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Hydrodynamics of Fischer-Tropsch Synthesis

in Slurry Bubble Column Reactors

Quarterly Technical Progress Report

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Dragomir B. Bukur, Russell F. Brown, James G. Daly and Dragan Petrovic

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

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I. ABSTRACT

Measurements of the average gas holdup were made in a small glass column (5.1 cm ID, 305 cm tall) with three distributors (1.85 mm and 4 mm single hole orifice plate and 40 μ m sintered metal plate) at 265-C, and in a large glass column (22.5 cm ID, 300 cm tall) equipped with a perforated plate distributor (19 holes of 1.85 mm in diameter equally spaced in a triangular pitch) at 200-C and 265°C using FT-300 paraffin wax as the liquid medium and nitrogen as the gas. The hysterisis behavior on the ε_g vs. u_g , which was reported in the previous quarter, was observed again in experiments conducted in the small glass column.

The flow patterns were photographed and video taped in both columns. In the small column slugs or a slug type large bubbles develop at superficial gas velocities greater than 3 cm/s, while in the large column slugging does not occur over the entire range of gas velocities (1-12 cm/s) that was investigated.

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

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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

During this quarter additional experiments have been performed in the small glass column (5.1 cm ID, 305 cm tall) at 265°C with three distributors (1.85 mm and 4 mm single orifice plate and 40 μm SMP) using FT-300 paraffin was as the liquid and nitrogen as the gas. In experiments with increasing gas velocity on abrupt transition from the "foamy" to the liquid circulation regime occurred with the 1.85 mm orifice plate when the velocity was increased from 4 cm/s to 5 cm/s. This transition was smoother with the 4 mm orifice plate, and with the 40 μ m SMP distributor it never occurred over the range of velocities that was employed (1-24 cm/s). It has been confirmed that if one uses a high startup velocity the liquid circulation regime is attained with all three types of distributors. The gas holdups obtained using the high startup velocity are not exactly the same as the ones obtained in the experiments with increasing gas velocity. Thus, for this system it is difficult to assert whether the liquid circulation regime is the only stable flow regime at high gas velocities. Experiments with the 1.85 mm orifice plate distributor show that an order of experiments (increasing or decreasing velocities) and/or duration of experiments have a strong effect on the average gas holdup. Comparison between the new results and the previous ones (June-August, 1985 Quarterly Report) reveals that there is excellent agreement for data in the liquid circulation regime, but that transitions between the two flow regimes and the gas holdups in the "foamy" regime are not completely reproducible. The latter is probably caused by differences in operating procedures, order of experiments and duration of experiments.

The effect of temperature (200°C and 265°C) and superficial gas

velocity (1-12 cm/s) on the average gas holdup was studied in the large glass column (22.5 cm ID, 300 cm tall) with a perforated plate distributor having 19 holes of 1.85 mm in diameter equally spaced in a triangular pitch. In experiments at 200 °C it was found that the gas holdup is a monotone increasing function of gas velocity and no foam was observed on top of the liquid level. At 265 °C a foam layer forms on top of the liquid level at lower superficial gas velocities (1-5 cm/s), but its height decreases as the velocity increases and the holdup passes through a local maximum. This type of behavior is characteristic for the "foaming" regime which transforms into the liquid circulation regime at higher gas velocities.

The flow patterns were photographed during the gas holdup measurements in both columns. Video tapes have been reviewed and qualitative analysis of the bubble size distribution and flow regimes has been completed. An important finding is that slugging has not been observed in the large glass column (the largest bubbles seen are about 3 cm in diameter), while it always occurs to some extent in the small column for all three types of distributors.

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

1. Review of Literature on the Gas Holdup Hysterisis Behavior

The existence of hysterisis type of behavior for the average gas holdup was found in our experiments in the 5.1 cm ID, 305 cm tall bubble column with a molten paraffin wax (FT-300) as the liquid medium (Quarterly Report, June-August, 1985). In these experiments it was found that there exists a range of superficial gas velocities within which for a given value of the superficial gas velocity two values of the average gas holdup are possible. The startup procedure determines which one of the two possible values will be attained. This type of behavior has not been observed in previous studies with a paraffin wax as the liquid medium, but similar behavior was found in some other gas-liquid systems and the relevant work will be briefly reviewed. The possibility of existence of two values of gas holdup for a given set of operating conditions has been predicted theoretically (e.g. Wallis, p. 92, 1969; Riquarts, 1979). Experimental evidence, however, for the existence of this type of behavior is still rather scarce.

A qualitative sketch of the observed gas holdup as a function of superficial gas velocity based on our experimental data and the data from

literature is shown in Figure 1. At low gas velocities, the bubbles are uniform in size and rise independently with fairly uniform spacing between them. This flow regime is known as the ideal bubbly or the homogeneous flow regime. At sufficiently high values of superficial gas velocities, there exists only one flow regime which has been referred to as the liquid circulation, heteregeneous flow, churn-turbulent, turbulent bubbling or The liquid circulation regime is characterized by a froth flow regime. non-uniform bubble size distribution. where virtually all the gas is transported as large, fast rising bubbles or bubble clusters. The small bubbles are entrained by local liquid circulation, and there is intensive turbulent mixing in the liquid. Between these two flow regimes there is a range of gas velocities where two values of gas holdup are possible. This is denoted as the transition region in Figure 1, and the upper branch is usually referred to as the "foaming regime" while the lower branch is part of the liquid circulation regime. The liquid curculation regime is a predominant flow regime with pure liquids. In the presence of surface active agents (e.g. contaminants, electrolytes, alcohols which surpress the bubble coalescence) the "foaming regime" has been observed by many investigators working with the air/water system (e.g. Zuber and Hench, 1962; Wallis, 1961; Freedman and Davidson, 1969; Anderson and Quinn, 1970; Zahradnik and Kastanek, 1979; Maruyama et al., 1981; Smith et al., 1983). The "foaming regime" is achieved with sintered metal plates distributors or the sieve plate distributors with small hole diameters, while the liquid circulation regime occurs with the sieve plate distributors having larger hole diameters (e.g. Zuber and Hench, 1962; Zahradnik and Kastanek, 1979; Pilhofer, 1980).

The existence of both types of flow regimes (i.e. the "foaming" and the liquid circulation) for a given set of operating conditions, has been reported only by Anderson and Quinn (1970) and Maruyama et al. (1981) for the system air/tap water. Anderson and Quinn found that the gas holdup increases with velocity up to $u_g = 7$ cm/s, at which point the level of the top surface dropped by 15 cm, i.e. the transition from the "foaming regime" to the "liquid circulation regime" (slug flow in their terminology) took place. Upon decreasing the velocity the lower curve in Figure 1 was traced all the way up to the ideal bubbly flow regime. Upon increasing the flow rate the original curve (i.e. the "foaming" regime) could not be reproduced, only the lower one. A possible explanation for this type of behavior was given in terms of the concentration gradient of a surface active impurities present in the tap water.

Maruyama et al. (Figure 4 of their paper) found that as the gas velocity increases the gas holdup follows the upper branch in Figure 1. Starting from the liquid circulation regime and decreasing the velocity the lower branch was traced all the way up to the ideal bubbly flow regime.

In our experiments with the FT-300 wax as the liquid medium the hysterisis behavior was observed under variety of conditions as described in the previous Quarterly Report (June-August, 1985). We found that transitions from the "foaming" regime to the liquid circulation regime (or the "nonfoamy" regime in our terminology), and from the "nonfoamy" to the "foamy" regime are possible. These transitions are denoted by broken lines with arrows in Figure 1. Also a startup procedure was established for achieving directly the liquid circulation regime in the transition region where the two flow regimes are possible.

2. Hydrodynamic Studies in the Small Glass Column

Additional experimental studies have been performed in 5.1 cm ID, 305 cm tall bubble column with FT-300 wax as the liquid. Three types of distributors were evaluated; two single orifice plate type distributors (1.85 mm and 4 mm) and the SMP with the average pore size of approximately 40 μ m. These experiments were undertaken with the following objectives: to study in more detail the transition region shown in Figure 1 and to determine whether the liquid circulation regime is the only stable flow regime at high velocities.

All experiments were conducted with the same batch of wax at 265°C and at atmospheric pressure using nitrogen as the gas. The same experimental procedure was employed as described in the last Quarterly Report, except that the nitrogen purge flow was not employed and that photographs of the flow field were taken during the experiments. The latter causes the temperature of the column to drop a few degrees, because the insulation has to be removed at locations where the photographs are taken.

2a. 1.85 mm Single Orifice Plate Distributor

Results obtained for the average gas holdup measurements, with the 1.85 mm single hole orifice plate distributor are shown in Figure 2. The liquid static height was 180 cm. New data are marked with circles, while the data obtained previously (June-August, 1985 Quarterly Report) are marked with triangles and are included for comparison. In the experiments with increasing gas velocity (open circles) the gas holdup increases rapidly to a value of 28.5% at $u_g = 4$ cm/s. Upon increasing the velocity to $u_g = 5$ cm/s, a transition from the "foamy" to the "nonfoamy" regime takes place which is accompanied by substantial decrease in the gas holdup

($\varepsilon_g = 13.6\%$ at $u_g = 5$ cm/s). After this the gas holdup is a monotone increasing function of the gas velocity, and it reaches a value of 25% at $u_g = 17$ cm/s. The same value of gas holdup in the "foamy" regime was obtained at $u_g = 3.5$ (cm/s).

In the next set of experiments the startup velocity of 9 cm/s was employed and the gas holdup measurements were made by increasing the superficial gas velocity in the order $u_g = 11, 13, 17 \text{ cm/s}$. The holdup increases with velocity (solid circles). The foam layer on the top of the liquid level was absent, but the holdup values obtained are somewhat higher than in the previous experiment (data points denoted with open circles). Upon decreasing the velocity the same curve is followed up to $u_g=9$ cm/s, but at lower gas velocities the holdup starts increasing and passes through a maximum at about $u_g = 6$ cm/s ($\varepsilon_g = 29\%$), and then decreases giving at u_g = 3 cm/s the same ε_{g} as in the experiment with the increasing velocity. This experiment was continued by increasing the velocity from 3 cm/s to 17 cm/s in finite increments, but the transition from the "foaming" regime to the liquid circulation regime did not take place. Results obtained earlier (Quarterly Report, June-August, 1985) are also shown for comparison with the new data. In the "foamy" regime there is good agreement between two sets of data up to $u_g = 4 \text{ cm/s}$. At higher values of u_g (i.e. $u_g = 5-9$ cm/s) the gas holdup values obtained earlier are significantly higher and the transition from the "foamy" to the "nonfoamy" regime did not occur. At u_{σ} = 11 cm/s essentially the same gas holdup was obtained in both sets of experiments. In the liquid circulation regime there is excellent agreement between these two sets of data.

This experiment demonstrates that transitions from one flow regime to

another are not completely reproducible. It appears that the experimental procedure (e.g. increasing or decreasing gas velocity) and the duration of experiments have effect on the stability of the two flow regimes, within the transition region, and on the gas holdup values in the "foamy" regime.

2b. 4 mm Single Orifice Plate Distributor

New results obtained with the 4 mm orifice plate distributor and $H_s = 190-215$ cm are shown in Figure 3 (points denoted by circles) together with the data obtained in the last Quarterly Report (points marked with triangles). In experiments with the low superficial gas velocity during the startup period ($u_g = 2$ cm/s at 265°C), and in the order of increasing velocity the holdup increases and passes through a local maximum (26.5% at $u_g = 4.5$ cm/s). As the velocity is increased further the gas holdup decreases and passes through a local minimum ($\varepsilon_g = 21\%$) at about 7 cm/s, and then increases again and reaches 33.3% at $u_g = 16$ cm/s. The height of foam layer at the top of the liquid level is as much as 40 cm at $u_g = 4$ cm/s, and then decreases as the velocity increases.

In the experiments with the startup velocity of 9 cm/s the foam layer was not observed and the gas holdup values (solid circles) are lower in comparison to the ones obtained in the experiments with the low startup velocity (open circles). The gas velocity was increased from 9 cm/s to 13 cm/s in increments of 2 cm/s, and then decreased back to 9 cm/s and the same value for the holdup was obtained. Upon further reduction of the gas velocity (up to 6 cm/s) the gas holdup was decreasing. The transition from the liquid circulation to the "foamy" regime occurred when the velocity was reduced to 5 cm/s. Then the velocity was reduced to 4 cm/s and the same value of the gas holdup was obtained as in the previous experiment with the

low startup velocity (i.e. $\varepsilon_g = 25\%$). After this the gas velocity was increased to 6 cm/s and the transition to the liquid circulation region occurred. The latter is not shown in Figure 3.

Comparison between the new and the previous results reveals the following. In the liquid circulation regime there is excellent agreement between the two sets of data. The only difference is in a value of critical velocity where the transition from the liquid circulation to the "foamy" regime takes place. On the other hand, in the "foamy" regime there are large differences in the gas holdup values for u_g in the range of (5 - 12) cm/s. This is largely caused by the amount of foam formed in these two sets of experiments. This again shows that different operating procedures, and/or the duration of experiments may give different values for ε_g in the "foamy" regime.

2c. Sintered Metal Plate Distributor

Results obtained with the SMP distributor with the average pore size of approximately 40 μ m and H_s = 60-150 cm are shown in Figure 4. The holdup initially increases very rapidly with the velocity ($\varepsilon_g = 70\%$ at 2 cm/s). Then it levels off ($\varepsilon_g = 75\%$ for u_g = 3-13 cm/s), and for u_g > 15 cm/s starts decreasing slowly. The breakup of foam did not occur even at u_g = 24 cm/s. These values are somewhat higher than the values reported in the last Quarterly Report (June-August, 1985) as shown in Figure 4. In experiments with the high startup velocity (u_g = 9 cm/s) much lower values of the gas holdup were obtained for u_g = (11-19) cm/s ($\varepsilon_g = 30-37\%$). When the velocity was decreased to 9 cm/s the foam began to develop giving $\varepsilon_g = 58.5\%$. Further decrease of velocity resulted in growth of the foam layer and thus to even higher holdups and at u_g = 7 cm/s the same gas holdup was

obtained as in the experiments conducted in the order of increasing velocity. The results obtained with the high startup velocity are in agreement with our previous data (solid triangles) except that the transition from the "nonfoamy" to the "foamy" regime occurred earlier this time.

3. Hydrodynamic Studies in the Large Glass Column

The average gas holdup measurements were made in the 22.5 cm ID, 300 cm tall glass column equipped with a perforated plate distributor having 19 holes of 1.85 mm in diameter equally spaced in a triangular pitch. The FT-300 wax was employed as the liquid medium and nitrogen as the gas. Experiments were performed at 200°C and 265°C with the static liquid height of 190 cm and the superficial gas velocities $u_g = (1-12)$ cm/s. Experimental procedure was the same as described in the Quarterly Report June-August, 1985. The differential pressure system was not operational and therefore the nitrogen purge flow was not employed.

Results of measurements are shown in Figure 5. Data obtained at 200°C are typical for the liquid circulation regime where the gas holdup is a monotone increasing function of velocity. In the experiments at 265°C, which were performed in the order of increasing velocity, the gas holdup increases rapidly between $u_g = 1$ cm/s and $u_g = 3$ cm/s. As the velocity was increased to 5 cm/s a small drop in the average gas holdup was observed, denoted by a broken line with the arrow, and after that the holdup continues to increase with the velocity. The observed behavior is characteristic for the "foaming" regime in Figure 1. When the velocity was decreased from 12 cm/s to 2 cm/s the holdup of 14.5% was obtained, which corresponds to the "foamy" regime. After increasing the velocity to 4 cm/s

the holdup of about 18% was obtained, indicating that this point might belong to the liquid circulation regime. A small decrease of velocity from 4 cm/s to 3.5 cm/s yields relatively large increase of the gas holdup ($\varepsilon_g =$ 22.5%), which is typical for transitions from the liquid circulation regime to the "foamy" regime.

These results, like the previous ones obtained in the 5.1 cm ID column, show that the temperature has strong effect on the average gas holdup. At lower temperatures it appears that the liquid circulation regime is the only stable flow regime while at higher temperatures the tendency towards foaming increases in this system. As stated previously (June-August, 1985 Quarterly Report) this is related to the effect of liquid viscosity on the bubble size distribution and the foam formation.

4. <u>Photographic Techniques for the Bubble Size Distribution Determination</u> and Mapping of Flow Regimes

During the gas holdup measurements in the glass columns (5.1 cm and 22.5 cm ID) photographs of the flow field were taken at three locations above the distributor with the still camera (Cannon AE1/P 35 mm SLR) and the video camera (Hitachi, Model GP-5AU). Video tapes have been reviewed and the following observations can be made:

<u>40 μ m SMP distributor</u>: For all the velocities, the resolution is not clear to identify the exact size of small bubbles nor to detect quantitatively changes in bubble size distribution with height from the distributor. At low superficial gas velocities (up to 2 cm/s) very fine bubbles (less than 1 mm in diameter) are found at all heights. This may be characterized as the ideal bubbly flow regime. At the velocity of 3 cm/s a few large bubbles (~ 2 cm in diameter) are seen at a distance 45 cm from the

distributor and slug type bubbles appeared at a distance 210 cm from the distributor. Initial size of these slug type bubbles is approximately 4 cm x 2 cm (length x width) and their frequency is about 0.2 s⁻¹. As the velocity increases the slug type bubbles appear at lower heights and their size and frequency increase to about 9 cm x 5 cm and 0.5 s⁻¹ respectively at 7 cm/s. The small bubbles are still present at all heights and this gives high gas holdups (70-75%). At 13 cm/s the slug frequency becomes approximately $1s^{-1}$.

1.85 mm single orifice plate distributor: At the velocity of 1 cm/s and 45 cm from the distributor there is a wide bubble size distribution: large bubbles (~ 2 cm in diameter); intermediate bubbles (0.8-1.5 cm), and small bubbles (0.4-0.8 cm). Some breakup of large and the intermediate bubbles occurs along the bed height and the density of small bubbles increases. As the velocity increases to 3 cm/s coalescence of large bubbles occurs and they are about 4 cm in diameter at height 120 cm from the distributor, becoming slugs at the height of 210 cm. The fraction of small bubbles (0.4-0.8 cm) increases with height, and at 210 cm fine bubbles (less than 1 mm) are also present. At 4 cm/s slugs are seen at distance 120 cm from the distributor already and their frequency is about 0.2 s⁻¹. At this velocity some fine bubbles (~ 1 mm) could be seen at a height 45 cm above the distributor. The density of these fine bubbles increases with height and the foam layer grows on the top. As the velocity increases the swirling flow patterns are observed. The wide bubble size distribution exists at the distance 45 cm above the distributor, but as the bed height increases only two groups of bubbles can be distinguished: slugs or slug type bubbles and fine bubbles. The slugs increase in size (i.e. their length

increases) and their frequency also increases. At 11 cm/s the slug size is about 10 cm x 5 cm, and their frequency 1 s⁻¹.

4 mm single orifice plate distributor: The observed behavior is qualitatively similar to the one found with the 1.85 mm orifice plate distributor. The flow regime may be characterized as the ideal bubbly up to 2 cm/s. The large bubbles near the distributor (45 cm) are about 2-3 cm in diameter, and small bubbles are in the range of 0.2-0.8 cm. As the height increases the bubble coalescence and the breakup occur and the two groups of bubbles become dominant, i.e. large bubbles and fine bubbles. At the velocity of 3 cm/s the large bubbles of about 4 cm in width appear at a height 120 cm above the distributor, and at 4 cm/s the tendency toward slugging increases (bell shaped large bubbles). These large bubbles are surrounded by a large number of fine bubbles less than 1 mm in diameter. The frequency of slug type bubbles is about 0.15 s^{-1} at 5 cm/s and increases to 0.5 s⁻¹ at $u_g = 8$ cm/s. At higher gas velocities ($u_g = 9-14$ cm/s) the picture resolution is not so good, but the flow pattern may be characterized as highly turbulent with intensive liquid mixing. The small bubbles near the wall are sometimes carried downwards and are in swirling motion. Large bubbles appear to be rising in groups which could not be characterized as slugs.

Perforated Plate Distributor (22.5 cm ID column; $T = 265 \circ C$): At $u_g = 1$ cm/s and 30 cm above the distributor small bubbles (0.1-0.5 cm) and fine bubbles (less than 1 mm) are the predominant ones. A few bubbles of 1-2 cm in diameter are also seen occasionally. At the column heights of 105 cm and 180 cm there is an increase in the fraction of fine bubbles, and a decrease in the number of small bubbles, while the number of large bubbles

(1-2 cm) remains small. As the velocity increases to 3 cm/s the density of fine bubbles increases at all heights while the fraction of small bubbles decreases and large bubbles have not been observed. At 5 cm/s a few large bubbles 2-3 cm are seen near the distributor, but not in the upper part of the column where the fine and the small bubbles are present. As the velocity increases (7 cm/s and 9 cm/s) these large bubbles are observed at all heights and their frequency increases. The fraction of fine bubbles also appears to be increasing and the swirling patterns are observed at all heights. Bubbles greater than approximately 3 cm have not been observed at any height. The flow regime at 1 cm/s may be characterized as the ideal bubbly and at higher velocities as the churn turbulent.

5. Future Work

The following activities are planned for the next quarter:

(a) Begin analysis of photographs of the flow field to determine the bubble size distribution using an image analyzer.

(b) Continue experiments in the large glass column with the multiple hole orifice plate distributor.

(c) Design and manufacture a perforated pipe distributor (manifold) and study the effect of operating conditions on the average gas holdup in the large glass column.

(d) Continue to work on a differential pressure method for axial gas holdup measurements.

D. Task 4 - Correlation Development and Data Reduction

No work on this Task has been planned for this quarter.

- V. Nomenclature
- d_o orifice hole diameter, (mm)
- H_s static liquid height, (cm)
- T column temperature, (°C)

u superficial gas velocity at operating conditions, (cm/s)

Greek Letters

 ε_{g} average gas holdup, (-)

Acronyms

- BC bubble columns
- DOE Department of Energy
- FT Fischer-Tropsch
- ID inside diameter
- SMP sintered metal plate
- TAMU Texas A&M University

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Superficial Gas Velocity

Figure 1. A qualitative sketch of average gas holdup dependence on superficial gas velocity.



Figure 2. Effect of superficial gas velocity on gas holdup in 5.1 cm ID, 305 cm tall column (open symbols: low startup velocity; solid symbols: high startup velocity; Δ/Δ - previous data; o/ \bullet - new data).



Figure 3. Effect of superficial gas velocity on gas holdup in 5.1 cm ID, 305 cm tall column (open symbols: low startup velocity; solid symbols: high startup velocity; Δ/\blacktriangle - previous data; o/• - new data).



Figure 4. Effect of superficial gas velocity on gas holdup in 5.1 cm ID, 305 cm tall column (open symbols: low startup velocity; solid symbols: high startup velocity; Δ/Δ - previous data; o/• - new data).

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Figure 5. Effect of superficial gas velocity on gas holdup in 22.5 cm ID, 300 cm tall column.