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GAS/SLURRY FLOW IN COAL LIQUEFACTION PROCESSES (FLUID DYNAMICS IN 3-PHASE FLOW COLUMNS)

Quarterly Technical Progress Report for the Period April 1-June 30, 1980

By
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R. Sivasubramanian
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August 1980

Work Performed Under Contract No. AC01-79ET14801

Air Products and Chemicals, Inc.
Allentown, Pennsylvania

U. S. DEPARTMENT OF ENERGY

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SUMMARY

This is the third quarterly report under Contract Number DE-AC01-79ET14801 titled "Gas/Slurry Flow in Coal Liquefaction Processes". This work covers the period 1 April 1980 to 30 June 1980. This work is a continuation of studies initiated by Air Products and Chemicals, Inc., on the fluid dynamics of 3-phase flow to support the design of the 6000 T/D dissolver for the SRC-I demonstration plant which began in July 1978. DOE supported these 3-phase flow studies under the Bridging Task program from 1 July 1979 to 30 September 1979 at the start of the current contract. A background of information developed at Air Products prior to DOE support was included in the first quarterly report.

The 6000 T/D SRC-I demonstration plant will employ verticle tubular reactors feeding slurry and gas concurrently upward through these vessels. In the SRC-I design this reactor is essentially an empty vessel with only a distributor plate located near the inlet. Because the commercial plant represents a considerable scale-up over either Wilsonville or Ft. Lewis, this program is addressing the need for additional data on behavior of three phase systems in large vessels. Parameters being investigated in this program are being studied at conditions that relate directly to the projected demonstration plant operating conditions.

Gas velocity is the key element to maintain solid particles in suspension. Above the critical gas velocity, which is defined as the minimum gas velocity to achieve full solids suspension, solid accumulation is practically independent of gas velocity. However, solid accumulation was found to increase linearly with decreasing liquid velocity at a fixed gas input rate. The effect of liquid and gas velocities on solids accumulation was studied in a 5-inch diameter column using 60/80 mesh sand particles. The superficial gas and liquid velocities were varied from 0.05 to 0.33 ft/sec and from 0.01 to 0.05 ft/sec, respectively, covering the flow conditions designed for the SRC-I demonstration plant. At a half slurry turndown velocity, the accumulation of the fines (140 mesh minus) did not increase substantially but the amount of large particles (60/80 mesh) increased three-fold. Of more concern is the settling of the larger particles which have critical gas velocities higher than the half gas turndown velocity.

The mass transfer coefficient, $k_L a$, is reduced by 30% in the presence of solids at all gas velocities. Batch and continuous experiments were conducted in this quarter to study oxygen transfer in air/water and air/water/sand systems. Data analysis of the steady state measurements (continuous experiments) is not complete and will be presented in the next quarterly report. Results from the 5-inch diameter column show that $k_L a$ increases with gas velocity in both 2-phase and 3-phase systems. However, $k_L a$ is lower by 30% in the presence of solids at all gas velocities. The results also indicate that the $k_L a$ is independent of column height for the 5-inch diameter column.

The effect of the absence of a distributor on fluid dynamics could not be clearly determined because of an unusual liquid level surge behavior. Gas holdup, liquid dispersion, and solid accumulation measurements were made in the 12-inch diameter column in the absence of a distributor. Unusual flow patterns were observed; the liquid level at the top of the column periodically surged such that the liquid level would rise above the exit line and then suddenly drop a few inches below the exit line. Periodic gas slugs were visually observed accompanying this liquid level surge and were suspected to be the major cause of the surging behavior.

The absence of a distributor has no effect on gas holdup. Results from liquid dispersion experiments confirm the earlier conclusion that liquid velocity has no effect on axial dispersion coefficients. However, due to the scatter in the data resulting from surging in the absence of a distributor, the effect of solids on axial dispersion coefficients in the absence of a distributor cannot be determined. The accumulation of fine particles was slightly reduced in the absence of a distributor plate. Again, this slight reduction is not definitive particularly with the unusual surging behavior with no distributor. Further experiments using a different distributor plate may shed some insight into the entrance effects on fluid dynamics of the columns.

A new distributor plate was designed and fabricated in this quarter. Experimental results using the new distributor plate will be presented in the next quarterly report.

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1.0 OBJECTIVE

The overall objective of this project is to study the solids accumulation and suspension of various gas/liquid/solid systems in cold-flow tubular columns aimed at providing data for the coal dissolver design in the SRC-I demonstration plant.

The specific objectives are:

1. To check the adequacy of the existing experimental apparatus using a two-phase system (air/water mixture).
2. To study the effects of slurry and gas velocities, solid particle size and concentrations, and liquid viscosity and surface tension on the performance of a cold-flow tubular column.
3. To develop an effective slurry withdrawal technique from the bottom of a tubular column as a means to control the solid concentration in the column.
4. To study the performance of cold-flow tubular column with an improved distributor and in the absence of a distributor.
5. To explore the use of multiple distributors in a tubular column.

2.0 INTRODUCTION

A major element of the coal dissolution section of any liquefaction plant is the dissolver. Although a considerable amount of liquefaction will occur in the preheater, a major amount of necessary chemical change will occur in the dissolver, namely sulfur removal, oil and distillate formation and solvent rehydrogenation.

Vertical tubular reactors are employed in all of the major processes currently under consideration for commercial liquefaction of coal. In all of these processes, SRC, EDS and H-Coal, slurry and gas are concurrently fed upward through these vessels. In the EDS and SRC processes, the reactors are essentially empty vessels, whereas for the H-Coal process a bed of ebullating catalyst is maintained in the reactor. The major differences between the EDS, SRC-I and SRC-II processes in dissolver

operation are the composition of the feed streams and reactants within the dissolver. Other hardware differences such as distributor plates, draft tubes or recycle loops can also cause differences in the behavior of slurries in these vessels. A requirement necessary to any design that will be technically feasible and cost effective is an understanding of the physical behavior of three phase systems in tubular columns.

All of the major processes under development require understanding of backmixed three phase systems. Each process employs at least a portion of its dissolver volume in a backmixed mode. As the design of the 6000 T/D SRC-I plant progresses, the increased vessel size (and other considerations) may dictate the use of reactors in series, which would decrease the overall backmixed characteristic of the commercial plant.

The SRC-I demonstration plant dissolver will represent a considerable scale-up over the Wilsonville and Ft. Lewis dissolver. To intelligently make good design decisions, more information is needed on the flow properties of three phase systems in large vessels. More important from the standpoint of slurry behavior is the difference in gas and liquid superficial velocities. This difference can have considerable impact on the process because the gas and liquid superficial velocities have a strong effect on (a) gas void volume (b) actual solids concentration in the dissolver and (c) the relative degree of backmixing. As velocity through the dissolver increases, the tendency for solids to remain behind diminishes, causing a decrease in the actual concentration of ash particles in the reactor. Those particles that do remain will tend to be larger in size. Since considerable evidence points to a definite catalytic effect of the reactor solids, these larger particles will have decreased surface areas exposed and will likely have diminished catalytic activity. Knowing the particle sizes that can accumulate under commercial flow conditions will give us some indication of size of dissolver solids that should be examined for catalytic activity.

Considerable work on the behavior of gas/liquid mixtures flowing through vertical columns has been reported in the literature. Information on three phase (gas/liquid/solid) systems is far less extensive. Detailed

background information was presented in the first quarterly report (1). Under this contract, work is being conducted in a 5-inch diameter by 5-foot tall Plexiglass column and a 12-inch diameter by 25-foot tall glass column which are located at the contractor's site. The physical dimensions, auxiliary equipment, and some of the experimental techniques employed in this study were extensively discussed in the first quarterly report. This report contains experimental results from runs conducted during this reporting period (1 April-30 June 1980).

3.0 TECHNICAL PROGRESS

3.1 Task 2 - Two and Three Phase Flow in Vertical Columns

The objectives of this task were:

- ° to study gas holdup and liquid dispersion in two and three phase systems
- ° to study gas/liquid mass transfer
- ° to study solid dispersion

The gas holdup and liquid dispersion studies were completed in the last quarter along with some experiments on solid dispersion. Gas/liquid mass transfer studies were completed during this quarter. Feed tank modifications were completed during this quarter and this enabled us to keep the 60/80 mesh particles in suspension in the feed tank. The effect of liquid and gas velocities on the distribution of 60/80 mesh particles in the 5" diameter column were also studied during this quarter.

3.2 Task 3 - Effect of Distributor on Flow

The objectives of this task are:

- ° to study the entrance effects on the performance of the 12-inch column

- to study the performance of the column in the absence of a distributor
- to design, fabricate and install a new distributor and to compare the performance of the different distributors

During this quarter gas holdup and liquid dispersion experiments were completed in the 12" column without a distributor. One experiment was conducted to compare the effect of distributor on the axial concentration of -140 mesh solids in the 12" column. A new distributor plate has been designed and fabricated. Experimental results using the new distributor will be presented in the next quarter.

4.0 EXPERIMENTAL SECTION

4.1 Cold Flow Model Equipment

Both the 5-inch diameter and 12-inch diameter columns used in these cold-flow studies, were described in detail in the first quarterly report (1). During this reporting period, modifications to the feed tanks to provide uniform feed concentration of large particles (60/80 mesh) were completed. These modifications include installing new axial flow turbine-type agitators and baffles in the feed tanks.

4.2 Experimental Procedures

4.2.1 Liquid Dispersion

Liquid phase dispersion in an air/water/sand system was investigated in a 12-inch diameter column using a tracer detection method. Sodium chloride was used as the tracer and a conductivity probe mounted at the exit line of the column monitored the ion conductivity of the solution continuously. Detailed experimental procedures can be found in the first quarterly report (1). During this reporting period, two particle sizes (20/30 mesh and 140 mesh minue) were studied in the absence of distributor plate.

4.2.2 Solids Dispersion

The axial distribution of solids were studied using both 5-inch and 12-inch diameter columns. The effect of liquid and gas velocities on the axial distribution of 60/80 mesh solid particles in the 5-inch diameter column were also studied. The axial distribution of 140 mesh minus solids at a single operating condition ($V_L = 0.05$ ft/sec, $V_g = 0.33$ ft/sec) was studied in the 12" column in the absence of a distributor. All experiments were conducted in a recycle mode which was described in the second quarterly report. In brief, the exit from the column was returned to the feed tank thereby creating a closed loop. The recycle loop was allowed to operate for several hours in order to achieve steady state. Solid concentration profile was measured from slurry samples withdrawn from sampling ports located at equal distance along the axis of the columns.

4.2.3 Gas/Liquid Mass Transfer

Gas/liquid mass transfer studies were conducted in the 5-inch and 12-inch diameter columns both in the absence and presence of liquid flow. The gas/liquid mass transfer rate was measured by the rate of oxygen dissolving in water. A polarographic probe was used to continuously monitor the concentration of dissolved oxygen in the solution with time. Both stationary and submersible probes were used in these experiments. The experimental procedure described below is essentially identical for both columns.

4.2.3.1 Batch Experiments

The oxygen content of water in the 5-inch diameter column for the batch experiments was measured with stationary probes installed at various heights of the column. For the 12-inch diameter column, submersible probes were used because column structure prohibits installing stationary probes. Several measurements

were made in the 5-inch diameter column at various heights using both types of probes. The negligible difference in the measurements ensures the validity of using the submersible probes.

Nitrogen was allowed to flow through the column filled with water at the beginning to remove the oxygen content. After the dissolver oxygen concentration leveled out at zero value for several minutes, the nitrogen flow was stopped and air was introduced at a pre-determined rate. The change in the dissolver oxygen concentration as a function of time at various levels in the column were recorded. These measurements were made at three different positions for the 5-inch diameter column and at 2 different positions for the 12-inch diameter column. The batch experiments were conducted for gas velocities ranging from 0.05 to 0.43 ft/sec. Methods of data analysis will be presented in the results and discussion section.

4.2.3.2 Continuous Experiments

Steady state oxygen concentrations were measured in the 5-inch and 12-inch diameter columns as a function of liquid and gas velocities. The liquid velocities ranged from 0.01 to 0.05 ft/sec and the gas velocities ranged from 0.05 to 0.43 ft/sec.

Deoxygenated water was pumped into the columns at a constant velocity. For experiments in the 5-inch diameter column, the 12-inch diameter column was used as a storage vessel of the deoxygenated water by purging with nitrogen continuously. For experiments in the 12-inch diameter column, deoxygenated water was stored in regular feed tanks which were equipped with special ring spargers through which nitrogen was purged continuously. The oxygen level of the water entering the column was monitored using submersible probes to ensure constant zero level. Then air was introduced at pre-determined rate. After attaining steady state, the dissolver oxygen concentrations at various heights of the column were measured by changing the position of the submersible

probes. In the case of the 5-inch diameter column, steady state measurements were also taken using the stationary probes. The equilibrium oxygen concentration C^* was also experimentally measured by using the submersible probes. The method of data analysis is presented in the results and discussion section.

5.0 RESULTS AND DISCUSSION

5.1 Axial Solids Distribution

The effect of liquid and gas velocities on the axial distribution of solids were investigated in a series of experiments conducted during this quarter which are listed in Table I. In these experiments as sand/ water slurry was fed to the column continuously, solids concentrations were measured at various heights in the column every 30 minutes over a period of 6 hours. Experimental data from a typical run are shown in Figure 1. Data were not taken in the first two hours of the experiment because previous experience had shown that steady state could be achieved in about 2 hours which is shown in the results in Figure 1. These data are similar to results obtained in other experiments. Actual steady state concentrations at each level of the column were determined by averaging the corresponding experimental values in any particular experiment.

From these steady state concentrations taken at each level, the amount of solids retained in the column could be calculated by integrating the experimentally determined solids distribution profile over the entire column. A computer program was developed to fit the data with a polynomial function, and to compute the area under the fitted curve in a solid concentration versus column height plot. The amount of retained solid equals to the product of this computer-generated value and the cross-sectional area of the column. For example, using data from the 60/80 mesh solids experiments in the 5-inch column, the amount of solids retained in the column were determined as shown in Table II. Since it was possible to remove the solids from the column and get an actual experimentally determined dried weight, a comparison could be made between calculated and experimentally determined values for the amount of

Table I
List of Axial Solids Distribution Experiments

Column Diameter	Particle Size	Liquid Velocity	Gas Velocity	Feed Solids Concentration
<u>inches</u>	<u>Mesh</u>	<u>ft/sec</u>	<u>ft/sec</u>	<u>lbs/ft³</u>
5	60/80	0.05	0.327	3.3
5	60/80	0.05	0.194	2.6
5	60/80	0.05	0.05	2.4
5	60/80	0.03	0.327	2.4
5	60/80	0.01	0.327	2.1
12	-140	0.05	0.327	2.9

FIGURE 1 - TYPICAL EXPERIMENTAL DATA FOR AXIAL SOLIDS DISTRIBUTION

Table II
Experimental Data - Solids Distribution
Particle Size - 60/80 Mesh
Column Diameter - 5"

V_L ft/sec	V_g ft/sec	Solids Concentration, lbs/ft ³					Solids Retained, lbs	
		<u>L=0.327 ft</u>	<u>L=1.473 ft</u>	<u>L=2.577 ft</u>	<u>L=3.66 ft</u>	<u>L=4.910 ft</u>	<u>Exptl</u>	<u>Calculated</u>
0.05	0.327	13.87	11.27	8.36	5.98	3.3	6	5.94
0.05	0.194	40.38	30.55	19.08	9.77	2.6	15.5	14.47
0.05	0.05	43.71	37.09	25.40	10.06	2.36	14.6	16.75
0.03	0.327	41.79	31.28	19.06	10.07	2.44	14.5	14.81
0.01	0.327	53.43	42.74	27.24	13.84	2.05	19.0	19.70

solids retained in the column. The comparisons (Table II) agree indicating that our experimental techniques for determining solids concentrations are valid.

Liquid velocity has a dramatic effect on the solids concentration profile in the column. The concentration of solids in the column increases as liquid velocity decreases as shown for 60/80 mesh particles in the 5-inch diameter column in Figure 2. This change is more pronounced toward the bottom of the column. These trends were observed in earlier experiments using 140 mesh minus material (1).

Since solids accumulation is very sensitive to the solids concentration in the inlet stream, comparisons between different runs must compensate for any variation in the feed stream. The exit concentrations, which should be identical to feed concentrations at steady-state conditions, varied slightly for the comparison shown in Figure 2. This slight variation in exit concentration was due to the experimental technique employed in this study. For all experiments a known amount of sand (50 lbs) was suspended in 100 gallons of water and this slurry was circulated through the column until steady state was achieved. Hence, the steady state feed concentration would be higher for those experiments with lower solids accumulation in the column. However, the direction of variation in fact supports the effect of liquid velocity discussed above. It is logical to believe that solid accumulation would increase with increasing feed concentration provided that other conditions are held constant. The lowest liquid flow rate having the least feed concentration had the highest retained solid concentration which strongly supported the results of increasing solid accumulation with decreasing liquid velocity.

Under the conditions tested in this experimental program a linear relationship exists between the liquid velocity and solids accumulation. Such a relationship for 60/80 mesh particles in the 5-inch column is shown in Figure 3. These experiments were conducted at gas velocities above the velocity at which particles will settle in the column, i.e., the critical gas velocity.

FIGURE 2 - EFFECT OF LIQUID VELOCITY ON SOLIDS DISTRIBUTION

COLUMN DIAMETER = 5 INCH

PARTICLE SIZE = 60/80 MESH

GAS VELOCITY = 0.327 FT/SEC

**FIGURE 3 - EFFECT OF LIQUID VELOCITY
ON SOLIDS ACCUMULATION**

Although we do not currently have data on the 60/80 mesh particles in the 12-inch column to confirm this relationship on scale-up, we have seen, as reported in the last quarterly report, such a relationship for -140 mesh particles (Figure 4). A linear relationship between solids accumulation and liquid velocity was observed.

The dramatic effect of particle size on solids accumulation can be seen by comparing Figures 3 and 4. The slope of the larger 60/80 mesh particles is 27 times higher than for the -140 mesh material.

Therefore, for a small reduction in liquid velocity much larger particle size material would accumulate in the column than the fine particles. As an illustration, a reduction in liquid velocity from 0.05 ft/sec to 0.04 ft/sec (a 20% reduction) could increase the solid accumulation by about 8.5% for the -140 mesh particles, whereas solid accumulation for the 60/80 mesh sand could increase by 100%. We are cautious in interpreting the data in Figures 3 and 4 since they were taken from different column configurations, therefore the comparison is on a qualitative basis. Results from 60/80 mesh experiments in the 12-inch diameter column are needed to correlate the effect of liquid velocity on solids accumulation.

Gas velocity also has an effect on solids accumulation if the velocity is less than the critical gas velocity. Earlier we had shown that for fine particles (-140 mesh) changes in the gas velocity did not significantly affect the solids accumulation or concentration profiles (2). All the velocities with the -140 mesh solids were above the critical gas velocities for these solids.

Recent data on 60/80 mesh particles in the 5-inch diameter column show a sharp decrease in solid concentration through the column (Figure 5). Although increasing gas velocity from 0.05 to .194 ft/sec did not change the solids accumulation, increasing up to .327 ft/sec resulted in a dramatic decrease. Since the critical gas velocity for 60/80 mesh particles ranges between 0.193 and 0.217 ft/sec, at the higher 0.327 ft/sec velocity no settled solids will be present in the column thereby sharply decreasing the solids accumulation in the column.

**FIGURE 4 - EFFECT OF LIQUID VELOCITY
ON SOLIDS ACCUMULATION**

**FIGURE 5 - EFFECT OF GAS VELOCITY
ON SOLIDS DISTRIBUTION**

COLUMN DIAMETER = 5 INCH

PARTICLE SIZE = 60/80 MESH

LIQUID VELOCITY = 0.05 FT/SEC

Therefore, the data on 60/80 mesh particles agree with the conclusion on the smaller particles under the limitation that the flow conditions be above the critical gas velocity. The data at zero liquid velocity (batch experiments) in the previous quarterly report (1) indicated that the dispersion of the large particles behaved similarly to fine particles above the critical gas velocity. Additional experiments using gas velocities between 0.194 and 0.327 ft/sec and some experiments beyond 0.327 ft/sec, need to be performed to clarify the effect of gas velocity. Qualitatively, one can say that these experimental results agree with earlier results from batch experiments.

Solids accumulation increases dramatically with increasing particle size. Comparison of steady-state solids concentration profiles for 60/80 mesh sand and 140 mesh minus sand in a 5-inch diameter column is shown in Figure 6. As the flow conditions exceeded the critical gas velocities, both particles were in full suspension thereby providing a justified comparison to evaluate particle size effect on solids accumulation. The larger 60/80 mesh particles show a very drastic increase in concentration from the top to the bottom of the column whereas the fines are fairly homogeneously distributed. Since the area under the data curves represents accumulation inside the column, the results shown in Figure 6 clearly indicate that solids accumulate much more with larger particle size.

The hydrodynamic effect in the absence of a distributor was evaluated using small particle solids. For fine particles at low concentrations the solids concentration decreases almost linearly with column height as shown in Figure 7 for the 12-inch diameter column for both cases (presence or absence of a distributor). The differences in solid concentrations between the top and bottom of the column are fairly small in both cases. The normalized solid concentrations, plotted as a function of height in Figure 8, show that solid accumulation increases slightly in the presence of a distributor. However, an unusual flow behavior observed in the absence of a distributor (which will be discussed later) prohibits a fair comparison. Reservation should be made to

**FIGURE 6 - EFFECT OF PARTICLE
SIZE ON SOLIDS DISTRIBUTION**

**FIGURE 7 - EFFECT OF DISTRIBUTOR
ON SOLIDS DISTRIBUTION**

**FIGURE 8 - EFFECT OF DISTRIBUTOR ON
NORMALIZED SOLIDS DISTRIBUTION**

interpret this slight difference. Later data will be taken on solids distribution using a distributor without bubble caps and the results will be compared with those using the distributor with bubble caps.

5.2 Gas/Liquid Mass Transfer

Batch and continuous experiments in both 5-inch and 12-inch diameter columns were conducted in this quarter to study oxygen transfer from air to water in air/water and air/water/sand systems. The mass transfer coefficient, $k_L a$, in a bubble column is normally calculated by one of the following three methods: a) by assuming complete mixing in the liquid phase, b) by assuming plug flow for liquid phase, or c) by assuming dispersed plug flow for the liquid phase. Since the flow pattern in a bubble column is very close to mixed flow but not completely backmixed, assuming dispersed plug flow for liquid phase is more accurate to correlate the data. However, this method involves a complicated expression. The data analysis of the steady state measurements (continuous experiments) is not complete yet, and will be presented in the next quarterly report.

The batch experimental results were analyzed on the simple assumption of complete liquid mixing, which is reasonable for the batch experiments. Values of $k_L a$ were calculated using the following equation:

$$k_L a = \frac{1 - \epsilon_G}{t} \ln \frac{C^* - C_i}{C^* - C_f}$$

where $k_L a$ = volumetric liquid phase mass transfer coefficient, $(\text{sec})^{-1}$

t = time, seconds

ϵ_G = gas holdup

C^* = equilibrium oxygen concentration, mg/l

C_i = initial concentration of dissolved oxygen, mg/l

C_f = final concentration of dissolved oxygen, mg/l

The mass transfer coefficient, $k_L a$, is found to be independent of height for the 5-inch diameter by 5 feet tall column. The bars on the data points shown in Figure 9 indicate the variations of $k_L a$ with height in the column at that particular gas velocity. The insignificant variation supports the independence of $k_L a$ on column height. Results from the 12-inch diameter column, which is five times taller than the 5-inch diameter column, should further clarify this finding.

The dependence of liquid-phase mass transfer coefficient ($k_L a$) on the gas velocity can be described fairly well by a straight line in a long-log plot as demonstrated in Figure 9. This trend and the magnitude of the values obtained for $k_L a$ agree well with other investigators (2, 3).

The mass transfer coefficient, $k_L a$, is lower in the presence of solids by approximately 30% at all gas velocities as shown in Figure 9. The decrease in $k_L a$ in the presence of solids could possibly be due to a reduction in interfacial area in the presence of solids. Other investigations indicated that the presence of solids enhanced bubble coalescence, thereby forming larger bubbles. For same gas volume, the interfacial area decreased with increasing bubble size which could explain the decrease in $k_L a$ value. Results from 12-inch diameter column experiments are awaited before a detailed discussion can be presented.

5.3 Gas Holdup

Gas holdup measurements in the 12-inch diameter column in the absence of a distributor were made both in the presence and absence of liquid flow and in the presence and absence of solids. Gas velocities varied from 0.05 to 0.43 ft/sec and liquid velocities ranged from 0.01 to 0.05 ft/sec. Two different particle sizes of solids, 20/30 mesh and -140 mesh, were used. In the case of the larger particle size (20/30 mesh), the sand was placed in the column and liquid and gas were allowed to flow through the column. However in the case of the fine particles (-140 mesh), a sand/water slurry was pumped into the column.

**FIGURE 9 - EFFECT OF SOLIDS ON MASS
TRANSFER COEFFICIENTS**

Unusual behavior was observed in the absence of a distributor when both liquid and gas flowed through the column. The liquid level at the top of the column periodically surged such that the liquid level would rise above the exit line and then suddenly drop a few inches below the exit line. This phenomena occurred at gas and liquid velocities above 0.2 ft/sec and 0.01 ft/sec, respectively. Because of this surging behavior, the holdup data at high gas velocities in the absence of a distributor may have a substantial error. Periodic gas slugs were visually observed in the column in the absence of the distributor and were suspected to be the major cause of the surging behavior.

Experimental gas holdup data from the 12-inch diameter column in the absence of a distributor indicate that at low gas velocities gas holdup is independent of liquid velocity, the presence or absence of solids, and the particular solids particle size as shown in the first row of Table III. The overall data also indicate that the gas holdup is independent of liquid velocity at all gas velocities. The only appreciable change in gas holdup with liquid velocity (in the absence of solids) is shown at very high gas and liquid velocities which were the conditions under which surging occurred. However, data taken in the presence of large 20/30 mesh solids clearly indicate that liquid velocity has no effect on gas holdup. A comparison of the data from the two different particle sizes also indicate that gas holdup is essentially independent of the particle size used at low concentrations (5 lbs/ft^3) of solids.

Gas holdup data from the 12-inch diameter column with and without the distributor plates shown in Figure 10 show that gas holdup does not change appreciably when the distributor plate is removed indicating that the large bubbles observed in the absence of the distributor probably occupied a very small fraction of the gas void volume. Data were obtained both without solids and in the presence of 20/30 mesh solids. These experimental results without the distributor confirm two important points:

- a) that large slugs are formed in the absence of a distributor that may result in unusual flow patterns in the column but

Table III

Gas Holdup Measurements from 12" Column in the Absence of a Distributor Plate

Gas Velocity, ft/sec	Holdup										
	In the absence of solids				In the presence of solids						
					Particle Size = 20/30 Mesh				Particle Size = -140 Mesh		
	$V_L=0.0$ ft/sec	$V_L=0.01$	$V_L=0.03$	$V_L=0.05$	$V_L=0.0$	$V_L=0.01$	$V_L=0.03$	$V_L=0.05$	$V_L=0.0$	$V_L=0.0$	
0.05	0.041	0.040	0.044	0.046	0.038	0.037	0.037	0.042	0.040	0.040	0.047
0.10	0.088	0.086	0.100	0.097							
0.15	0.123				0.109	0.104	0.105	0.113	0.102	0.111	0.131
0.20	0.138	0.131	0.162	0.174							
0.25	0.141				0.142	0.133	0.132	0.138	0.127	0.135	0.159
0.28	0.146										
0.33	0.149	0.152	0.178	0.178	0.163	0.152	0.153	0.154	0.146	0.152	0.167
0.37	0.153	0.162	0.184	0.162							
0.43	0.156	0.172	0.188	0.154	0.179	0.166	0.169	0.173	0.164	0.170	0.182

FIGURE 10 - EFFECT OF DISTRIBUTOR PLATE ON GAS HOLDUP

b) gas holdup does not change appreciably.

Gas holdup data alone cannot sufficiently reflect the size of the bubbles present in the column. We are considering some photographic method which could provide an indication of the fraction of large bubbles present in the column. This will be discussed in the next quarterly report.

5.4 Liquid Dispersion

Liquid dispersion experiments in the absence of a distributor plate were limited due to the surging behavior of the column. In the absence of the distributor, the electronic noise in the experimental tracer curve increases at higher gas and liquid velocities reducing data accuracy. Results from these experiments are presented in this section. However, detailed discussion on the entrance effects on axial liquid dispersion coefficients will be presented after data using the new distributor plate become available.

The experimental conditions for the liquid dispersion runs conducted in this quarter are listed in Table IV. The fits of all the experimental tracer curves with the theoretical curves from the axial dispersion model are displayed in Appendix A. In these curves, the circles represent the experimental tracer curve while the solid line represents the theoretical curve obtained from the axial dispersion model. Detailed description of the procedure can be found in the first quarterly report (1).

Liquid dispersion data can be reproduced reasonably well. The dispersion numbers and axial dispersion coefficients for all the experiments conducted in this quarter are listed in Table V. The 20/30 mesh experiments were conducted by placing a known amount of sand in the column and passing liquid and gas through the column. The -140 mesh experiments were carried out by pumping a sand/water slurry through the column. As pointed out earlier the surging occurred at very high liquid and gas velocities but the presence of solids diminished the surging. Experimental runs XX1-1 and XX1-1R were conducted essentially at the same operating

Table IV
Experimental Conditions for Liquid Dispersion Runs

Liquid Velocity - 0.01-0.05 ft/sec

Gas Velocity - 0.0-0.327 ft/sec

Particle Size - 20/30 mesh, 140 mesh minus

Reactor Solids Concentration - 5 lbs/ft³

Distributor Used - None

Column Diameter - 12"

Table V
Data from Liquid Dispersion Experiments
in the Absence of Distributor Plate

Run No.	V_L ft/sec	V_g ft/sec	P_s Mesh	C_s lbs/ft ³	$\frac{D}{LV_L}$	$\frac{D}{ft^2/sec}$
XX-1	0.046	0.327	---	---	0.80	0.928
XX-2	0.053	0.194	---	---	0.56	0.749
XX-3	0.050	0.050	---	---	0.18	0.227
XX-4	0.051	0.100	---	---	0.25	0.324
XX-5	0.048	0.0	---	---	very low	---
XX-6	0.041	0.327	---	---	0.50	0.513
XX-7	0.015	0.327	---	---	1.30	0.475
XX1-1	0.049	0.194	20/30	5.0	0.38	0.472
XX1-1R	0.053	0.194	20/30	5.0	0.33	0.437
XX1-2	0.027	0.327	20/30	5.0	0.62	0.418
XX1-3	0.055	0.050	20/30	5.0	0.25	0.348
XX1-4	0.021	0.194	20/30	5.0	0.88	0.466
XX11-1	0.053	0.194	-140	5.0	0.30	0.404
XX11-2	0.054	0.05	-140	5.0	0.15	0.204
X11-3	0.024	0.327	-140	5.0	0.62	0.369
X11-4	0.023	0.194	-140	5.0	0.78	0.452

V_L - liquid velocity

V_g - gas velocity

P_s - particle size

C_s - solids concentration

L - heights of column

D - axial dispersion coefficient, ft²/sec

conditions to check the reproducibility of the experiments. Our results indicate that our reproducibility is quite reasonable under the experimental conditions. The first two experiments shown in Table IV (Run number XX-1 and XX-2), which were conducted under severe surging conditions, were excluded in the following discussion. The dispersion coefficients obtained from these two experiments are completely out of line with the rest of 14 data points. These very high coefficient values may be due to surging or due to some experimental malfunctions.

5.4.1 Effect of Liquid Velocity

Liquid velocity has no effect on liquid axial dispersion coefficient in the absence of a distributor. Since the independence of dispersion coefficient on liquid velocity was well established in previous experiments using a distributor, only a limited number of runs were made here to determine any difference in the absence of a distributor. The results (Table V) indicate (with the exception of runs XX-1 and XX-2) that liquid velocity has no effect on axial liquid dispersion coefficient. This is true irrespective of the presence or absence of solid particles and their particle size. For example, in the absence of solids, increasing liquid velocity from 0.015 to 0.041 ft/sec resulted in an insignificant change in axial dispersion coefficients (0.475 to 0.513 ft²/sec). Similarly, increasing liquid velocity from 0.021 to 0.053 ft/sec in the presence of 20/30 mesh and -140 mesh particles resulted in a negligible change in axial dispersion coefficient from 0.466 to 0.472 ft²/sec and from 0.452 to 0.404 ft²/sec, respectively. All these results show consistently that liquid velocity has no effect on axial dispersion coefficients.

5.4.2 Effect of Gas Velocity and Solids

No consistent pattern as a function of gas velocity and solid particle size was observed in the absence of distributor plate. The effect of gas velocity and solids on axial dispersion coefficients from the 12-inch diameter column in the absence of a distributor is summarized in Table VI. In this table, wherever possible, dispersion coefficient

Table VI
Effect of Gas Velocity and Solids on Axial Dispersion Coefficients
in the Absence of a Distributor

Gas Velocity ft/sec	Axial Dispersion Coefficients, ft ² /sec		
	<u>No. Solids</u>	<u>20/30 mesh solids</u>	<u>-140 mesh solids</u>
0.05	0.227	0.348	0.204
0.10	0.324	--	--
0.194	--	0.458*	0.428*
0.327	0.494*	0.418	0.369

* - average values.

averaged over values obtained at different liquid velocities. When no solids were present, the axial dispersion coefficient increases with gas velocity as expected. However, the presence of solids complicates the pattern. For both -140 mesh and 20/30 mesh particles the dispersion coefficient initially increases with gas velocity, but decreases with a further increase in gas velocity. Furthermore, at the highest gas velocity employed, the presence of solids resulted in a decrease in axial liquid dispersion coefficients. This is in agreement with earlier results obtained in the presence of a distributor. However, at a gas velocity of 0.05 ft/sec, the presence of -140 mesh solids results in a decrease in the value of the axial dispersion coefficient whereas the presence of 20/30 mesh particles results in an increase. These results indicate that the absence of a distributor plate results in some unusual patterns. At this point, detailed discussion and explanation of these results are not possible. Results from experiments using a different distributor plate may shed some insight into the entrance effects on fluid dynamics of the columns.

6.0 FUTURE WORK

A new distributor plate will be installed in the 12-inch diameter column to perform gas holdup, liquid dispersion, solid dispersion and gas/liquid mass transfer experiments. Efforts will be continued to determine the solid dispersion coefficients independently. Efforts will also be made to determine slurry viscosities. Experimental plans for solids removal study under Task 4 will start towards the end of next quarter.

7.0 REFERENCES

1. "Gas/Slurry Flow in Coal Liquefaction Processes" - Quarterly Technical Progress Report for period 1 October 1979 - 31 December 1979, FE 14801-3, January 1980
2. Deckwer, W. D., Bwickhart, R. and G. Zoll, Chemical Engineering Science, 29, 2177 (1974)
3. Akita, K. and F. Yoshida, Ind. Eng. Chem. Process Des. Develop., 13, 84 (1974)

APPENDIX A

Experimental Tracer Data (Figures A-1 through A-15)

Figure A-1

Figure A-2

Figure A-3

Figure A-4

Figure A-5

Figure A-6

Figure A-7

Figure A-8

Figure A-9

Figure A-10

Figure A-11

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Figure A-12

Figure A-13

Figure A-14

Figure A-15