



### GAS/SLURRY FLOW IN COAL-LIQUEFACTION PROCESSES (FLUID DYNAMICS IN 3-PHASE-FLOW COLUMN). QUARTERLY TECHNICAL PROGRESS REPORT, 1 JANUARY-31 MARCH 1981

AIR PRODUCTS AND CHEMICALS, INC. ALLENTOWN, PA

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Gas/Slurry Flow in Coal-Liquefaction Processes (Fluid Dynamics in 3-Phase-Flow Column)

> Quarterly Technical Progress Report For Period 1 January - 31 March 1981

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### Summary

This is the sixth quarterly report under Contract Number DE-AC22-79ET14801 titled "Gas/Slurry Flow in Coal Liquefaction Processes". This work covers the period 1 January to 31 March 1981. 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. Air/water/sand 3-phase flow systems in both a 5-inch diameter and a 12-inch diameter column is used under Tasks 2, 3 and 4 in this cold-flow simulator study program.

Tetralin was the organic fluid chosen to be investigated under Task 5 of this program. The objective is to study the effect of fluid properties such as surface tension and viscosity on the performance of a cold-flow tubular column. During this quarter, both gas holdup and solids axial distribution in nitrogen/ tetralin/sand system were measured. Our results show that gas holdup in tetralin is higher than that in water for any given gas velocity. Akita and Yoshida's correlation, which describes our air/water/sand gas holdup results very well, fails to fit the tetralin gas holdup data. It is speculated that the failure of Akita and Yoshida's correlation is due to its overestimated dependence on liquid viscosity which causes the predicted values to be lower than the observed data. However, we have found a graphical correlation developed by Hughmark to describe gas holdup in both air/water and nitrogen/tetralin systems reasonably well. The higher gas holdup observed in tetralin is primarily due to its lower surface tension.

Solids axial distribution experiments were performed in both the 5-inch and 12-inch diameter columns with nitrogen/tetralin/sand system in a batch mode (no liquid flow). The behavior of solids in tetralin is similar to that observed previously in air/water/sand system. The distribution of fine particles (140 mesh minus) is nearly homogeneous whereas the 60/80 mesh particles show a substantial concentration gradient. The values of  $V_p/E_{zp}$ , ratio of particle terminal velocity to the solids dispersion coefficient, measured in tetralin are generally lower than those measured in water. These relatively lower  $V_p/E_{zp}$  values are primarily due to the relatively higher viscosity of tetralin which reduces the particle terminal velocity  $V_p$ . It is also found that above the critical gas velocity, which is defined as the minimum velocity to maintain complete solids suspension, the gas flow rate has no effect on solids distribution in good agreement with earlier finding in air/water/sand system.

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### 1.9 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 vesses1. In the EDS and SRC processes, the reactors are essentially empty vessels, whereas the H-Coal process a bed of ebullating catalyst is maintained in the reactor. The major difference 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 system. 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 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 will have decreased surface areas exposed and will likely have diminished catalytic activity. Knowing the particle sizes that can accumulate under commerical 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-foct tall Plexiglas 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 January - 31 March 1981).

### 2.0 TECHNICAL PROGRESS

### 2.1 Task 5 - Organic Fluid Phases

The objective of this task are:

- Select the organic fluid to be used in the 12-inch diameter column by conducting screening experiments in the 5-inch diameter column
  - To design and conduct liquid dispersion experiments for the organic liquid.

To study the effects of:

(a) particle size,

(b) solids concentration,

(c) column diameter,

(d) gas velocity, and

(e) liquid velocity

on axial solids distribution, gas holdup and liquid dispersion using the organic fluid.

### 3.0 EXPERIMENTAL SECTION

### 3.1 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 (January 1980). In addition, a 1 1/2-inch diameter plexiglas column was used in the screening experiments to select the organic liquid to be used under Task 5. Distributor No. 1 (a description of the distributor can be found in Quarterly Report FE-14801-12) was used in all the experiments conducted in this quarterly.

The 5-inch diameter column was placed inside a walk-in hood for experiments using organic liquids. The entire 12-inch diameter column was enclosed to provide safe handling of volatile flammable liquids. A schematic drawing of the enclosure and the safety features associated with it are shown in Figure 1.

The enclosure completely encompassing the 12-inch diameter column is made of 1/2-inch thick plexiglas and 1/8-inch thick aluminum sheets bolted to an aluminum angle-iron framework standing on the floor independently. Horizontal bars were used to connect the enclosure to the surrounding scaffolding ensuring a rigid structure. The bottom 8-feet of the column are completely leak proof and their strength was checked with a hydraulic test to guarantee holding the entire contents of the glass column in case of any breakage. A set of damper blades were installed as a top cover for the enclosure. A large blower was installed to provide adequate venting of the nitrogen and any entrained volatile fumes.

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of 12" Column



Bottom View



### 3.2 Experimental Procedure

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Several experimental procedures are commonly used as part of this program. In this section, a brief description of the experimental procedures for the runs conducted in this quarter are presented.

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### 3.2.1 Gas Holdup

Gas holdup was measured in the 5-inch and 32-inch diameter columns. Several different liquids were used in these experiments. The experiments in the 5-inch diameter column were conducted in the absence of fluid flow. Experiments in the 12-inch diameter column were conducted both in the absence and presence of fluid flow. Gas holdup measured in both the 5-inch and 12-inch diameter columns will be presented in this quarterly report.

In the absence of liquid flow, the experiments were performed by completely filling the column with liquid and then passing nitrogen through the liquid at specified rates. Excess liquid exited the column at the top through a side opening. A waiting period of 5 minutes was allowed to ensure that a steady-state was achieved. The bottom valve was then closed to shut off the gas input. The final liquid level was measured, and the difference between the initial and the final levels represented the gas holdup at that particular gas flow rate. Gas flow rates ranging from 0.05 ft/sec to 0.40 ft/sec were studied.

With fluid flow in the 12-inch diameter column, the liquid and gas passed into the column through a centrally located opening at the bottom. Excess liquid exited the column through a side opening at the top. After steady state was reached, the liquid level was measured. Then a common valve at the bottom was closed stopping both liquid and gas flow simultaneously. The gas void fraction was measured as described above.

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### 3.2.2 Solids Dispersion

Solid dispersion experiments in the batch mode were conducted in both the 5-inch and 12-inch diameter columns. Both sand and glass beads were used in this organic phase study. In the batch operation, gas was bubbled through the column which was filled with tetralin and a known weight of solid particles. During a 30 minute bubbling period at each gas velocity steady state conditions were established. Then slurry samples were withdrawn from sampling ports at various heights of the columns and measured for solids concentration.

The axial solids concentration distribution profile at each operating condition was measured by drawing two samples at each of the four different column levels. One sample was collected from the column center and the other near the wall. The average of these two values represents the solid concentration at that column level.

### 4.0 RESULTS AND DISCUSSION

### 4.1 Screening Tests

The solutions examined in the screening test are listed in Table I. Tetralin was chosen because:

- the physical properties of tetralin (viscosity and surface tension) closely resemble those of the coal liquid.
- tetralin is an aromatic compound typically found in coal-derived process solvent
- the foaming characteristic of tetralin is within experimentally acceptable level.

### 4.2 Gas Holdup

Column diameter has no effect on the gas holdup as illustrated in Figure 2. The gas holdup in the tetralin/nitrogen system measured in both the 5-inch and 12-inch diameter columns show no difference for the

### Table I

Solutions Used in Screening Test

Butanol 5% wt., 95% wt. water Butanol 9.5% wt., 90.5% wt. water Dodecane Ethylene glycol 70% wt., 30% wt. water Ethylene glycol 50% wt., 50% wt. water Ethylene glycol 30% wt., 70% wt. water Ethylene glycol 10% wt., 90% wt. water Hexadecane Methanol 50% wt., 50% wt. water Tetralin Kerosene

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entire range of gas flow rates investigated. This independence on column diameter is in perfect agreement with our previous findings in air/water systems and with other investigators' work on other systems. Three correlations were used to examine the different effects of the various parameters on the gas holdup: Akita and Yoshida, Pilhofer et al., and Hughmark.

Akita and Yoshida's <sup>(1)</sup> correlation is described in detail in the first Quarterly Report (FE-14801-3). Effects of physical properties such as viscosity, surface tension and liquid density are included in this correlation. Although Akita's correlation was found to be adequate for the case of air-water systems as shown in Quarterly Report (FE-14801-12) it did not agree well with the tetralin data. It is speculated that the failure of Akita and Yoshida's correlation is due to its overestimated dependence on liquid viscosity which causes the predicted values to be lower than the observed data. The average root means square error was between 15 and 20% for both the 5-inch and 12-inch columns.

Pilhofer et al correlation (2) includes the effect of viscosity, density and density difference between gas and liquid as follows:

$$\frac{\epsilon_{g}}{1-\epsilon_{g}} = 0.115 \, V_{g}^{3} / (\upsilon_{L} \cdot \Delta_{\rho} / \rho_{L}))^{0.23}$$
(1)

The correlation of Filhofer et al fits the data well in the range of velocities used (i.e. 0.0 - 0.4 ft/sec). An estimate of the root mean percentage error for the 5-inch column was approximately 5% and that for the 12-inch column was approximately 2%. This correlation shows the dependency of gas holdup on the liquid viscosity and the superficial gas velocity.  $\Delta_{\rho}/\rho_{L}$  was considered as unity due to the experiments being conducted at atmospheric conditions because  $\rho_{\sigma}$  is very small.

The correlation of Hughmark (3) is also used here. It is normally a graphical solution for the gas holdup versus a certain dimensional quantity proportional to the gas superficial velocity as follows:

$$\varepsilon_{g}$$
 = function of ((V<sub>G</sub> ( $\frac{62.4}{P_{I}} \times \frac{72.0}{\sigma}$ ))<sup>2.33</sup>)

 $\sigma$  = liquid surface tension in dynes/cm.

This correlation is given as a function of diameter up to 4-inches after which no dependency on the diameter is suggested.

Figure 2 shows the comparison of the Hughmark correlation with the data and is in good agreement with the tetralin data. Comparison of experimental data for gas holdup between water and tetralin suggests that gas holdup is higher in tetralin than it is in water at the same gas velocity. This is due to the lower surface tension of tetralin which increases the possibility of rupture of bubbles to form smaller ones hence increasing drag per unit volume and gas holdup.

### 4.3 Solids Dispersion

All batch experiments conducted in this quarter are listed in Table II. Glass beads of 140/170 mesh and 60/70 mesh were used only in the 5-inch diameter column. Sand of 140 mesh minus and 60/80 mesh were used in both the 5-inch and 12-inch diameter columns.

### 4.3.1. Theoretical Background

Considering a steady state one-dimensional axial dispersion model in a batch operation (no liquid flowing) the solids mass balance at any cross section is

$$V_{p}C_{s} + E_{zp} \frac{dC_{s}}{dL} = 0$$
 (1)

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### Table II

### List of Batch Experiments

### Liquid Phase-Tetralin

### Gas Phase - Nitrogen

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Column	Type of	Particle	Solid	Range of
Diameter,	Solid	Size	Concentration	Gas Velocities
Inches	<u>Particles</u>	Mesh	<u>lb/ft<sup>3</sup></u>	<u>ft/sec</u>
5	Glass beads	140/170	8.32	0.1-0.40
5	Glass beads	140/170 .	25.1	0.1-0.40
5	Glass beads	60/70	8.28	0.1-0.40
1:5	Glass beads	60/70	26.41	0.1-0.40
H 5	Sand	-140	8.28	0.1-0.4
5	Sand	-140	25.74	0.7-0.4
5	Sand	60/80	8.295	0.1-0.4
5	Sand	60/80	26.11	0.1-0.4
12	Sand	-140	6.19	0.133-0.392
12	Sand	-140	22.01	0.133-0.392
12	Sand	60/80	6.65	0.133-0.392
12	Sand	60/80	23.66	0.133-0.392

Vp = settling velocity of solid particles, (ft/sec)

 $C_s = \text{concentration of solid particles in liquid slurry, lb/ft}^3$ 

 $E_{zp}$  = axial dispersion coefficient of solid particles ft<sup>2</sup>/sec

This equation can be written as

$$\frac{d\ln c_s}{dL} = -v_p / E_{zp}$$
(2)

Theoretically a semi-logarithmic plot of  $C_s$  versus L would give a straight line and the value of  $V_p/E_{zp}$  can be measured from its slope. Our experimental data exhibit this linear behavior as shown in the semi-logarithmic plots of Figure 3 through 14.

### 4.3.2 Fine Particles (140/170 Mesh Glass Beads and 140 Mesh Minus Sand)

The results of the fine particles for the 5-inch and 12-inch diameter columns are summarized in Figures 3 through 8. Two concentrations were used for each particle size: 7.5  $lbs/ft^3$  and 24.0  $lbs/ft^3$ . Solids distribution along the length of the column is independent of gas velocity above a velocity of 0.1 ft/sec in good agreement with our previous air/water/sand data.

The  $V_p/E_{zp}$  values measured from the slopes of the straight line plots shown in Figure 3 through 8 are summarized in Table III. Within experimental error  $V_p/E_{zp}$  is independent of gas velocity.

The effect of column diameter on the  $V_p/E_{zp}$  is also summarized in Table III. It shows clearly that the values of  $V_p/E_{zp}$  for the larger diameter column are lower for the entire range of gas velocity investigated. This is not surprising since for the same average concentration but using a larger diameter column one would expect the same  $V_p$  but a much larger value of  $E_{zp}$  due to increased turbulence in the column.

### Table III

# Summary of $V_p/E_{zp}$ for Fine Particles as a Function of Column Diameter and Gas Velocity

### Particle size = 140/170 Mesh for Glass Beads Particle size = 140 Mesh Minus for Sand

Gas	V <u>p/E</u>						
Velocity	$\frac{C_2 = 6.19 - 8.32 \text{ lb/ft}^3}{2}$		$C_{5}$ 22.01 - 25.74 lb/ft <sup>3</sup>				
ŕt/sec	- 5 în.	5 in.	12 in.	5 in.	5 in.	12 in.	
	Glass Beads	Sand	Sand	Glass Beads	Sand	Sand	
0.10	0.147	0.29		.101	.008		
0.133			0.0123			0038	
0.15	0.182	0.64		.115	. 032		
0.20	0.203	0.67		.117	. 023		
0.216			0.015			. 0052	
0.25	0.179	0.48		.114	. 032		
0.308			0.018			. 0045	
0.33	0.183	0.70		. 109	. 028		
0.365			0.0172			.0049	
0.392			0.016			.0031	
0.400	0.172	0.42		. 121	. 030		













As discussed above, the behavior of the solid particles in tetralin closely resembles that in the aqueous system. A quantitative comparison of the solids behavior in these two systems is summarized in Table IV. It is interesting to note that for the same average solids concentration the  $V_p/E_{zp}$  values are higher in the water system than in the tetralin system. This difference in  $V_p/E_{zp}$  values is partially due to the lower particle settling velocity  $V_p$  in the tetralin because of its higher viscosity. The dependence of  $E_{zp}$  on other variables at this stage is not very well understood. The continuous slurry flow experiments to be performed in the next quarter will provide more data to evaluate the behavior of the solid dispersion coefficient.

### 4.3.3 Large Particles (60/70 Mesh Glass Beads; 60/80 Mesh Sand)

There is a marked difference in the behavior of the large particles as opposed to the fine ones. Steeper slopes in the semi-logarithmic plot of  $C_s$  versus L are shown in Figures 9 through 14. Complete suspension could only be achieved at the higher velocity range.

The values of  $V_p/E_{zp}$  for the different flow conditions at two solids concentration 7.5 and 25  $1b/ft^3$  are summarized in Table V. The  $V_p/E_{zp}$  values are independent of gas velocity which was in good agreement with the results using fine particles. This observation is similar to that in air/water/sand systems.

The effect of column diameter is shown in Table V. Increasing the size of the bubble column will decrease the value of  $V_p/E_{zp}$  because of  $E_{zp}$  increases. This observation is in agreement with values calculated for air/water/sand data.

The values of  $V_p/E_{zp}$  for glass beads are higher than that for sand in the entire range of gas velocity investigated. This could be explained in part due to the irregular shape of sand as opposed to the more rounded shape of glass beads which could mean less resistance to settling in the case of glass beads than it is with sand. This leads to basically higher settling velocity for glass. Also the narrow size range of the

### Table IV

Concentration 1b/ft <sup>3</sup>	Water	Tetralin	Column Diameter Inches	Particle Type
6.19 - 8.32	C.275 - O.307	0.147 - 0.172	5	- Glass Beads
6.19 - 8.32	0.029 - 0.073	0.029 - 0.07	5	Sand
6.19 - 8.32	0.015 - 0.024	0.0123 - 0.016	12	Sand
22.01 - 28.6	0.148 - 0.162	0.101 - 0.121	_ 5	Glass Beads
22.01 - 28.6	.013019	.008028	. 5	Sand
22.01 - 28.6	.005007	.00310052	12	Sand

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### Comparison of V<sub>p</sub>/E<sub>zp</sub> For Water and Tetralin Data for 140/170 Mesh Glass Beads and 140 Sand

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Summary of Vp/E for Large Particles as a Function of Column Diameter and Gas Velocity

Particle size = 60/70 Mesh for Glass Beads Particle size = 60/80 Mesh for Sand

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Gas	Vp/Ezp						
Velocity	$C_2 = 3.65 - 8.295  lb/ft^3$			$C_{23.66} - 26.4 \text{ lb/ft}^3$			
ft/sec	- 5 in.	5 in.	12 in.	5 in.	5 in.	12 in.	
	Glass Beads	Sand	Sand	Glass Beads	Sand	Sand	
0.10	0.541	0.369		0.77	0.216		
0.133			0.128			0.120	
0.15	0.573	0.455		0.468	0. <b>466</b>		
0.20	0.605	0.479		0.431	0.605		
0.216			0.118			0.104	
0.25	0.641	0.488		0.514	0.5 <b>84</b>		
0.308			0.115			0.1 <b>09</b>	
0.33	0.652	0.484		0.634	0.580		
0.365 .	•		0.131			0.0956	
0.392			0.127				
0.400	0.691	0.484		0.465	0.497	0.0961	











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beads (60/70 mesh) results in an average particle diameter larger than the undersize range of sand (60/80 mesh). Since particle settling velocity is higher for a larger particle diameter, the glass beads have higher  $V_p$ value, thereby resulting in higher  $V_p/E_{zp}$  value.

### 5.0 FUTURE WORK

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Experiments on solid and liquid dispersion in a continuous mode will be run. The data acquired from these experiments along with measurements of liquid axial dispersion coefficients will help in simulating the solids distribution along the column length.

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