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Hydrodynamics of Fischer-Tropsch Synthesis in Slurry Bubble Column Reactors

Quarterly Technical Progress Report

for the Period 1 September 1986 - 30 November 1986

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I. Abstract

Experiments were conducted in Unit AM–9S (24.1 cm ID, 300 cm tall stainless steel column) with FT–300 wax for the purpose of obtaining photographs of the flow field near the center of the column. During these experiments, measurements of the axial hold–up in the column were also made. Photographs obtained from these experiments were processed using image analysis to convert the photographic data to histographic data. Cumulative bubble size distributions and Sauter mean bubble diameters were then calculated.

Photographs of the flow field, taken in the previous quarter in the Unit AM–9G, were processed using image analysis and the corresponding cumulative distributions and Sauter mean diameters were estimated.

Also, experiments were conducted in Unit AM–2G at 265°C using Mobil and SASOL reactor waxes as the liquid media and nitrogen as the gas. During these experiments average hold–up data as well as dynamic gas disengagement (DGD) data were collected. These data were then analyzed to obtain bubble sizes, volume fraction distributions and Sauter mean diameters.

II. Objective and Scope of Work

The overall objective of this contract is to determine effects of reactor geometry, distributor design, operating conditions (i.e., temperature and gas flow rate), and oxygenated compounds on hydrodynamics of slurry bubble column reactors for Fischer–Tropsch synthesis, using a hard paraffin 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 estimated costs and manhours expended by month for each task.

Task 2 – Bubble Column Reactor Design/Construction

Two bubble columns made of borosilicate glass of approximately 2″ ID and 9″ ID, and 10 ft tall will be designed and assembled for measurement of the gas hold–up and the bubble size distribution. After the design, 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. During this period instruments will be calibrated.

Task 3 – Process Variable Studies

The objective of this task is to determine the effect of various system variables (e.g. gas flow rate, temperature, and addition of minor amounts of oxygenated compounds) on hydrodynamic properties using the two bubble columns (2″ and 9″ ID) and different types of distributors. All experiments will be conducted using nitrogen at atmospheric pressure. It is planned to determine the following hydrodynamic characteristics: gas hold–up, flow regime characterization, bubble size distribution, and the gas–liquid interfacial area.

Task 4 – Correlation Development and Data Reduction

Correlations based on our experimental data for prediction of average gas hold–up and the gas–liquid interfacial area will be developed.

III. Summary of Progress

Results for the Sauter mean bubble diameter and bubble size distribution were obtained by photography in conjunction with image analysis, in Units AM–9G and AM–9S.

Results from Unit AM–9G (T = 265°C, 19 × 1.85 mm perforated plate distributor) for photos taken at three different heights (41 cm, 112 cm and 196 cm above the distributor) indicate that the Sauter mean diameter (d_{vs}) decreases from approximately 0.1 cm at a superficial gas velocity (u_g) of 1 cm/s to approximately 0.04 cm at 9 cm/s. This is different than what was obtained from DGD studies in Unit AM–9G (Run 1–3) where the value for d_{vs} approached 0.1 cm as u_g approached 12 cm/s ("non-foamy" regime). This difference demonstrates the main drawback of photographing the flow field near the wall, i.e. there is a definite bias towards small bubbles. In order to offset this bias photographs of the flow field near the center of the column were taken in Unit AM–9S using the special viewing port at the height 137 cm above the distributor. Results from photographs in Unit AM–9S (T = 265°C, 19 × 1.85 mm perforated plate distributor) indicate that d_{vs} approaches 0.09 cm at higher values of u_g (> 5 cm/s). These results are similar to those from DGD (Run 1–3). Results from photography in Units AM–9G and AM–9S indicate that bubbles near the wall are significantly smaller than those near the center of the column.

Experiments conducted with Mobil and SASOL reactor waxes (T = 265°C, d_o = 1.85 mm) in Unit AM–2G gave average hold–up values which were similar to those for FT–300 wax in the "non–foamy" regime. Unlike FT–300 wax, these waxes showed no tendency to foam over the entire range of velocities. Sauter mean bubble diameters were obtained for these waxes using DGD measurements. These values were significantly different from those obtained for FT–300 wax. Values for d_{vs} approached 0.2 cm for SASOL and 0.5 cm with Mobil wax at high values of u_g (> 7 cm/s) compared to 0.1 cm with FT–300 wax under similar conditions.

IV. Detailed Description of Technical Progress

A. Task 1 – Project Work Plan

The work on this Task was completed during the first quarter of the project.

B. Task 2 – Bubble Column Reactor Design/Construction

The work on this Task was completed during the fourth quarter of the project.

C. Task 3 – Process Variable Studies

3.1 Experiments in the Unit AM – 2G

In majority of the experiments conducted during this project thus far FT–300 was used as the liquid medium. Hydrodynamic properties and foaming characteristics of this wax have been studied in detail during the course of the project. In actual Fischer–Tropsch synthesis paraffin waxes are usually used as the start–up liquid medium, however, their composition changes with time on stream due to the accumulation of heavy molecular weight products and the evaporation of its lighter components. Researchers at Mobil (Smith et al., 1984; Kuo et al., 1985) have found that reactor waxes do not have a tendency to foam, whereas FT–200 paraffin wax foams under similar conditions. Experiments were therefore performed in the 5.1 cm ID, 305 cm tall bubble column (Unit AM-2G) using Mobil reactor wax (obtained from DOE; blend of product reactor waxes from Mobil's Run nos. CT-256-9, CT-256-11 and CT-256-12 conducted in their 10.2 cm ID bubble column) and SASOL reactor wax (obtained from UOP) as the liquid medium, with the following objectives: (1) to study the effect of superficial gas velocity and start–up procedure on the average gas hold–up; and (2) to estimate bubble sizes, volume fraction distributions, and Sauter mean diameters using the dynamic gas disengagement method (DGD). The photographic method for bubble size measurements could not be employed in these experiments due to the dark color of the waxes. These experiments were performed at 265°C using a 1.85 mm single hole orifice plate distributor, and superficial gas velocities of 1 cm/s to 12

cm/s were employed. For superficial gas velocities between 1 and 5 cm/s a minimum time of one and a half hours per velocity was used, and for velocities greater than 5 cm/s a minimum time of one hour per velocity was used.

3.1.1 Experiments with SASOL Reactor Wax

Figure 1 shows average gas hold–up results for runs made using SASOL wax. The liquid static height in these runs was approximately 200 cm. In the run made using increasing order of superficial gas velocities (open circles; Run 8–1) foam was never observed. Hold–up values increased gradually as the gas velocity was increased. These values are similar to those reported earlier (Quarterly Reports September–November 1985, December 1985 – February 1986, March–May 1986) using FT–300 wax in the "non-foamy" regime. Run 8–2 (solid circles) was made using decreasing order of superficial gas velocities (start–up velocity was 12 cm/s). Hold-up values for this run agree very well with those from Run 8–1 (using increasing order of gas velocities). Once again foam was never observed during this run. These results show that unlike FT–300 wax, hold–up values for SASOL reactor wax are not affected by operating procedure.

It was found, from visual observations of the flow field near the wall, that bubbles with this wax are larger than those observed with FT–300 wax under similar conditions. A notable difference was the absence of fine bubbles ($<<1$ mm) that were observed with FT–300 wax. Also the density of bubbles was smaller with this wax in comparison to FT–300 wax. Oscillations at the top of the expanded height had an amplitude in the range 2–10 cm, with larger oscillations occurring at higher velocities. At velocities greater than 3 cm/s slugs occupying the entire column cross section could be observed. A similar behavior was observed with FT–300 wax.

3.1.2 Experiments with Mobil Reactor Wax

Results illustrating the effect of gas velocity and operating procedure on average hold–up for Mobil wax are shown in Figure 2. The liquid static height in these runs was approximately 200 cm. Run 9–1 was made using increasing order of superficial gas velocities (open circles) and Run 9–2 was made using decreasing order of gas velocities (solid circles). Once again foam was not observed in either of the two runs. Operating procedure did not affect the hold–up values for Mobil wax. Hold–up values are similar to those for FT–300 wax in the "non-foamy" regime, and to the ones obtained using the SASOL reactor wax.

Visual observations of the flow field for Mobil wax were limited due to the dark color of this wax which is caused by the presence of small amounts of iron catalyst particles. However, backlighting of the column did show the presence of slugs at superficial gas velocities greater than 3 cm/s. The amplitude of oscillations were similar to those for the SASOL wax and the FT–300 wax.

In general, reactor waxes do not foam for the conditions under which FT–300 wax tends to foam (u_g = 2–5 cm/s). This observation is similar to the one made by workers at Mobil (Smith et al., 1984; Kuo et al., 1985). However, experiments conducted at Mobil with their reactor waxes (Kuo et al., 1985), in their 5.1 cm ID column at 260°C using a 1 mm orifice plate distributor, showed hold–up values which were consistently higher than those found in our experiments. They report hold–up values of 30% at a superficial gas velocity of 10 cm/s as compared to about 20% found in our studies. This difference could be due to the differences in wax used. Experiments at Mobil were conducted with wax from their Run nos. 7 and 8, while the wax used inthis study was from Mobil's Run nos. 9, 11 and 12. Experiments conducted earlier (March–May 1986 Quarterly Report) showed that the addition of oxygenates (5 or 10% by weight) did not prevent foaming of the FT–300 wax. Thus, the reasons for the absence of foam with reactor waxes are still not clearly understood. Additional experiments will be conducted in the AM–2G column with

a sintered metal plate distributor (SMP) to study the effect of distributor type on average gas hold–up. Experiments will also be conducted in the larger columns (Units AM–9G and AM–9S) using SASOL wax to study the effect of column diameter. Experiments will not be conducted with Mobil wax because there is not enough of this wax to charge the large columns.

3.2 Experiments in the Unit AM – 9S

A run was made with FT–300 wax at 265°C in the 24.1 cm ID stainless steel column using a 19 × 1.85 mm perforated plate distributor. The objectives were: (1) to take photographs of the bubbles near the center of the column; (2) to obtain axial gas hold–up data in order to study the effect of column diameter on axial gas hold–up. A minimum of one and a half hours were allowed for velocities from 1 to 5 cm/s and a minimum of one hour was allowed for velocities greater than 5 cm/s.

Photographs were taken through the specially constructed viewing port located 137 cm above the distributor (details were given in the June–August 1986 Quarterly Report). A Canon AE1/P (35 mm SLR) camera and a Vivitar Model 283 flash were used for all photos. The flash was mounted approximately 25 cm from the column at a 45° angle from the front of the column. The camera and a Canon 50 mm lens were mounted on Canon Auto Bellows with an extension of 70 mm. An f/stop of 16 was found to produce the best results. The photographs were taken after a run time of one hour at a given velocity, for velocities of 1 to 5 cm/s, and after 45 minutes for gas velocities greater than 5 cm/s.

Axial gas hold–up measurements were made by differential pressure method using a DP–cell (Validyne Model DP 15 TL). The DP–cell was calibrated with water at room temperature. This was followed by calibration with FT–300 wax at 265°C. The analysis of data from this run has not yet been completed, and results will be discussed in the Final Report. Additional axial gas hold-up measurements will be made in the Unit AM–9S using

SASOL wax.

D. Task 4 – Correlation Development and Data Reduction

4.1 Average Gas Hold – Up

Average hold–up values for the two reactor waxes studied are similar to those for FT–300 wax in the "non–foamy" regime. These values are plotted in Figure 3 along with values for FT–300 wax obtained in the Units AM–2G and AM–9G. All values are for runs made at 265°C. Also plotted is the curve obtained using the correlation for gas hold–up by Bach and Pilhofer (1978). The physical properties (i.e. liquid density and viscosity) required in this correlation were taken from Deckwer et al. (1980, 1982). This figure shows that in the absence of foam, wax type, distributor type and column diameter do not affect the average gas hold–up. Results from Mobil's experiments with FT–200 wax (Kuo et al., 1985) are also shown in this figure. Bach and Pilhofer's correlation is in good agreement with these results and can be used to predict average hold–up values for the different waxes in the "non–foamy" regime.

4.2 Dynamic Gas Disengagement

The dynamic gas disengagement technique developed by Sriram and Mann (1977) was used to estimate bubble size distributions for runs made with SASOL and Mobil reactor waxes. The theory, related equations, and procedure for this technique were presented earlier (Quarterly Reports March–May 1986 and June–August 1986) and are not repeated here.

Dynamic gas disengagement data for Run 8–1 (SASOL wax) and Run 9–2 (Mobil wax), conducted in the 5.1 cm ID column, were analyzed. The results from these analysis are summarized in Tables 1 and 2. Gas hold–up values for these two runs were similar and comparable to gas hold–up values in the "non–foamy" regime with FT–300 wax. The curve for the normalized change in liquid height liquid level as a function of time could be

depicted by three straight lines for all velocities. This gave rise to three dominant bubble sizes. The shapes of these curves were similar for the different velocities, unlike FT–300 (March–May 1986 Quarterly Report) where curves for velocities when foam was produced were different from velocities when no foam was present.

4.2.1 Bubble Size Distributions

The effect of superficial gas velocity on bubble size for SASOL wax is shown in Figure 4. The size of small bubbles remains essentially unchanged between 0.3 and 0.4 mm with changes in velocity. This is similar to FT–300 (March–May 1986 Quarterly Report) where the small bubbles were around 0.4 mm. Medium bubbles are between 0.5 and 0.6 mm in diameter and their size is also unaffected with changes in gas velocity. Large bubbles, however, increase in size as velocity is increased and approach the column diameter at high gas velocities ($u_g > 5$ cm/s). This could be attributed to the presence of slugs, which is in agreement with visual observations.

The variation of bubble sizes with superficial gas velocity for Mobil wax is shown in Figure 5. The results are qualitatively similar to those for SASOL wax, however, individual bubble sizes are significantly larger than corresponding sizes for SASOL wax. Small bubbles are between 0.5 and 0.6 mm in diameter, while medium size bubbles are approximately 1 mm in diameter at most velocities. Medium size bubbles at 9 and 12 cm/s are significantly larger than those at lower velocities, this is probably due to experimental error. Large bubbles show an increase in size with an increase in gas velocity and approach the column diameter at higher velocities ($u_g > 5$ cm/s).

Limitations of the DGD technique coupled with approximate values for physical properties of the waxes used in this study prevent us from making definite conclusions regarding bubble sizes. However, these results are in fairly good agreement with visual observations of the gas–liquid dispersion.

4.2.2 Sauter Mean Diameters

The bubble size and number distributions obtained from DGD data reduction were used to calculate the Sauter mean diameters and these values are shown in Figure 6. Also shown here are values for FT–300 wax obtained in the previous quarter. Despite similar hold–up values, the Sauter mean diameters for the three waxes are significantly different.

For FT–300 wax the Sauter mean diameters stabilized at 1 mm at high superficial gas velocities ($u_g > 5$ cm/s). Sauter mean diameters for SASOL wax approach 2 mm at high velocities ($u_g > 5$ cm/s) and those for Mobil wax approach 5 mm at $u_g = 12$ cm/s. These results are supported by visual observations of the dispersion. Since the Sauter mean diameter and the average gas hold-up determine specific interfacial area available for mass transfer, it is obvious that for the same value of gas hold–up, larger Sauter mean diameters would result in lower interfacial areas. These findings have significant implications on bubble column reactor scale–up and design.

4.3 Bubble Size Distributions by Photographic Technique

Selected photographs of bubbles in the Unit AM–9G taken during the previous quarter were analyzed using an image analyzer. Additional photographs of bubbles near the center of the column taken through the special viewing port in the Unit AM–9S were also analyzed. A detailed description of the technique used was given in the previous Quarterly Report and is not included here. In most cases two photos were analyzed for a given condition and the results were combined. In general, approximately 1500 bubbles per condition were counted, sized and analyzed.

4.3.1 Bubble Size Distributions – Near the Column Wall

Photographs of bubbles in the Unit AM–9G were taken along the wall at three different heights (41 cm, 112 cm and 196 cm above the distributor). The Sauter mean bubble diameters estimated from bubble size distributions are shown in Figure 7.

Results show that the Sauter mean diameter tends to decrease initially as gas velocity is increased and approaches a constant value at higher gas velocities. At 41 cm above the distributor the Sauter mean diameter decreases from approximately 1.2 mm at 1 cm/s to approximately 0.7 mm at higher velocities. Visual observations of the flow field support these results. However, at 112 cm and 196 cm above the distributor, bubbles are significantly smaller at high velocities, approaching 0.4 mm at a gas velocity of 9 cm/s. Again, this is in agreement with visual observations. At high gas velocities, bubbles near the wall form a very fine dispersion.

Zaidi et al. (1979) and Deckwer et al. (1980) report Sauter mean diameters of about 0.7 mm for molten paraffin wax in bubble columns equipped with porous plate spargers. Quicker and Deckwer (1981) estimated Sauter mean diameters for a hard paraffin wax in a bubble column using porous plate and perforated plate spargers. They took photographs of bubbles along the column wall for experiments performed in the homogeneous flow regime. Their results (at 170°C, d_o=0.9 mm), included in Figure 7, show that d_{vs} is around 2.5 mm at low velocities (u_g<1 cm/s) and it decreases to about 0.7 mm at a velocity of 4 cm/s. Sauter mean diameter values for FT–300 from the current study at a height of 41 cm above the distributor, where the flow regime is homogeneous, are in good agreement with the findings of these workers considering the differences in distributors and operating conditions.

4.3.2 Bubble Size Distributions – Near the Column Center

Photographs of bubbles near the center of the column were taken in the Unit AM–9S through the special viewing port for gas velocities from 1 to 9 cm/s. The height of the location where photos were taken was 137 cm above the distributor. Two photos at each velocity were selected and analyzed, and Sauter mean diameters for the individual photographs calculated. Results from the two photos were then combined and the Sauter

mean diameter for the combined data was calculated. Figure 8 shows the Sauter mean diameter for the combined data (open circles) as well as Sauter mean diameters from the individual photographs (connected by the vertical bar) at each velocity.

Visual observations of the flow field through the viewing port showed bubbles that were significantly larger than those observed near the wall in the Unit AM–9G. Bubbles upto 4–5 cm in diameter were visible through the window at high gas velocities, whereas only fine bubbles were visible at the wall in the glass column. Sauter mean diameters shown in Figure 8 further support this observation. The diameters are significantly larger than those obtained from Unit AM–9G at the three different locations (Figure 7). Sauter mean diameters reach a maximum of 1.5 mm at 3 cm/s and then stabilize at approximately 0.9 mm for higher gas velocities. Sauter mean diameters for the individual photographs, at a given velocity, compare satisfactorily considering the limitations of this procedure.

4.3.3 Comparison of Bubble Size Distributions from DGD and Photography

Three different approaches were used to estimate the Sauter mean diameter of bubbles in the larger columns (Units AM–9G and AM–9S) using FT–300 wax. The dynamic gas disengagement technique gives an average for the entire column; photographs of bubbles near the wall and near the center of gave point estimates of the Sauter mean diameter. Results from the DGD technique could be considered as the base case since this technique accounts for all bubbles unlike the photographic technique. Results for runs made at 265°C are summarized in Figure 9. DGD results for the Unit AM–9G from the previous quarter are shown along with results from photos in Unit AM–9G at a height of 196 cm, and results from Unit AM–9S (at a height of 137 cm).

Figure 9 illustrates the tendency of large bubbles to migrate towards the center of the column, leaving tiny bubbles close to the wall. These results also show that Sauter mean diameters obtained using photographs taken near the center of the column in the

Unit AM–9S are in good agreement with those from DGD at gas velocities greater than 4 cm/s. The lower Sauter mean diameter at 3 cm/s for values from DGD is because of the presence of foam at that velocity. Results from DGD and photography suggest that the Sauter mean bubble diameter for FT–300 wax at superficial gas velocities greater than 4 cm/s is approximately 1mm.

V. Future Work

The following activities are planned for the next quarter

(a) Conduct additional experiments with reactor waxes at 200°C and with the sintered metal plate distributor in the Unit AM–2G, and with SASOL wax in the Unit AM–9G.

(b) Complete analysis of axial hold–up data taken in this quarter. Make additional measurements of axial hold–up in Unit AM–9S using SASOL wax.

(c) Measure physical properties (density and viscosity) of FT-300, SASOL, Mobil and FT–200 waxes.

(d) Develop correlations for the prediction of specific interfacial area.

(e) Write the Final Report.

VI. Nomenclature

d_{bi}	diameter of bubbles of size i (cm)
d_c	bubble column diameter (cm)
d_o	orifice hole diameter (mm)
d_{vs}	Sauter mean diameter
f_i	fraction of bubbles of size i
T	column temperature (°C)
u_g	superficial gas velocity (cm/s)

Greek Letters

ϵ_g	average gas hold–up (%)

Acronyms

DGD	dynamic gas disengagement
DOE	Department of Energy
FT	Fischer Tropsch
ID	inside diameter
PP	perforated plate
SMP	sintered metal plate

Subscripts

g	gas
L	large bubbles
M	medium size bubbles
S	small bubbles

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Table 1. Hydrodynamic parameters obtained from dynamic gas disengagement method (Unit AM–2G, SASOL wax, Run 8–1, T = 265°C, d_o = 1.85 mm)

u_g (cm/s)	ϵ_g (%)	d_{BS} (cm)	d_{BM} (cm)	d_{BL} (cm)	f_S	f_M	f_L	d_{vs} (cm)
1	4.65	0.041	0.114	2.320	0.31	0.26	0.43	0.101
2	7.59	0.040	0.059	2.050	0.21	0.22	0.57	0.108
3	9.99	0.036	0.055	2.910	0.18	0.21	0.62	0.112
4	12.03	0.038	0.055	3.370	0.15	0.16	0.69	0.140
5	13.33	0.035	0.055	2.370	0.12	0.15	0.73	0.153
7	16.71	0.034	0.050	5.320	0.10	0.11	0.78	0.184
9	19.22	0.033	0.055	3.150	0.12	0.08	0.81	0.193

Table 2. Hydrodynamic parameters obtained from dynamic gas disengagement method (Unit AM–2G, Mobil wax, Run 9–2, T = 265°C, d_o = 1.85 mm)

u_g (cm/s)	ϵ_g (%)	d_{BS} (cm)	d_{BM} (cm)	d_{BL} (cm)	f_S	f_M	f_L	d_{vs} (cm)
1	4.86	0.051	0.310	1.590	0.35	0.21	0.44	0.129
2	7.64	0.050	0.111	3.070	0.29	0.17	0.54	0.133
3	9.35	0.049	0.088	2.850	0.17	0.11	0.71	0.197
4	11.24	0.051	0.092	3.240	0.17	0.09	0.74	0.223
5	13.19	0.053	0.136	3.550	0.14	0.09	0.77	0.289
7	15.96	0.050	0.113	4.720	0.11	0.08	0.81	0.321
9	19.21	0.052	0.450	5.100	0.11	0.11	0.78	0.408
12	21.84	0.058	0.390	4.730	0.08	0.09	0.83	0.550

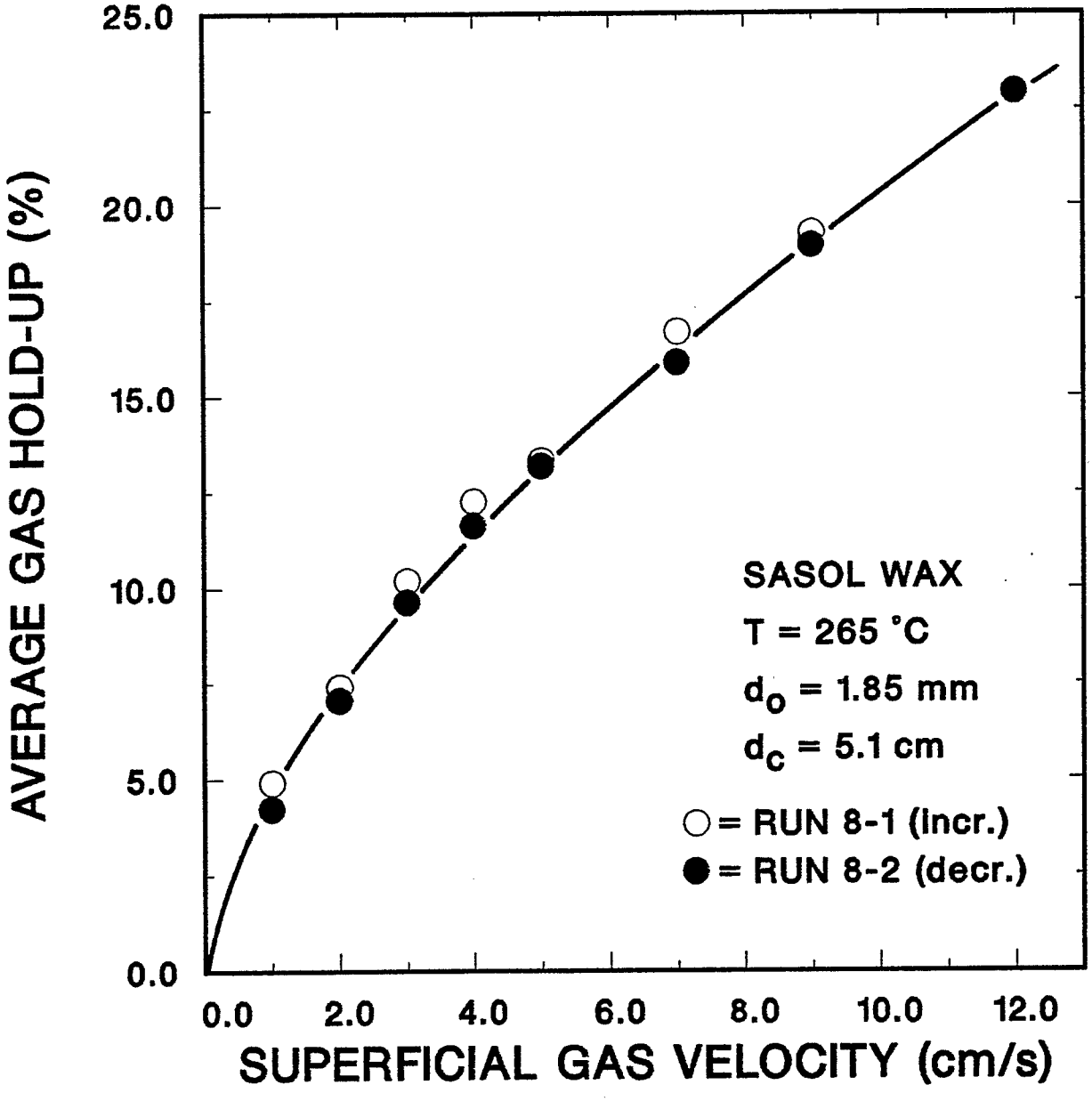

Figure 1. Effect of gas velocity and operating procedure on the average gas hold-up for SASOL wax

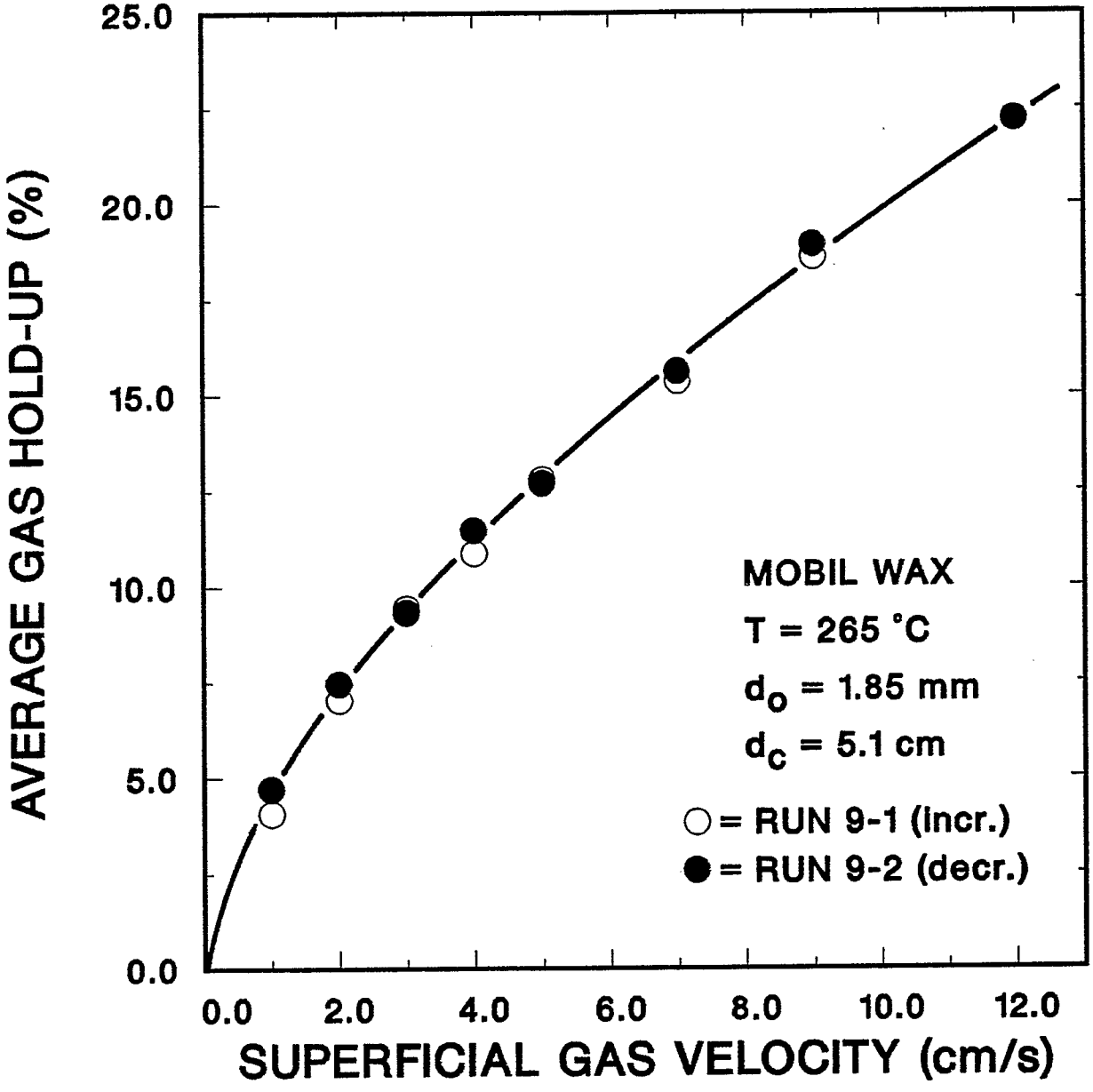

Figure 2. Effect of gas velocity and operating procedure on the average gas hold–up for Mobil wax

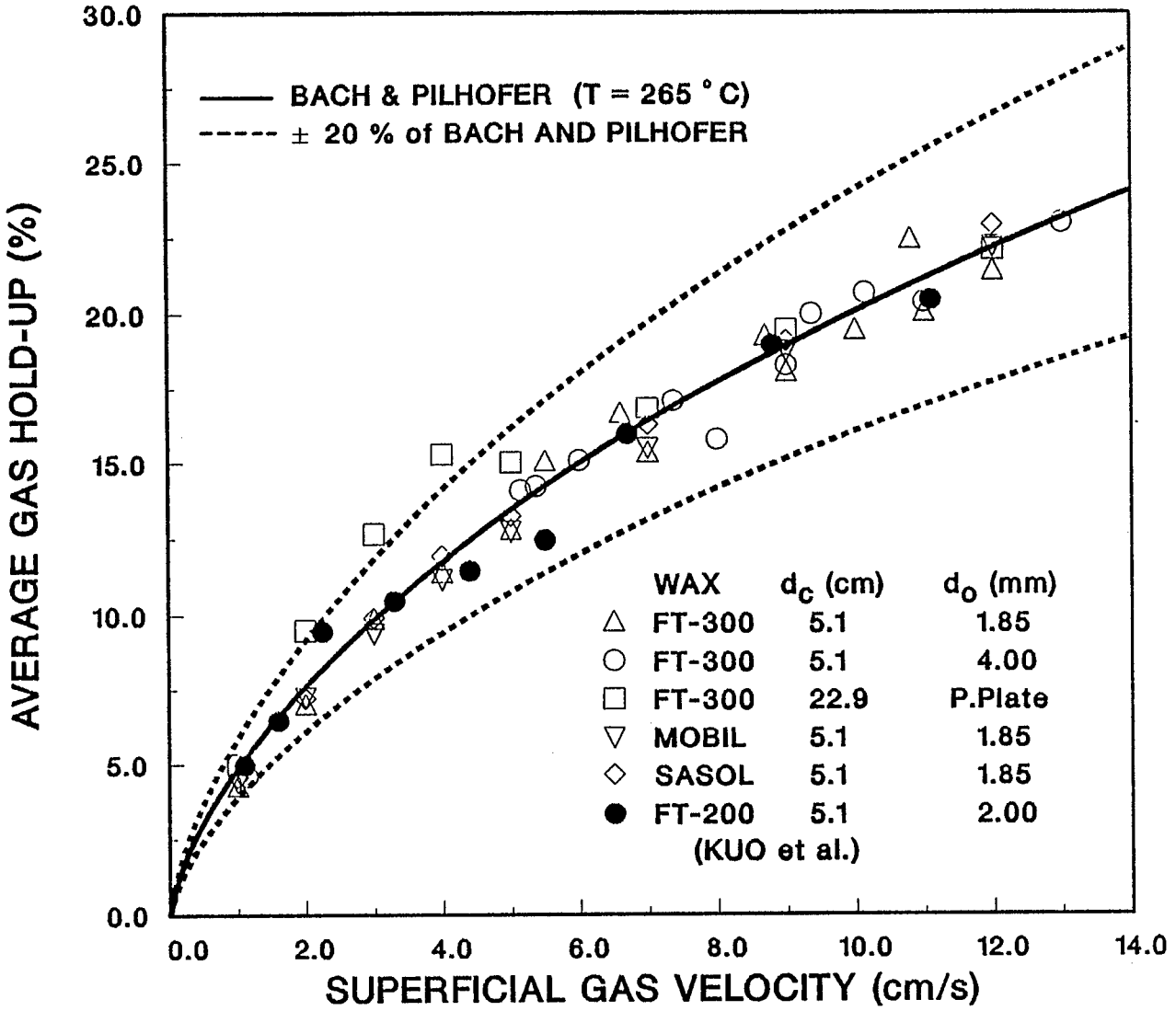

Figure 3. Effect of wax type, distributor type and column diameter on average gas hold-up in the "non-foamy" regime and comparison with literature

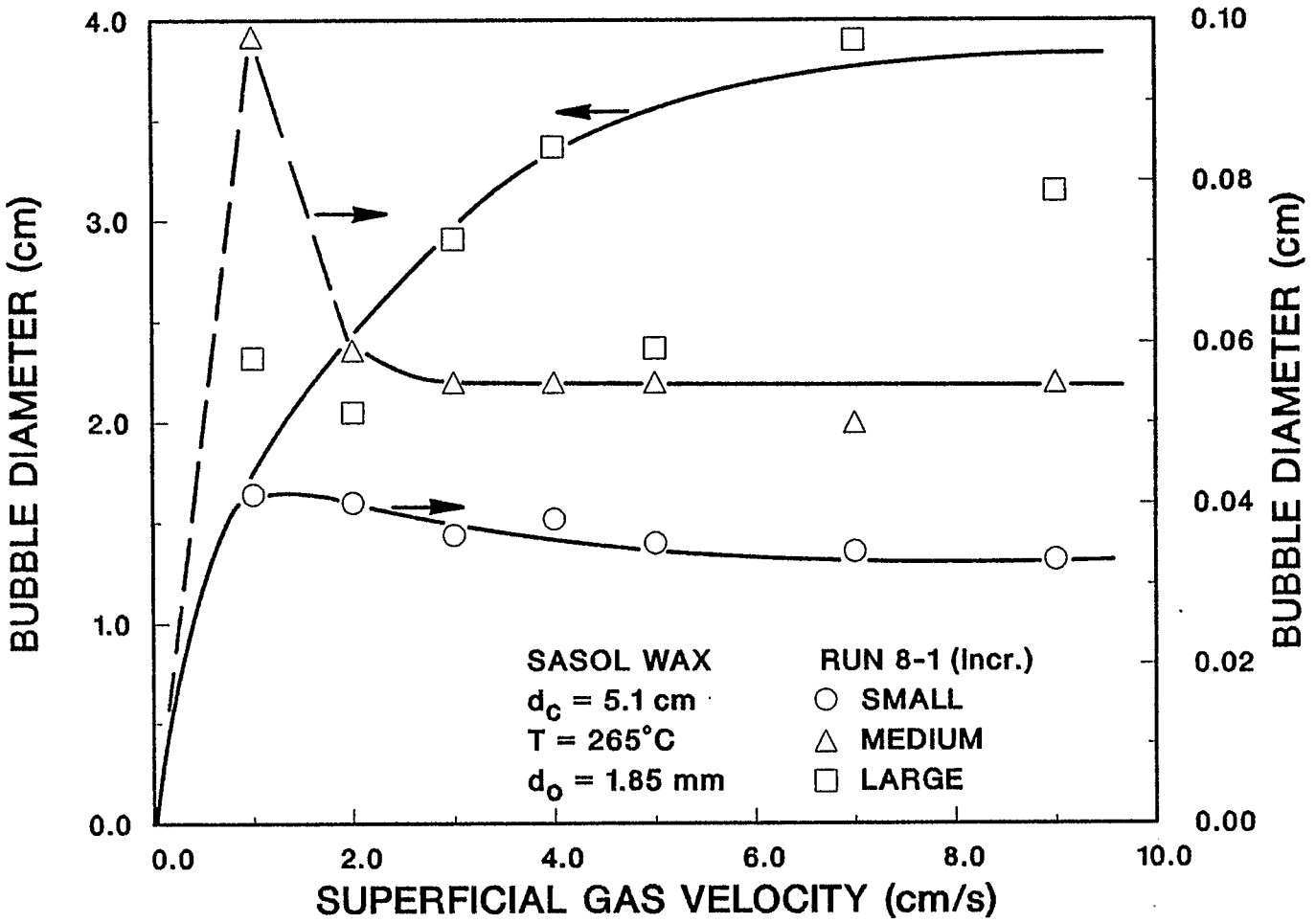

Figure 4. Effect of superficial gas velocity on bubble size distribution for SASOL wax (DGD method)

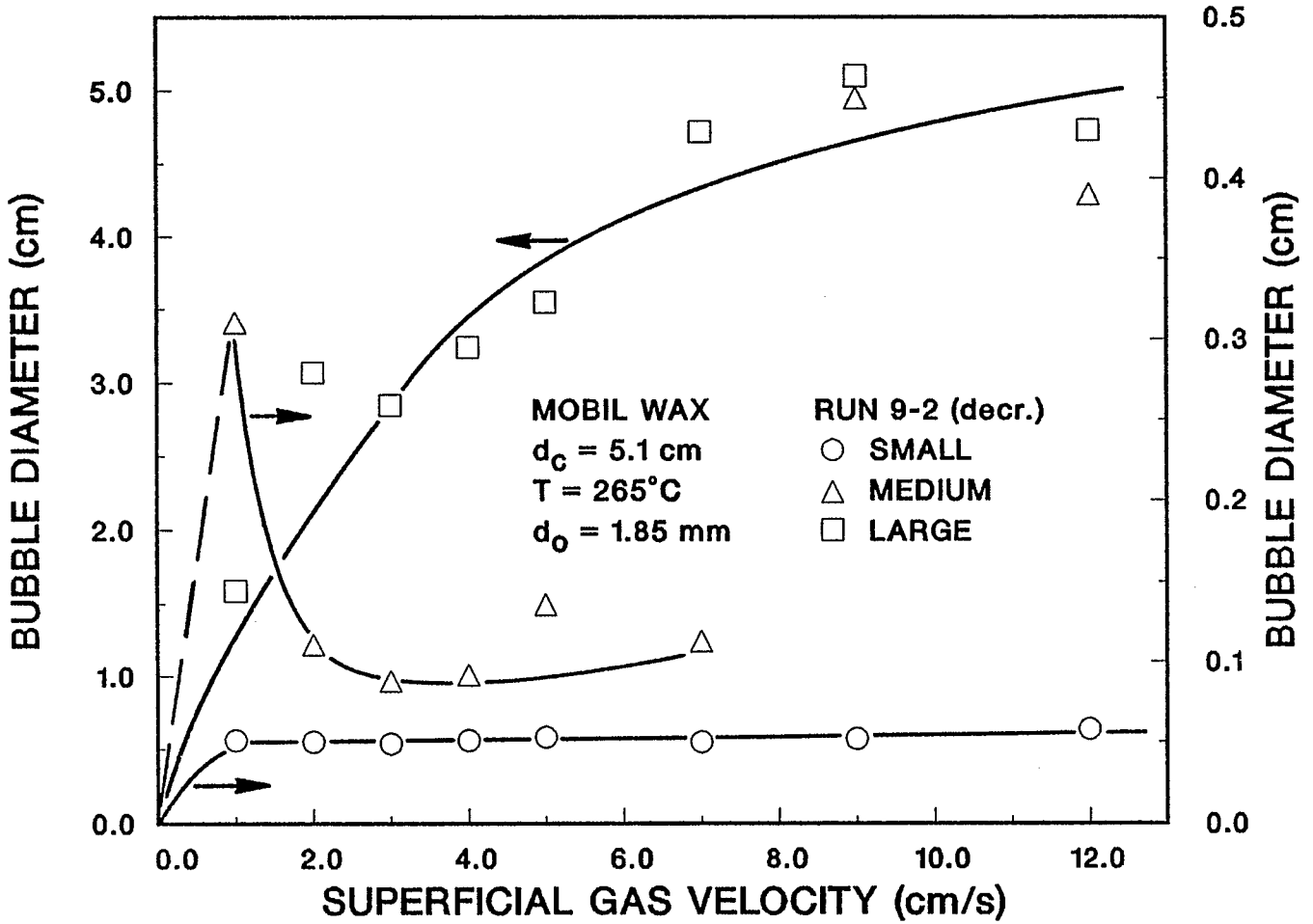

Figure 5. Effect of superficial gas velocity on bubble size distribution for Mobil wax (DGD method)

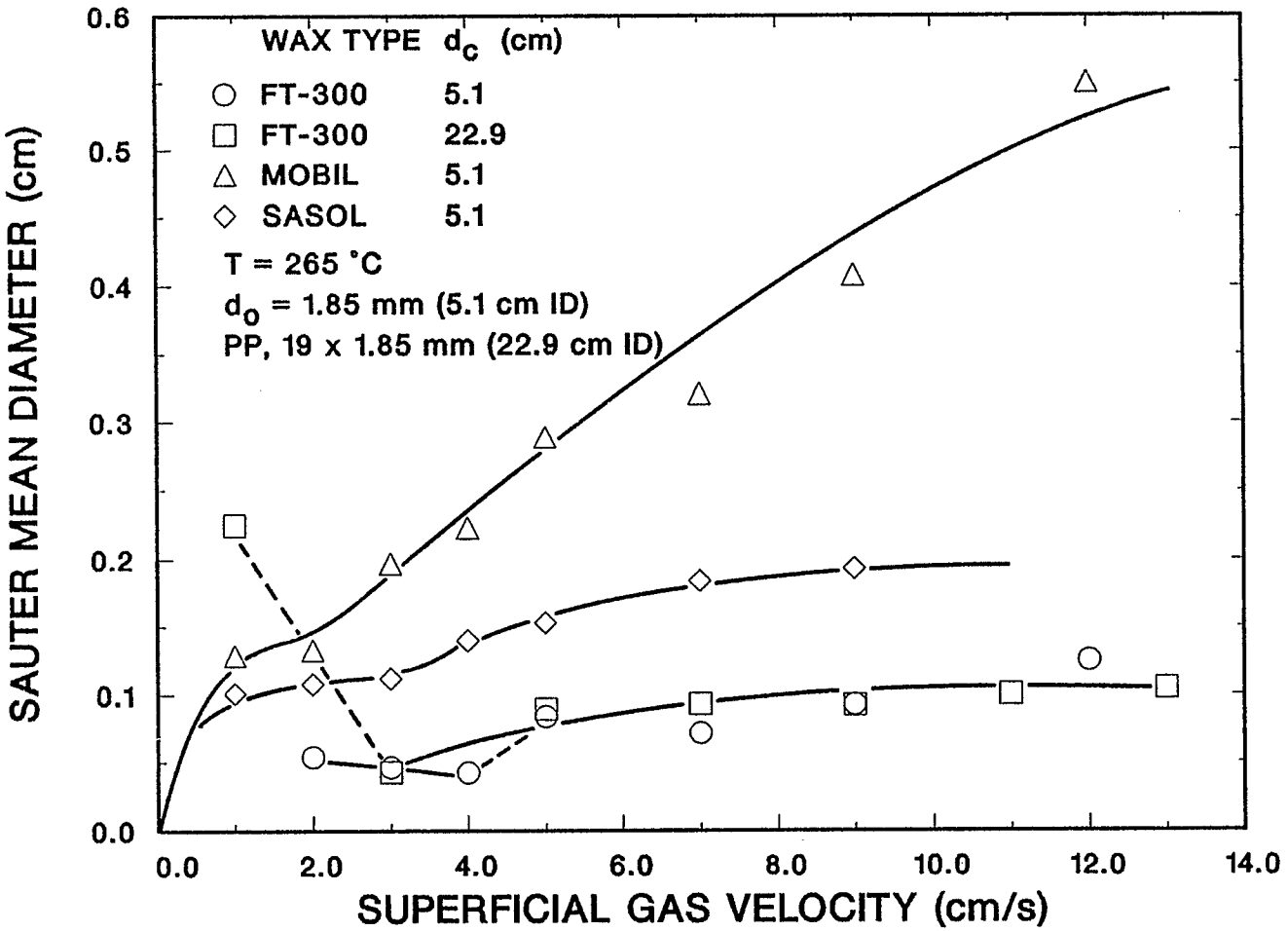

Figure 6. Effect of wax type and column diameter on the Sauter mean diameter (DGD method)

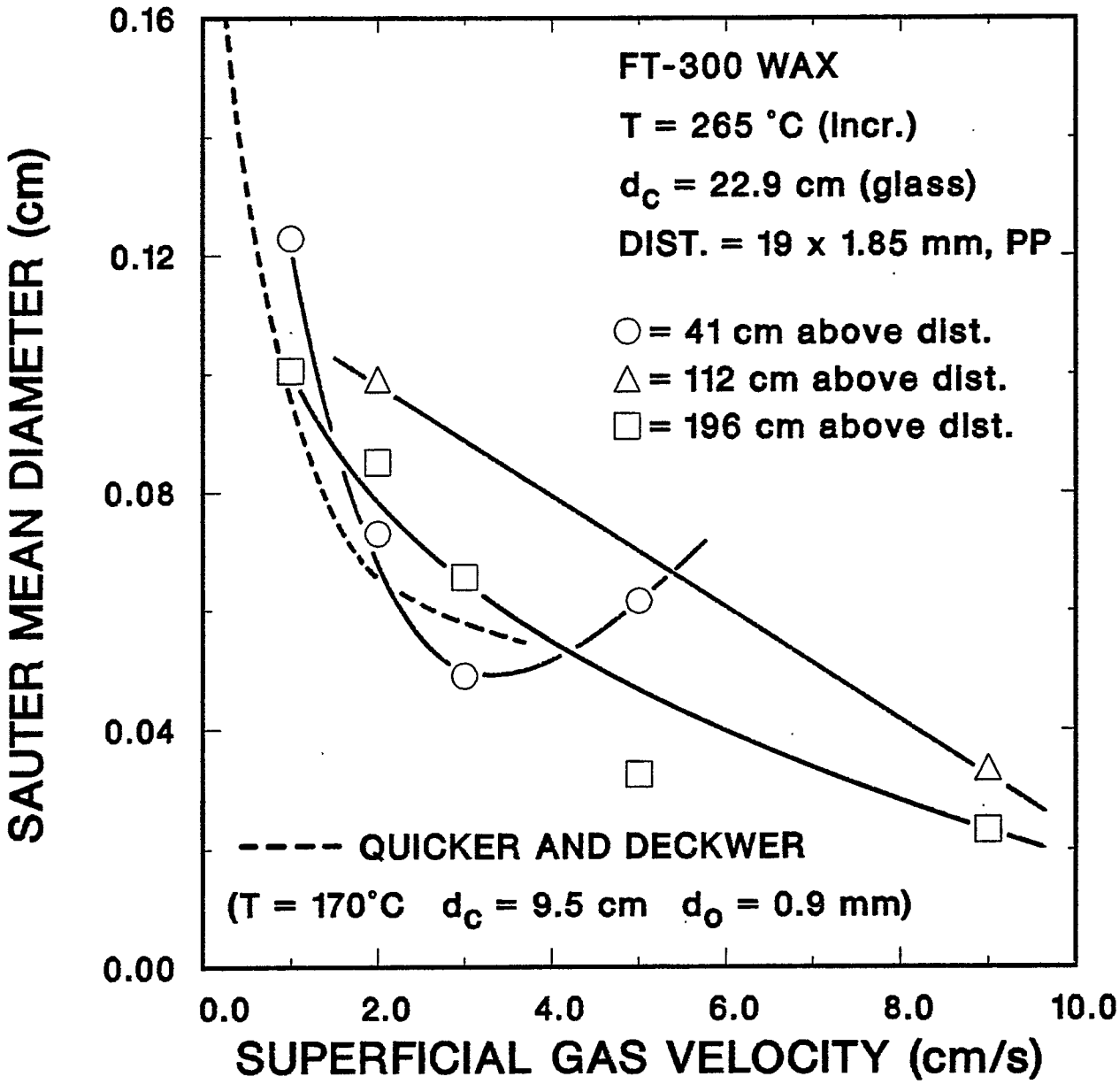

Figure 7. Effect of superficial gas velocity and height above distributor on the Sauter mean diameter and comparison with literature (Photos taken at the wall of the column)

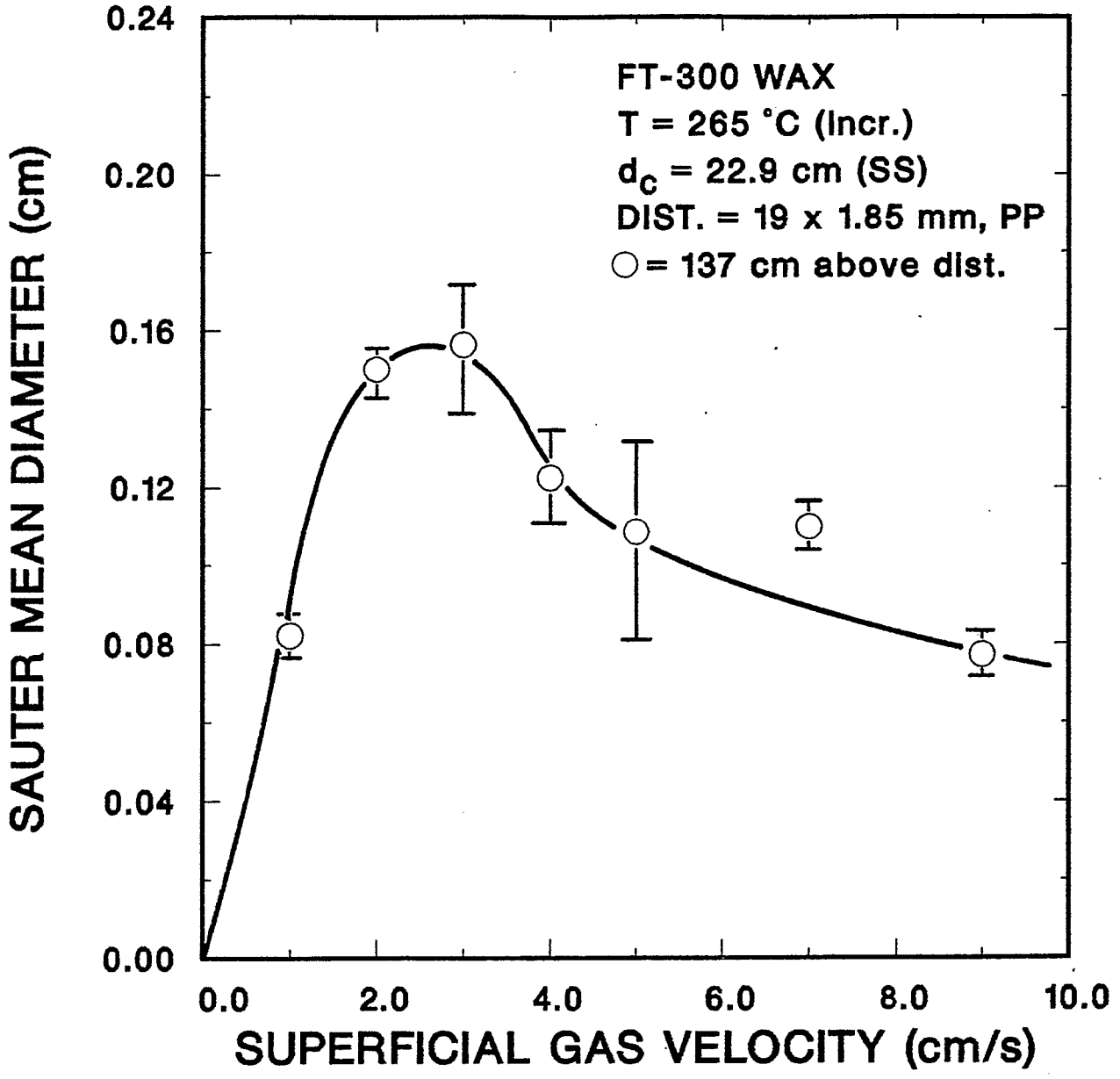

Figure 8. Effect of superficial gas velocity on the Sauter mean diameter (Photos taken near the center of the column; vertical bars join Sauter mean diameters from individual photographs, open circles represent Sauter mean diameters when results from the two photos are combined)

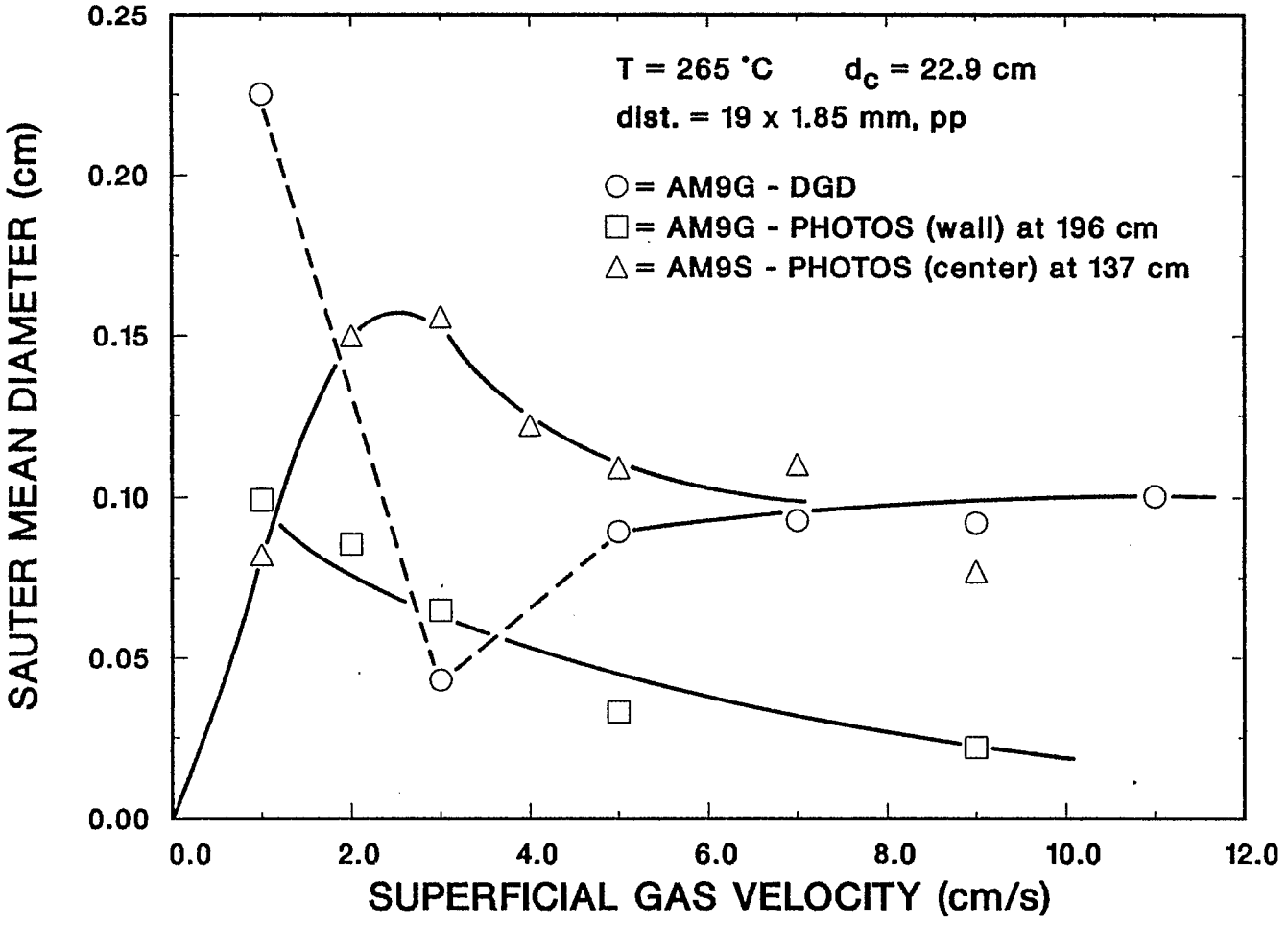

Figure 9. Comparison of techniques used to determine the Sauter mean diameter