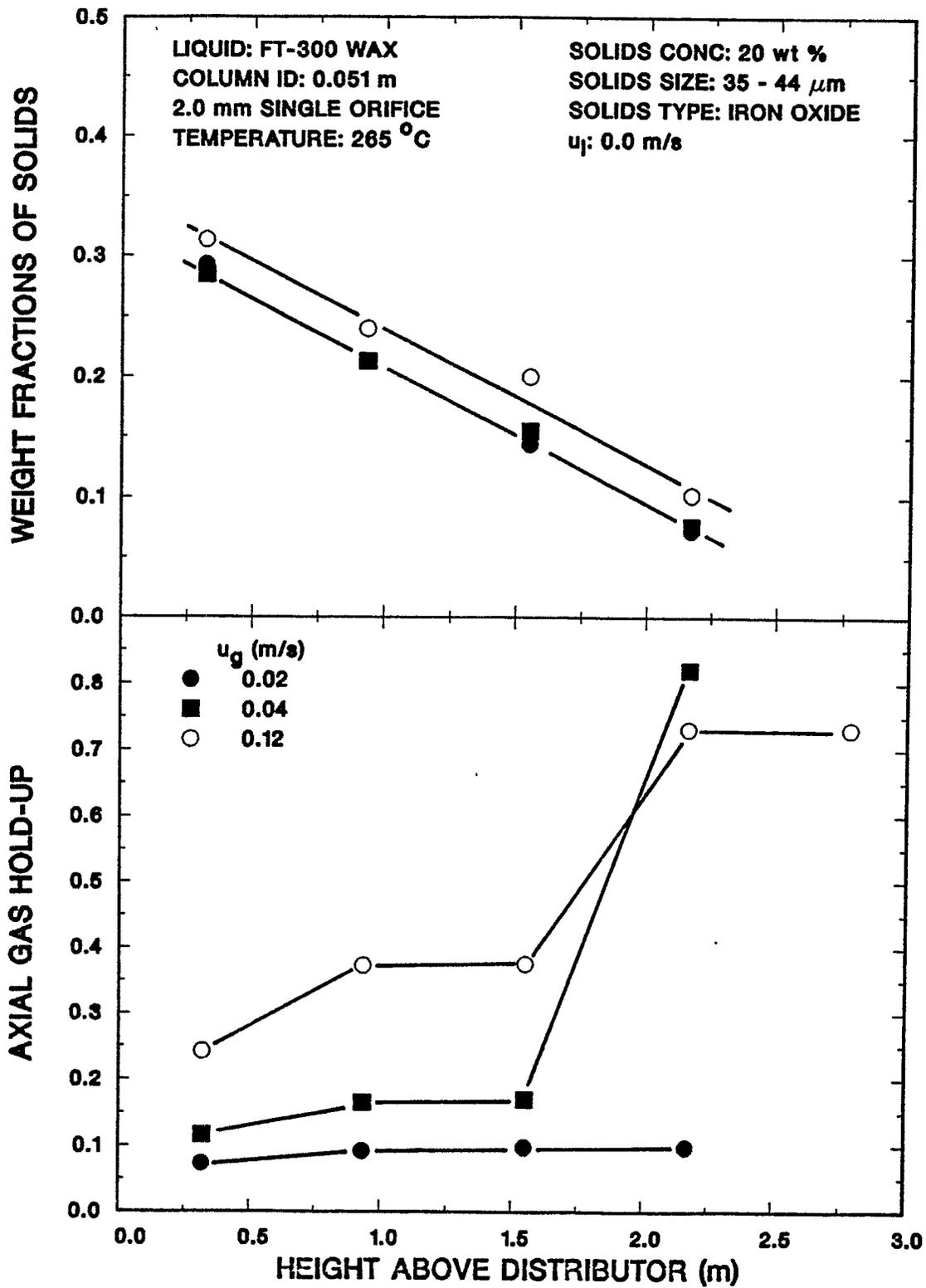


Figure A-8. Effect of superficial gas velocity on axial solids distribution (Top) and on axial gas hold-up (Bottom).

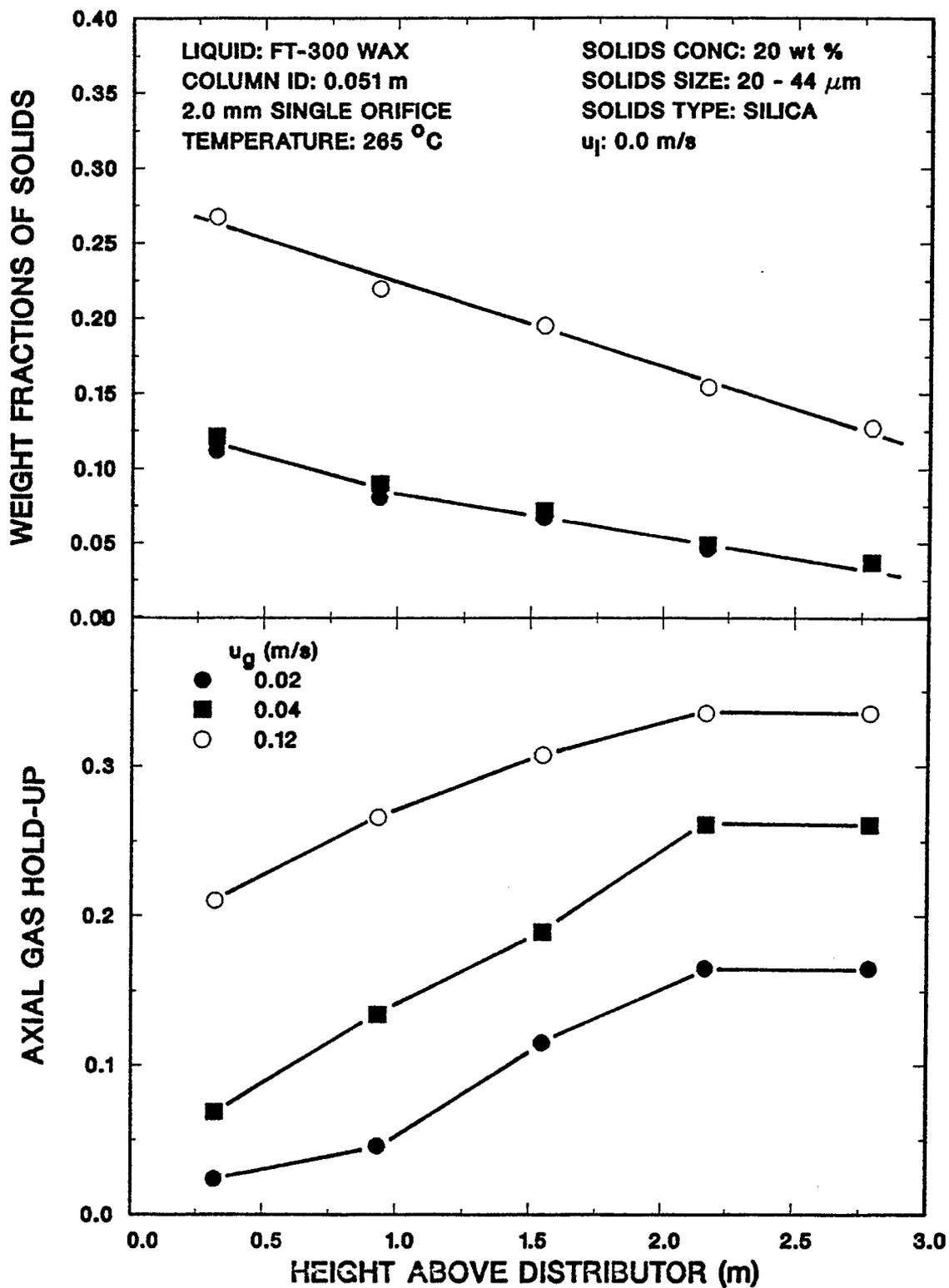


Figure A-9. Effect of superficial gas velocity on axial solids distribution (Top) and on axial gas hold-up (Bottom).