

ABSTRACT

Cold-flow simulation of the SRC-I dissolvers has been performed on three columns with different height-to-diameter ratios to provide a better understanding of the fluid dynamics and operation of a three-phase gas/liquid/solid system. The experiments were conducted in 5-in.-, 12-in.-, and 6-ft-diameter columns. The effects of various parameters on gas holdup, liquid axial dispersion coefficients, solids axial distribution, and gas/liquid mass-transfer coefficients were investigated. In general, ICRC found that flow conditions such as superficial gas and liquid velocities, physical properties of the liquid such as surface tension and viscosity, and the solids loading and particle size could dramatically affect column performance by affecting gas holdup, axial liquid dispersion, gas/liquid mass-transfer coefficients, and solids distribution. This, in turn, could affect scale-up of the dissolver to demonstration plant size.

EXECUTIVE SUMMARY

This report summarizes all the cold-flow work performed in support of the dissolver design for the SRC-I Demonstration Plant. The work is detailed in two separate reports (Ying et al., 1982; McDermott and Ying, 1983). The cold-flow studies included experiments on columns of three different diameters--5 in., 12 in., and 6 ft. The objective was to generate more data relating to the scale-up parameters involved in design of the dissolver for the demonstration plant. The SRC-I plant requires two dissolvers, each 11 ft in diameter by 66 ft high.

Gas holdup data were required to determine the active volume of the dissolver in operation. In all three columns, gas holdup was found to be independent of the column diameter. This was true for all the liquids tested, including water, tetralin, and different mixtures of water and ethylene glycol.

Liquids with different surface tensions, viscosities, and densities were tested to provide data on the variation of column performance with physical properties. These physical properties were found to affect gas holdup, which decreased with an increase in surface tension and viscosity. Also, gas holdup was found to remain steady over the range of superficial liquid velocities tested, but it increased with an increase in the superficial gas velocity. The presence of solid particles in the column did not affect holdup at low gas velocities, but holdup decreased slightly at higher velocities. Correlations by Akita and Yoshida (1973), Pilhofer et al. (1981), and Hughmark (1967) were found to fit the gas holdup data reasonably well.

Two distributor configurations were tested in the 12-in. column, but they were found to have little effect on gas holdup. In the 6-ft column, gas flowing through sparger tubes was found to increase the gas holdup more than gas introduced with other internal configurations such as target plates.

Tests in each column showed that the liquid axial dispersion coefficient increased with an increase in column diameter and superficial gas velocity. In the 12-in. column, the dispersion coefficient was found to be independent of liquid velocity and the configuration of the

inlet distribution. However, the presence of solids decreased the liquid axial dispersion coefficient. Solids accumulation was found to increase when liquid velocity was reduced, and gas velocity had relatively little effect on solids distribution behavior. Larger particle sizes were found to have a steeper solids axial concentration profile than the fines. Increasing the column diameter was found to increase the homogeneity of the solids concentration because mixing increased.

The dissolver column for the demonstration plant is designed so that the hydrogen consumption step is not controlled by the rate of gas/liquid mass transfer. To confirm this, an investigation to calculate this gas/liquid mass-transfer coefficient was made; it showed that the coefficient was independent of column height and inlet distribution using different distributors in the 12-in. column. However, the sparger distribution arrangement used in the 6-ft column increased this transfer coefficient.

A solid dispersion model was developed to predict the solids concentration gradient and accumulation in a gas/liquid/solid upflow system. This model compared favorably with 40 experimental runs using three different liquids in the 12-in. column.

There is concern that pressure will have an effect on gas holdup. The two dissolvers for the demonstration plant are to be connected in series. The gas holdup at design superficial gas velocities is expected to be 20%. However, if 20% is not conservative enough, because of the effect of pressure and gas holdup, the plant design can allow parallel connection of the dissolvers, which would decrease the gas velocities in each dissolver and, hence, lower gas holdup accordingly. Lowering the gas holdup provides more of the total dissolver volume for chemical reaction. The cold-flow work has shown and supported the fact that gas/liquid mass-transfer coefficients are not the limiting factors in the conversion of coal in the dissolver. The work also shows that internals, in general, would not be an added advantage in the dissolver design, because gas holdup and, more importantly, mass-transfer coefficients would not be dramatically enhanced. Even if internals were advantageous to a certain degree, solids might deposit on them, creating serious operational problems if chunks were to break off and flow downstream.

The cold-flow studies support design assumptions that the dissolver will have a more or less homogeneous mixture of solids in the slurry due to the fact that most coal particles will be in the 100-200-mesh size range, which could be well predicted by the dispersion model developed. The SRC-I Baseline Design includes a nozzle arrangement for use as a future solids removal system. This need would arise in case a coarser size coal would be used in the future. A blowdown system will be maintained as part of this design package which would remove any residual buildup of solids in the dissolver. The 6 ft diameter column cold flow studies showed some deposits, in the form of a tapered cone, around the dished bottom inlet to the dissolver. It is to be noted, however, that the specific gravity of sand is about 2.4 while that of coal is 1.4. This should mean that settling in the dissolver bottom would probably not be as significant as in the cold flow tests.

INTRODUCTION

A major part of the coal dissolution section of any liquefaction plant is the dissolver. Although considerable liquefaction occurs in the preheater, most of the necessary chemical changes occur in the dissolver, including sulfur removal, oil and distillate formation, and solvent rehydrogenation.

Vertical tubular dissolvers are used in all of the major processes currently under consideration for commercial coal liquefaction. In the Solvent Refined Coal (SRC), Exxon Donor Solvent (EDS), and H-Coal processes, slurry and gas are concurrently fed upward through these vessels. In the EDS and SRC processes, the reactors are basically empty vessels, whereas in the H-Coal process a bed of ebullating catalyst is maintained in the reactor. Among the EDS, SRC-I, and SRC-II processes, the major differences in dissolver operation are the compositions of the feed streams and reactants within the dissolver. Different hardware such as the distributor plates, draft tubes, or recycle loops can also affect the behavior of slurries in these vessels.

In order to design a technically feasible and cost-effective dissolver, the physical behavior of three-phase systems in tubular columns must be clarified. All of the major processes under development require an understanding of backmixed three-phase systems. In each process, part of the dissolver volume is backmixed. As the design of the 6,000-ton-per-day (tpd) SRC-I Demonstration Plant progresses, the increased vessel size (and other considerations) may dictate the use of reactors in series, which would decrease the overall backmixed characteristics.

The SRC-I Demonstration Plant dissolver will be scaled up considerably from the dissolvers used at the Advanced Coal Liquefaction Facility in Wilsonville, Alabama and at the Ft. Lewis, Washington Pilot Plant. The relative sizes of the dissolvers for the pilot facilities and the SRC-I Demonstration Plant are compared below (at a residence time of 0.56 hr, a gas-feed rate of 20,000 scf of hydrogen per ton of dried coal, and a 38% by weight coal slurry):

Plant	Size (tpd)	Volume (ft ³)	Dissolver diameter	Height (ft)	Superficial velocities (ft/sec)	
					Liquid	Gas
Wilsonville	6-10	18.1	12 in.	23	0.012	0.074
Ft. Lewis	50-61	106.8	24 in.	34	0.017	0.10
SRC-I Demo Plant	5,590*	12,734.4	11 ft	2 x 67	0.06	0.2-0.28

The reactor volumes and diameters differ dramatically. Thus, to make intelligent decisions about the demonstration plant design, we needed more information on the flow properties of three-phase systems in large vessels.

Also important is the effect of differences in gas and liquid superficial velocities on slurry behavior. A fivefold difference in velocity exists between Wilsonville and the demonstration plant design. This difference can have a 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. Some evidence suggests that reactor solids have a definite catalytic effect (Report No. DOE/OR/03054-29). Larger particles will have fewer surface areas exposed, and thus will probably have diminished catalyst activity. Knowing the particle sizes that can accumulate under demonstration plant flow conditions will give us some indication of the size of dissolver solids that should be examined for catalytic activity.

Gas/liquid mass-transfer coefficients ($k_L a$) should also be examined in an industrial size dissolver column. Results from the Wilsonville plant show that the chemical reaction, not gas/liquid mass transfer,

*Of the 6,000 tpd coal fed to the demonstration plant, only 5,590 tpd is fed to the dissolvers; the remainder is gasified to produce hydrogen.

is the rate-limiting step for hydrogen consumption. Results from the 12-in. and 6-ft column studies (Ying et al., 1982) indicate that the demonstration plant dissolver should not be limited by the rate of gas/liquid mass transfer, even when substantial reduction in the transfer coefficient in the presence of solids is considered. Recent work by Kataska et al. (1979) on gas/liquid mass transfer in an 18-ft-diameter vessel also confirms the correlation for two-phase systems. The SRC-I Demonstration Plant dissolver design employs a 2-ft-diameter inlet, which is about four times larger in area than any inlet opening tested. These large entrance areas could create large initial bubbles, whose effect on the overall mass-transfer coefficient is still unknown. Direct measurement of $k_L a$ in a large-scale column with a large inlet would provide the data needed to resolve such questions and provide better data for scale-up.

Because data from the Wilsonville plant show substantial solids accumulation in the dissolver, a solids removal system may be required to control solids in the demonstration plant. A system that can remove the large particles but not the fines, which may be catalytically active, is preferable. Since the amount of accumulation would vary with column dimensions as well as gas and slurry flow rates, evaluation of the removal system in a large-scale dissolver column would provide necessary data for confirming or improving the design.

A series of cold-flow experiments was conducted on 5-in., 12-in., and 6-ft columns in order to generate the data required to ensure effective design. The experiments had the following overall objectives:

1. Study the effects of slurry velocity, gas velocity, solid particle size, solids concentration, liquid viscosity, and surface tension on the performance of a cold-flow tubular column.
2. Develop an effective technique to withdraw slurry from the bottom of a tubular column as a means to control solid concentration.

3. Study the performance of cold-flow tubular columns both with and without internals, i.e., distributors, target plates, and spargers.
4. Study the effect of solids and several internal designs on the gas/liquid mass-transfer rate.

The experiments were performed under two separate research programs, one conducted by Air Products and Chemicals, Inc. (APCI) and the other by ICRC. Results of both programs are integrated and summarized in this report, to provide an overview of all cold-flow dissolver modeling work connected with SRC-I design. Descriptions of the programs follow.

Gas/Slurry Flow in Coal Liquefaction Processes

In this program, APCI studied the effects of fluid dynamics in a three-phase flow dissolver system. The program developed from earlier work performed by APCI when it was a partner with Wheelabrator-Frye in the Joint Venture that later became ICRC. Because the program was related to the EDS and H-Coal processes, as well as SRC-I, it was funded directly by the Department of Energy (Contract DE-AC22-79ET14801).

Both a 5-in. diameter by 5-ft long Plexiglas column and a 12-in. diameter by 25-ft long glass column were used. Various flow conditions and physical properties of liquids were tested, including gas and liquid superficial velocities, plus liquid viscosity, solids concentration, and surface tension. Effects of these parameters on gas holdup, axial liquid dispersion coefficients, axial solids concentration distribution, and gas/liquid mass-transfer coefficients were determined. All runs were conducted at ambient temperature and pressure. The tests were intended to provide the design engineer with data showing the degree to which important design parameters would vary with physical properties of the dissolver system. Because the actual SRC-I process will occur in a dissolver of considerably different dimensions (11 ft wide by 67 ft high) and conditions (840°F and 2,000 psi), ICRC intended that these studies would provide good qualitative trends of the physical process.

Large-Scale Dissolver Cold-Flow Modeling

This test program evolved from the program just described; that work revealed the need for further data on a dissolver of larger diameter, to more closely simulate SRC-I dissolver effects. Therefore, a 6-ft-diameter by 25-ft-long column was used to run two- and three-phase fluid dynamics tests to determine the effects of gas and liquid superficial velocities and different internals on solids behavior and on gas/liquid mass transfer. The importance of this project was the scale-up information that was acquired by using such a large diameter column, one midway in diameter size between that of the demonstration plant and Ft. Lewis dissolvers.

Also, a solids removal system was tested to show the design's feasibility should an increase of solids accumulating in the bottom of the dissolver necessitate use of such a system.

Demonstration Plant Dissolver Design

Details concerning the design of the SRC-I dissolver, and the historical development of the design basis are provided in Harris and Fazekas, Coal Dissolver Design Basis, SRC-I Technical Report, July-December, 1983, DOE/OR/03054-94.

EXPERIMENTAL RESULTS AND DISCUSSION

Gas Holdup

Effect of Column Diameter. Three column diameters were tested: 5 in., 12 in., and 6 ft. The following liquids were tested to study the effect of column diameter on gas holdup: (a) water; (b) tetralin; and (c) glycol (100, 90, 70, and 50% by wt with water). Physical properties of these liquids are compared in Table 1.

In general, column diameter did not affect the gas holdup appreciably. This can be seen in Figures 1 and 2, two examples representing extensive work that was performed. These figures compare gas holdup measured in 5- and 12-in. columns for tetralin and 12-in. and 6-ft columns for water, respectively.

Figure 3 compares gas holdup measured for a 100% glycol/air system in the 5- and 12-in. columns. The data plotted in Figure 3 represent the only instance, out of the large amount of data generated, when gas holdup was observed to differ due to a difference in column diameter. The difference in holdup is thought to be due to the higher viscosity of the 100% glycol, compared to water, i.e., 19 cP for glycol vs. 1 cP for water. This might have caused the wall effect to persist in the 5-in. column, which showed higher holdup. While the differences are at most between 2 and 3% on an absolute scale at the higher superficial gas velocities, gas hold-up in the 5 in. column is 20-40% higher than the 12-in.

Effect of Liquid Velocity. Gas holdup was not appreciably affected by variations in superficial liquid velocity. Velocities tested were between 0.01 and 0.05 ft/sec, which are extremely low. The liquid almost acted as a stagnant fluid, leaving the buoyancy effect as the major force acting on the gas bubbles, with interfacial drag acting as a balancing force against buoyancy. Some of these results are shown in Figures 4, 5, and 6 for air/water in the 12-in. column, tetralin/nitrogen in 12-in. columns, and air/water in the 6-ft column, respectively.

Effect of Gas Velocity. Gas holdup increased with superficial gas velocity, primarily because of the increased breakup of bubbles in the

Table 1
Physical Properties of Different Liquids

Liquid	Temp (°C)	Density (g/mL)	Viscosity (cP)	Surface tension (dynes/cm)
Water	20.0	1.0	1.0	72.0
Tetralin	20.0	0.97	2.67	32.0
100% glycol	2.5	1.17	19.0	48.2
90% glycol	21.6	1.107	13.08	45.9
70% glycol	21.6	1.091	6.40	48.8
50% glycol	18.5	1.078	4.10	56.7

gas phase when the regime was bubbly. The expected increase in bubble frequency will also increase the interfacial drag force per dissolver unit volume, which in turn will increase liquid entrainment and gas holdup. This effect is clearly shown in Figures 1, 2, 3, and 6.

Effect of Solids. Gas holdup was not significantly affected by solids in the dissolver. Figures 7 and 8 show the effects of solids on gas holdup in the 12-in. column. The air/water results plotted in Figure 7 show a decrease in holdup of about 14.0% at higher velocities for systems with solids, compared to the results for the system without solids. However, the tetralin data (Figure 8) show very little variation with solids.

Effect of Distributors (Entrance Effects) and Internals. Entrance effects due to different distributor geometries did not seem to affect gas holdup in the 12-in. column. Figures 9-14 show the different results. For details and photographs of the shape of the two distributors used, refer to the final report by Ying et al. (1982). With or without distributors, liquid flow, or solids present, the holdup values are practically the same. The negligible effect of the distributors on gas holdup in the 12-in. column may be due to the fact that the inlet pipe and the openings in the distributor plates are almost the same size--1 in. and 9/16 in., respectively. The bubbles coming from the inlet tube are probably of the same order of magnitude as the pipe diameter. Since the distributor holes are not that much smaller than the pipe and, consequently, the bubbles, there probably is not a major breakup of the bubbles as they pass through the distributors.

Another possible explanation for the lack of change in gas holdup, with or without distributors, is the high length-to-inside diameter ratio (L/D) of the 12-in. column--25.0. This high ratio would help establish a flow situation unaffected by inlet and exit conditions.

Figure 15 summarizes the variation of gas holdup with the different internals that were tested in the 6-ft column. Target plates, a bubble cap, and gas spargers were tested to see how effective they are in dispersing the gas bubbles and making the entrance region more homogeneous.

Without internals and with the gas introduced to the 6-ft dissolver through the main 1-ft feed pipe, there was a bubble-free region near the entrance. The jet of gas coming in through the feed pipe needed about 1 column diameter length before it occupied the entire cross-sectional area of the dissolver. This is indicative of an idle volume, where no gas mixing occurs. The different internals tested exhibited quite a range of gas holdup values for the same gas velocity, especially at higher gas velocities. The highest holdup was achieved with the gas spargers, which consisted of relatively small pipes (about 1.5 in. in diameter) having small holes around the periphery to break up bubbles before they entered the dissolver. In contrast, the lowest gas holdups were produced by the 1-ft target plate, the 4-ft bubble cap, and the absence of internals. This indicates that a combination of idle dissolver volume coupled with relatively larger bubbles than those produced by the spargers could still exist.

The relatively low L/D ratio of the 6-ft column, i.e., 4.16 compared to 25 for the 12-in. column, is suspected to have contributed to the large differences in gas holdup shown by the use of the different internals. However, the demonstration plant dissolver's L/D ratio will be 6.1; thus, the gas holdup should be closer to the 6-ft column data.

It is preferable to avoid using internals such as distributor plates in the demonstration plant dissolvers (apart from the spargers), since internals may cause thermal stresses in the dissolver shell. Also, chunks of solid material may form on the internals, which could break off periodically and threaten the operation of other process components.

Gas Holdup Correlations. Predicting gas holdup reasonably well is important, as it affects the design of the column. In this study, three correlations were examined (see details in Table 2):

- ° Hughmark's correlation (1967), which includes the effect of gas superficial velocity, slip velocity, liquid density, surface tension. Column diameter is used as a parameter.
- ° Akita and Yoshida's correlation (1973), which includes the effect of liquid density, viscosity, and surface tension, along with gas superficial velocity.

Table 2

Correlations for Predicting Gas Holdup

◦ Hughmark (1967)

$$\epsilon_g = \frac{V_{sg}}{U_s} \frac{62.4}{\rho_L} \frac{72}{\sigma}^{1/3}$$

◦ Akita and Yoshida (1973)

$$\frac{\epsilon_g}{(1 - \epsilon_g)^4} = C_1 (N_{Bo})^{1/8} (N_{Ga})^{1/2} (N_{Fr})$$

◦ Pilhofer et al. (1981)

$$\frac{\epsilon_g}{1 - \epsilon_g} = 0.115 [V_{sg}^3 / (v_L g \Delta \rho / \rho)]^{0.23}$$

- where:
- ϵ_g = gas void fraction
 - V_{sg} = superficial gas velocity (ft/sec)
 - U_s = slip velocity (ft/sec)
 - ρ_L = liquid density (lb/ft³)
 - σ = liquid surface tension (dynes/cm)
 - N_{Bo} = $gD^2\rho_L/\sigma$ (Bond number) (σ in lb/sec²)
 - N_{Ga} = gD^3/v_L^2 (Galileo number)
 - N_{Fr} = $V_{sg}/(gD)^{0.5}$ (Froude number)
 - C_1 = 0.20 for nonpolar solutions; 0.25 for polar solutions
 - D = column diameter (ft)
 - v_L = liquid kinematic viscosity (ft²/sec)
 - g = gravitational acceleration (32.2 ft/sec²)
 - $\Delta\rho$ = $\rho_L - \rho_g$; ρ_g - gas density (lb/ft³)

- ° Pilhofer et al.'s correlation (1981), which includes the effects of superficial gas velocity, kinematic liquid viscosity, and liquid density.

Figure 12 compares the correlations of Akita and Yoshida and Pilhofer et al. for the air/water system in the 12-in. column. Pilhofer's correlation drastically overpredicts the gas holdup, while that of Akita and Yoshida predicts the data very reasonably, except at higher gas velocities ($V_g > 0.37$ ft/sec), where the correlation overpredicts the gas holdup by about 10%. Figure 16 shows how Hughmark's and Pilhofer's correlations compare for the 5- and 12-in.-column data using the tetralin/nitrogen system, where both correlations show good agreement with the experimental data. Hughmark's correlation is a graphical solution of gas holdup, where the diameter is considered as a parameter. Akita and Yoshida's correlation shows a lower correlation for nonpolar fluids, with $C_1 = 0.2$, than for polar fluids, with $C_1 = 0.25$, with the latter case showing improvement in the gas holdup prediction, although tetralin is considered nonpolar.

Figures 17-20 present the results of gas holdup measurements of different glycol/water mixtures in a 12-in. column. Pilhofer's correlation gives slightly better results, except for the 90% glycol/water mixture.

In general, no one correlation could describe all the gas holdup data for the different fluids tested. Pilhofer's correlation seems to predict the organic fluid data rather well, i.e., tetralin and glycol. In contrast, Akita and Yoshida's correlations modeled water and glycol rather well, but underpredicted tetralin by about 20-25% ($C_1 = 0.2$). At this stage, the data available are for a very limited variety of fluids, hence limiting the use of any correlation derived from these data to interpolation among the data only. The data show that, primarily, gas velocity, liquid viscosity, and surface tension affect holdup. Apparently, reducing the liquid viscosity or surface tension will increase gas holdup. Also, liquid density is suspected to affect holdup. However, at this time, separating the effect of each of these properties from the others is rather difficult.

Another important result that has been mentioned in the literature is the effect of pressure on the system's gas holdup. Figure 21 shows results of gas holdup measurements in systems at elevated pressures; increased pressure clearly increases gas holdup (see effect at 2,000 vs. 500 psi). These results were obtained from Exxon's work (1980) on development of the Exxon donor solvent (EDS) coal liquefaction process. The geometric system is the 2.6-in.-diameter dissolver of the 1-ton/day pilot plant Exxon used. These trends in gas holdup were also confirmed in their 250-ton/day pilot plant, which has a 2-ft-diameter dissolver (see Figure 22).

There is concern that the predicted gas holdup in the SRC-I dissolver might be in considerable error if one uses gas holdup values measured at ambient conditions. Exxon data indicate gas hold-up could be twice that at ambient. This would reduce liquid residence time by 25%, thereby reducing coal conversion. Additional experiments with pressure as a variable should be conducted to clarify the effect of pressure on gas holdup.

Liquid Axial Dispersion Coefficient

Liquid axial dispersion coefficients are important in the design of the SRC-I dissolver. An idea of the values to be expected can indicate the degree to which the dissolver will be backmixed. Backmixing affects the suspension of solids in the dissolver and the rate of mass transfer. Experiments to determine the values of this dispersion coefficient were made in two columns of diameters 12 in. and 6 ft. Details of how the coefficient is calculated are found in Ying et al. (1982) and McDermott and Ying (1983). The experiments were run in both batch and continuous modes. Batch experiments have no liquid flow, while the continuous experiments do.

Effect of Liquid Velocity. Liquid velocity was investigated in the 12-in. column and found to have a negligible effect on the axial dispersion coefficient. Tables 3 and 4 show the effect of liquid velocity on an air/water/sand system, and Table 5 shows the effect of liquid velocity on the 50% glycol/air mixture. All the data show some obvious random fluctuations, but no general trend was observed as a function of liquid velocity.

Table 3

Effect of Liquid Velocity on Axial Liquid Dispersion
Coefficient in the Presence of Distributor No. 1
(Gas Velocity = 0.327 ft/sec;
Water/Air; 12-in. Column)

Liquid velocity (ft/sec)	Axial dispersion coefficient (ft ² /sec)				
	No solids	20/30 mesh		60/80 mesh	
		$C_S^a = 5.0$ lb/ft ³	$C_S = 20.0$ lb/ft ³	$C_S = 5.0$ lb/ft ³	$C_S = 20.0$ lb/ft ³
0.009	0.61	0.51			
0.013			0.20		
0.018				0.43	
0.026			0.38		
0.030		0.48	0.50	0.38	
0.035				0.44	
0.040		0.47	0.36		
0.049	0.62		0.38	0.42	
0.053		0.47		0.48	
0.059		0.48			

^a C_S = concentration of solids.

Table 4

Effect of Liquid Velocity on Axial
Dispersion Coefficients in the Presence of Distributor No. 2
(Gas Velocity = 0.327 ft/sec; Water/Air; 12-in. Column)

V_L (ft/sec)	Axial dispersion coefficients (ft ² /sec)				
	No solids	20/30 mesh		-140 mesh	
		$C_S = 5 \text{ lb/ft}^3$	$C_S = 20 \text{ lb/ft}^3$	$C_S = 3.7 \text{ lb/ft}^3$	$C_S = 14.9 \text{ lb/ft}^3$
0.02	0.54	0.43	0.37	0.45	0.34
0.04	0.61	0.43	0.32	0.42	0.36
0.05	0.48	0.40	0.36	0.41	0.40

Table 5

Axial Liquid Dispersion Values (E_{ZL}) in
a 50% Glycol Mixture with a 12-in. Column

V_g (ft/sec)	V_L (ft/sec)	E_{ZL} (ft ² /sec)	Average value (ft ² /sec)	Remarks
0.05	0.00	0.381		No solids
0.05	0.01	0.479		No solids
0.05	0.03	0.434	0.496	No solids
0.05	0.05	0.575		No solids
0.10	0.00	0.568		No solids
0.197	0.00	0.607		No solids
0.197	0.01	0.618		No solids
0.197	0.03	0.618	0.57	No solids
0.197	0.05	0.475		No solids
0.40	0.00	0.717		No solids
0.40	0.01	0.841		No solids
0.40	0.03	1.1	0.891	No solids
0.40	0.05	0.915		No solids
0.40	0.05	0.915	0.915	60/80-mesh sand used 16.5-lb/ft ³ average concentration

Hence, for the demonstration plant, the effect of slurry velocity on the liquid dispersion coefficient is not critical, provided the design superficial liquid velocity is within the limit of 0.06 ft/sec.

Effect of Gas Velocity. By far, superficial gas velocity (V_g) affects the liquid axial dispersion coefficient (E_{ZL}) the most. Its effect is also obviously closely tied to the average gas holdup in the column. The liquid axial dispersion coefficient increases with an increase in gas velocity. Figures 23-26 plot the variations of E_{ZL} with superficial gas velocity in water, tetralin, and glycol in the 12-in. column and in water in the 6-ft column, respectively. In all these graphs, E_{ZL} increases with V_g . This could be explained by the increased turbulent activity that accompanies increases in gas velocity. Hence, when establishing operating flow conditions, the liquid axial dispersion coefficient must be determined and matched with the requirements of the coal conversion design criteria.

Effect of Solids. Most of the effects of solid concentration on E_{ZL} were studied using air/water and tetralin systems in the 12-in. column. Figures 23 and 24 show these results. Solids clearly decreased the value of E_{ZL} , either because they dampened turbulent liquid fluctuations or because of increased coalescence of bubbles, or both.

Effect of Internals. Tables 3 and 4 and Figure 26 show that adding different internals to the column does not seem to affect the liquid axial dispersion coefficient. Figure 26 shows that introducing part of the total gas flow through the gas spargers and the rest through the 12-in. inlet line does not seem to influence the value of E_{ZL} , within the accuracy of the data. This means that use of internals or distributors in the dissolvers of the SRC-I Demonstration Plant to enhance any axial liquid mixing does not seem warranted.

Liquid Axial Dispersion Coefficient Correlations. Many correlations have been suggested to predict variation of the liquid axial dispersion coefficient as a function of gas superficial velocity, column diameter, liquid viscosity, and liquid density. Of the many correlations that predict liquid axial dispersion coefficients, two were found to predict values close to the experimental data and, hence, are presented here for comparison:

Hikita and Kikukawa (1974):

$$E_{ZL} = (0.366 + 0.674V_g^{0.77})D^{1.25}(1/\mu_L)^{0.12} \quad (1)$$

Baird and Rice (1975):

$$E_{ZL} = 0.35D^{1.33}(V_g g)^{0.33} \quad (2)$$

where E_{ZL} = liquid axial dispersion coefficient (ft²/sec)
 V_g = superficial gas velocity (ft/sec)
 μ_L = liquid viscosity (cP)

Both correlations include superficial gas velocity and the column diameter effect; that of Hikita and Kikukawa also includes a correction due to liquid density.

Figure 27 plots different values of E_{ZL} measured in the 12-in. and 6-ft columns. The 12-in. data include air/water, nitrogen/tetralin, and air/50% glycol solutions. The figure also compares the data with the two correlations. All values of E_{ZL} measured in the 12-in. column agree reasonably well with both correlations. For the 6-ft column, Hikita and Kikukawa's correlation underpredicted the observed data, whereas Baird and Rice's correlation overpredicted the experimental data.

Note that the length-to-diameter ratio for the 12-in. column is 25.0, while that for the 6-ft column is only 4.16. The L/D for the demonstration plant dissolver is 6.0. The correlations shown do not consider the effect of column length, because of the assumption that the flow developing length is of minimal importance compared to the total height. At this time, we do not know the importance of the effect of length for a dissolver of demonstration plant size. Thus, an arithmetic mean of the two correlations should give a pretty good estimate of the E_{ZL} experimental values, based on the experimental data from the 6-ft column.

Another important observation is that liquid physical properties minimally affect the prediction of E_{ZL} . In Hikita and Kikukawa's correlation (1974), the viscosity enters the formulation, but the exponent is very small, i.e., $\mu_L^{0.12}$. The mixtures that were tested in this

study had viscosities varying between 1 and 4.1 cP, yet Baird and Rice's correlation predicted those reasonably well. Thus, we can conclude that physical properties, especially viscosity, do not seem to strongly impact E_{ZL} ; this is particularly true for lower viscosity values. The average viscosity in the dissolver is expected to be low, i.e., 0.5-1.0 cP, which seems to support the use of these correlations.

By extrapolating these correlations and assuming a negligible entrance effect, one can predict a range of feasible values for the axial liquid dispersion coefficient. Using $D = 11$ ft, $V_g = 0.4$ ft/sec, and $\mu_L = 1.0$ cP, values can be derived for either correlation:

Hikita and Kikukawa (1974):

$$E_{ZL} = 14.00 \text{ ft}^2/\text{sec} \quad (3)$$

Baird and Rice (1975):

$$E_{ZL} = 18.11 \text{ ft}^2/\text{sec} \quad (4)$$

As expected, this value is larger than that calculated in the 6-ft column. The importance of this term will be seen later in the effect of this parameter on axial solids distribution and scale-up for the demonstration plant.

Gas/Liquid Mass-Transfer Coefficient

Gas/liquid mass-transfer coefficients ($k_L a$) were measured in the 5-in., 12-in., and 6-ft columns. Liquid dispersion tests in the three columns showed that the liquid phase is very close to being completely backmixed. Using the tanks in series model (see page 45), it has been estimated that the 12-in. column can be represented by 1.1 stirred tanks in series. Hence, one can use simplifying assumptions relevant to backmixing to calculate the gas/liquid mass-transfer coefficient.

In general, the $k_L a$ increases with an increase in gas superficial velocity. This was found to be true with the 5-in. and 6-ft columns, whereas the 12-in. column data showed a very slight increase over a wide range of gas velocities.

Figures 28-32 plot the data for the three columns. $k_L a$ values for the 5- and 12-in. columns ranged between 0.01 and 0.04 sec^{-1} over a range of gas velocities from 0.05 to 0.4 ft/sec. $k_L a$ values for the 6-ft column ranged from 0.02 to 0.05 sec^{-1} for velocities between 0.1 and 0.5 ft/sec. These data are averages of various axial positions along the column. The individual values did not show much scatter, indicating that these columns were very well backmixed. An increase in the value of $k_L a$ due to an increase of V_g is due mainly to an increase in the interfacial area per unit volume of dissolver. This has been demonstrated recently by Mangartz and Pilhofer (1981), who showed that the calculated values of k_L alone are almost constant with respect to gas velocity.

Figures 29-32 also plot the mass-transfer coefficient data as a function of different internals in the column. In the 12-in. column study, the effect of different distributor geometries was minimal (Figure 29). The 6-ft column data show there is practically no difference in the $k_L a$ values for the different target plates and bubble caps tested. The mass-transfer coefficient increased noticeably when spargers were used to inject air bubbles through the system. This increase could be attributed to the increase in interfacial area and gas holdup as the bubbles broke into smaller sizes upon passing through the sparger holes. The increase was about 20%.

The effect of solids on the mass-transfer coefficient can be summarized from Figures 28-32 for all three columns studied. The use of -140 mesh sand slightly decreased the value of $k_L a$ in both the 5- and 12-in. columns. However, no other trends were apparent for the effect of solids on $k_L a$ in the 12-in. and 6-ft columns.

Comparison of the $k_L a$ data with the Akita/Yoshida correlation is shown in Figures 29 and 32. Akita and Yoshida expressed $k_L a$ in a non-dimensional form (the Sherwood number), as the product of four other dimensionless quantities:

$$N_{Sh}(aD) = C_1 (N_{Sc})^{0.5} (N_{Bo})^{0.62} (N_{Ga})^{0.31} (\epsilon_g)^{1.1} \quad (5)$$

where N_{Sh} = the Sherwood number = $k_L D / D_L$ (dimensionless)
 a = specific gas/liquid interfacial area (l^{-1})
 D = column diameter (l)
 C_1 = 0.60, dimensionless constant¹
 N_{Sc} = Schmidt number = ν_L / D_L (dimensionless)
 N_{Bo} = Bond number = $g D^2 \rho_L / \gamma$ (dimensionless)
 N_{Ga} = Galileo number = $g D^3 / \nu_L^2$ (dimensionless)
 ϵ_g = gas holdup (dimensionless)
 k_L = liquid-phase mass-transfer coefficient ($l T^{-1}$)
 D_L = liquid-phase diffusivity ($l^2 T^{-1}$)
 ν_L = liquid-phase kinematic viscosity ($l^2 T^{-1}$)
 g = gravitational constant ($l T^{-2}$)
 ρ_L = liquid density ($M l^{-3}$)
 γ = surface tension ($M T^{-2}$).
 l = length dimension
 M = mass dimension
 T = time dimension

The correlation shows the effect of physical properties of the liquids, as well as the column's inside diameter (D). It is interesting to note that, in this correlation, $k_L a$ varies as $D^{0.17}$, which is a relatively small power compared to the power to which the diameter was raised in the axial liquid dispersion coefficient (1.25-1.5) (Baird and Rice, 1975; Hikita and Kikukawa, 1974).

In this correlation, the empirical constant (C_1) equals 0.60. ICRC's measured $k_L a$ values were appreciably lower than this predicted curve (see Figures 29 and 32). Sufficient data are not yet available to develop a better in-house correlation to describe $k_L a$ behavior in a larger diameter vessel. However, with C_1 values of 0.40 and 0.50, a first-order approximation adjusting the empirical constant yielded data that fell reasonably within the curves.

The gas/liquid volumetric mass-transfer coefficient in SRC-I Demonstration Plant dissolvers is estimated to be 230 hr^{-1} by using the Akita/Yoshida correlation and the modified empirical constant of 0.48

¹Note that in some figures this dimensionless constant is represented by β .

(which fit the 6-ft column data). The maximum hydrogen-transfer rate can then be calculated by assuming zero hydrogen concentration in the bulk liquid, i.e., instantaneous consumption of the dissolver hydrogen. The results indicate that this maximum transfer rate is more than one order of magnitude higher than the Baseline design hydrogen consumption rate. Therefore, it is concluded that the SRC-I dissolver design is not limited by gas/liquid mass transfer.

Note also that if, as mentioned earlier, the gas holdup is higher than measured in the cold-flow experiments due to pressure effects, at the same gas velocity, the k_L value from the correlation would be higher by a factor of ~2.0 than what it is now. Hence, the design of the hydrogen intake to the slurry in the dissolver is on the conservative side.

Solids Dispersion

Experimental runs were also conducted in the 5-in., 12-in., and 6-ft columns using different solid materials of varying particle size. The aim was to determine how flow variables along with the physical properties of the fluids affect the solids distribution axially. Two general modes of running the solids dispersion experiments were used: batch and continuous. In the batch mode, gas alone was bubbled through a stagnant slurry in the dissolver. Among the objectives to be determined were gas holdup and the ratio of the solids settling velocity to the solids axial dispersion coefficient (V_p/E_{zp}). In contrast, the continuous mode was run by circulating the slurry plus gas up through the column. This method allowed continuous study of solids distribution and determination of the effects of feed rate and slurry flow on gas holdup and axial solids distribution. The solids dispersion experiments used a variety of solid particles, including glass beads and sand particles. The effect of particle size was assessed by using -140, 30/50, 60/80, and 100/200 mesh sand and 60/70 and 140/170 mesh glass beads.

Theoretical Background. In a batch operation (with no liquid flowing), at any cross section of the column, the mass balance of solids particles at steady-state conditions results in the following expression:

$$V_p C_s + E_{Zp} (dC_s/dL) = 0 \quad (6)$$

where V_p = settling velocity of solid particles (ft/sec)
 C_s = concentration of solid particles in liquid (lb/ft³)
 E_{Zp} = dispersion coefficient of solid particles (ft²/sec)
 L = distance from the bottom of the column (ft)

Equation 6 can be rewritten as:

$$(d \ln C_s)/dL = -V_p/E_{Zp} \quad (7)$$

Therefore, a plot of $\ln C_s$ vs. L should yield a straight line, provided that both V_p and E_{Zp} are not functions of either solid concentration or column level. Figures 33 to 40 are semilogarithmic plots of C_s vs. L as a function of gas velocity for some of the experiments conducted in this research program. In general, the experimental points follow the straight-line relationship suggested by the theory. The range of gas velocities included those expected in the SRC-I dissolver, while the solids concentrations were higher than those anticipated in the demonstration plant.

Fine Particles (Batch, 140/170-Mesh Glass Beads and -140-Mesh Sand).

The final report by Ying et al. (1982) details the different run conditions. Figures 33-35 graph the axial distribution of fine particles in the 5- and 12-in. columns using tetralin as the liquid phase. Data on other liquids can be found in Ying et al. (1982). In general, these profiles are flat, with very small slopes, which is expected because the terminal velocity of small particles is usually small. In fact, as the particles become smaller, the interfacial drag per unit volume usually increases, thus decreasing the terminal velocity. Particles of this size, therefore, will stay well-mixed in the dissolver under a wide range of gas velocities.

In addition, the distribution of solids concentration was independent of gas velocity, which implies that the solid dispersion coefficient is also independent of gas velocity. However, if the effective

V_p in equation 6 varies with gas velocity, the solid dispersion coefficient will be proportionally dependent on gas velocity.

Some of the V_p/E_{Zp} vs. column length plots are shown in Figures 33-35 since V_p/E_{Zp} is measured from the slopes of these figures, and all the V_p/E_{Zp} results are summarized in Tables 6-9. The results obtained from the air/water/solid system (Table 6) showed that the glass beads had consistently higher V_p/E_{Zp} values than sand for both concentrations; the V_p/E_{Zp} value for sand is about 10-15% of that for glass beads. This difference can be attributed to a higher settling velocity for glass beads, because the average particle size for the 140/170-mesh glass beads was at least a factor of two larger than that of the -140-mesh sand. Based upon Stoke's law, the V_p for glass beads would be at least a factor of four higher than the sand. Therefore, the V_p/E_{Zp} value for sand is expected to be at least less than 25% of the value for glass beads, for a constant E_{Zp} . This explains the difference in V_p/E_{Zp} values observed between glass beads and sand particles.

The effects of column diameter and solid concentration on V_p/E_{Zp} are quite obvious, as shown in Table 6. V_p/E_{Zp} decreased with both increasing column diameter and solids concentration. The dependence on column diameter is not surprising, because the degree of liquid back-mixing and the liquid dispersion coefficient increased with increasing column diameter; hence, the solid dispersion coefficient is expected to increase if there is any column diameter effect. The decrease in the V_p/E_{Zp} value observed with increasing column diameter directly reflects an increase in E_{Zp} , which is in qualitative agreement with the above expectation. The reason for the dependence on solid concentration is not clear, however. It is speculated that the particle settling velocity is hindered by the neighboring particles. This qualitatively explains the decrease in V_p/E_{Zp} with increased solids loading.

The fine particles behave similarly in tetralin (Table 7) and glycol/water mixtures (Tables 8 and 9) as in the aqueous system (Table 6). The V_p/E_{Zp} values were consistently higher when glass beads were used instead of the fine silica (sand) particles. The V_p/E_{Zp} ratio decreased as the sand concentration and the column diameter were increased. The V_p/E_{Zp} ratio was found to be essentially independent of gas velocity in all three systems, although there is wide data scatter for low concentrations of sand in the 5-in. column.

Table 6

Summary of V_p/E_{Zp} for Fine Particles as a Function of Gas Velocity
 [Particle Size = 140/170 Mesh for Glass Bead Experiments;
 Particle Size = -140 Mesh for Sand Experiments (Water)]

Gas velocity (ft/sec)	V_p/E_{Zp} (ft ⁻¹)					
	$C_s = 7.5$ lb/ft ³			$C_s = 28.6$ lb/ft ³		
	5-in. column		12-in. column,	5-in. column		12-in. column,
	Glass beads	Sand	sand	Glass beads	Sand	sand
0.10	0.307	0.029	0.016		0.013	
0.15	0.312		0.018	0.156	0.014	0.006
0.20	0.292	0.047	0.017	0.157	0.016	0.005
0.24			0.019			0.007
0.28			0.016			0.005
0.30		0.034				
0.33	0.330	0.043	0.017	0.162	0.019	0.006
0.37			0.015			0.006
0.43	0.275	0.073	0.021	0.148		0.006

Table 7

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 for Sand (Tetralin)]

Gas velocity (ft/sec)	V_p/E_{zp} (ft ⁻¹)					
	$C_s = 6.19-8.32$ lb/ft ³			$C_s = 22.01-25.74$ lb/ft ³		
	5-in. column		12-in. column,	5-in. column		12-in. column,
	Glass beads	Sand	sand	Glass beads	Sand	sand
0.100	0.147	0.29		0.101	0.008	
0.133			0.0123			0.0038
0.150	0.182	0.64		0.115	0.032	
0.200	0.203	0.67		0.117	0.023	
0.216			0.0150			0.0052
0.250	0.179	0.48		0.114	0.032	
0.308			0.0180			0.0045
0.330	0.183	0.70		0.109	0.028	
0.365			0.0172			0.0049
0.392			0.0160			0.0031
0.400	0.172	0.42		0.121	0.030	

Table 8

Values of V_p/E_{z_p} for -140-Mesh Sand in Glycol/Water Mixtures
(12-in.-Diameter Column)

Gas velocity (ft/sec)	V_p/E_{z_p} (ft ⁻¹) (90% glycol)	V_p/E_{z_p} (ft ⁻¹) (70% glycol)
0.216	0.0043	0.0050
0.392	0.0040	0.0046
0.043	0.0030	0.0044
Viscosity (cP)	13.08	6.2

Table 9

Values of V_p/E_{zp} for Fine Particles as a Function of
 Gas Velocity in a 12-in.-Diameter Column
 [Particle Size = -140 Mesh; Liquid = Glycol Mixture (50% by Wt)]

Gas velocity (ft/sec)	C_s (lb/ft ³)	V_p/E_{zp} (ft ⁻¹)
0.2	6.57	0.0210
	18.37	0.0096
	20.76	0.0065
0.4	6.57	0.0161
	18.37	0.0107
	20.76	0.0071
0.5	6.57	0.0163

In the 5-in. column with glass beads, the values of V_p/E_{Zp} decreased by 30-40% when water was substituted by tetralin at the same velocities. This result shows the effect of viscosity on solids distribution and V_p/E_{Zp} . As viscosity increases, the solid settling velocity (V_p) decreases, thereby decreasing the V_p/E_{Zp} values.

However, the viscosity effect is less distinct with the fine sand particles. For an order of magnitude increase in viscosity from 1 cP (pure water) to 13 cP (90% glycol solution), only a twofold difference in V_p/E_{Zp} was measured. For this fine particle size sand, the concentration profiles were almost completely flat. The value of V_p/E_{Zp} measured from the slope of a flat curve is a very small quantity. Therefore, the viscosity effect is less apparent. It is also likely that E_{Zp} decreases with increasing viscosity, further dampening the viscosity effect on the V_p/E_{Zp} ratio.

Large Particles (Batch, 60/70-Mesh Glass Beads and 60/80-Mesh Sand).

Marked differences are seen in the behavior of large particles compared to fine particles. Significant gradients in the solids concentration were measured for both sand and glass beads. Complete suspension of these large particles could not be achieved at low gas velocities. Sampling from the bottom port was extremely difficult, and the reliability of this sample is questionable. Hence, data from the lowest sampling port were excluded from the analysis. [Details of sampling techniques are found in Ying et al. (1982).]

Some of the V_p/E_{Zp} values for these large particles measured from the slopes of the plots are shown in Figures 36-40 at different solids concentration and column diameters, and all the V_p/E_{Zp} values are summarized as a function of gas velocity in Tables 10-13. V_p/E_{Zp} for large particles increases markedly compared to the values for fine particles, primarily because the particle terminal velocity increases due to an increase in particle size.

In most of the experiments conducted, complete suspension was achieved only at higher gas velocities. The critical gas velocity, which is defined as the velocity above which all particles are in complete suspension, for the 60/80-mesh sand particles in water was deter-

mined to be between 0.193 and 0.217 ft/sec. The results shown in Table 10 indicated that, above the critical gas velocity, all the V_p/E_{Zp} values showed no systematic change with gas velocity. With the limited amount of data available, the V_p/E_{Zp} values seemed to be independent of gas velocity, which is consistent with the behavior observed with the fine particles.

The results with water (Table 10) and tetralin (Table 11) as the liquid phase show that increasing the column diameter resulted in a decrease in V_p/E_{Zp} values. This means an increase in solid dispersion coefficient with increasing column diameter, and is consistent with the observation that the V_p/E_{Zp} values for the sand particles are slightly less than those for the glass beads, although the same particle size range, namely 60/70 mesh, was used. This slight difference in V_p/E_{Zp} values for sand and glass beads possibly reflected some intrinsic difference in the distribution of these particles within the 60/70-mesh range and the effect of particle shape.

The V_p/E_{Zp} ratio decreases as the liquid velocity increases. The results from Tables 10-13 are summarized in Table 14 to illustrate the effect of viscosity. Within the range of gas velocity and solids concentration in this study, the V_p/E_{Zp} decreased with increasing liquid viscosity. This decrease in V_p/E_{Zp} is primarily due to the reduction of solid terminal velocity with increasing viscosity. This finding agrees with observations on the fine particles.

The straight-line plots in Figures 33-40 and the good agreement in the V_p/E_{Zp} behavior with the wide range of parameters strongly support the validity of this one-dimensional mass-balance model (equation 7) for describing the behavior of solids in a three-phase upflow column in a batch mode.

Continuous-Flow Solids Distribution. Continuous-flow solids distribution was measured in the 5-in., 12-in., and 6-ft columns. The effect of liquid velocity, gas velocity, feed slurry concentration, particle size, column diameter, column internals, and physical properties of the liquid on the solids distribution profile were studied. The measurement techniques are detailed in Ying et al. (1982).

Table 10

Summary of V_p/E_{Zp} for Large Particles as a Function of Gas Velocity
 [Particle Size = 60/70 Mesh for 5-in.-Column Experiments;
 Particle Size = 60/80 Mesh for 12-in.-Column Experiments)

Gas velocity (ft/sec)	V_p/E_{Zp}					
	$C_s = 7.5 \text{ lb/ft}^3$			$C_s = 28.6 \text{ lb/ft}^3$		
	5-in. column		12-in. column,	5-in. column		12-in. column,
	Glass beads	Sand	sand	Glass beads	Sand	sand
0.10						
0.15	0.959		0.174	0.117		
0.20	0.916	0.757	0.175	1.115	0.789	0.193
0.24			0.181			0.179
0.28			0.205			0.152
0.30		0.740		0.828		
0.33	0.864	0.662	0.204	0.982	0.979	0.154
0.37						0.154
0.43	0.846	0.729		0.940		0.946

Table 11

Summary of V_p/E_{zp} for Large Particles as a
Function of Column Diameter and Gas Velocity
[Particle Size = 60/80 Mesh for Glass Beads;
Particle Size = 60/80 for Sand (Tetralin)]

Gas velocity (ft/sec)	V_p/E_{zp}					
	$C_s = 6.65-8.295 \text{ lb/ft}^3$			$C_s = 23.66-26.4 \text{ lb/ft}^3$		
	5-in. column		12-in. column,	5-in. column		12-in. column,
	Glass beads	Sand	sand	Glass beads	Sand	sand
0.100	0.541	0.369		0.77	0.216	
0.133			0.128			0.120
0.150	0.573	0.455		0.468	0.466	
0.200	0.604	0.479		0.431	0.605	
0.216			0.118			0.104
0.250	0.641	0.488		0.514	0.584	
0.308			0.115			0.109
0.330	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	0.9961

Table 12

V_p/E_{Zp} for 90 and 70% Glycol in 12-in.
Column Using 60/80-Mesh Sand

Gas velocity (ft/sec)	90% glycol	70% glycol
0.216	0.0216	0.0690
0.392	0.0220	0.0699
0.043	0.0373	0.1100
Viscosity (cP)	13.08	6.2

Table 13

Summary of V_p/E_{Zp} for Large Particles as a
Function of Gas Velocity

[Particle Size = 60/80 Mesh; Liquid = Glycol Mixture, 50% by Wt)

Gas velocity (ft/sec)	12-in. column	
	C_s (lb/ft ³)	V_p/E_{Zp} (ft ⁻¹)
0.05	1.41	0.077
	11.81	
0.20	6.64	0.113
	18.05	0.101
0.40	6.64	0.118
	18.05	0.080

Table 14

Summary of Effects of Liquid Properties on V_p/E_{z_p} for
Large Particles (60/80-Mesh Sand) in a 12-in.-Diameter Column^a

	Water	Tetralin	70% glycol	90% glycol
V_p/E_{z_p} (ft ⁻¹)	0.145-0.195	0.092-0.128	0.069-0.110	0.022-0.037
Viscosity (cP)	1.0	2.7	6.2	13.1

^aSolids concentration, 23-28 lb/ft³; gas velocity, 0.10-0.43 ft/sec.

Variations in the gas velocity up to one order of magnitude did not seem to significantly affect the continuous-flow solids distribution profile. Figures 39 and 40 show representative samples of these results for air/water/tetralin and 50% glycol systems. This result was true for fine as well as large particles. The results using the batch-mode operation showed similar trends in the effects of gas velocity on solids distribution, provided the gas velocity was higher than the minimum value needed to create full suspension. Figures 41-43 show the negligible effect of gas velocity on average solids concentration. Figure 43 shows the concentration normalized with respect to the feed concentration. Normalization was done because different run conditions had different slurry feed rates. The graph again shows that the average solids concentration was independent of gas velocity.

Figures 44 and 45 illustrate the effect of gas velocity on solids distribution in the 6-ft column. The scatter in the data was due mainly to the very small concentrations that were obtained in this column, which can contribute to data uncertainty. The average concentration of the sand fed into the column for most of these runs was between 0.05 and 2.0 lb/ft³. The randomness seen in Figure 44 shows data points on a normalized scale below the value of 1.0, which means that their concentrations are lower than the feed value and are not realistic. In Figure 45, this randomness in the data is subdued, and the agreement between earlier conclusions about the effect of gas velocity on solids profiles seems to be justified. Note that Figure 45 shows the distribution of large and small particles together in the column.

In general, the solids axial profile increased in steepness as the liquid velocity decreased. This was most obvious in the case of the 5-in. column when larger particles were used and the liquid was water.

Figures 46-51 are typical plots of the effects of liquid velocity on the profile. Two trends were noted: (1) the lower the liquid velocity, the steeper the solids distribution profile; (2) the larger the column, the flatter the profile for the same liquid velocity. These trends are not surprising, since lower velocities mean less convective effects and less tendency to obtain a homogeneous mixture. Larger columns create better mixing and tend toward more homogeneous profiles.

Figure 51 shows the decrease of solids accumulation (defined as the average solids concentration in the column divided by the solids concentration in the slurry feed) with an increase in liquid velocity, reflecting the increase of homogeneity of the slurry mixture. Ultimately, at very high liquid velocities, this dimensionless ratio will be asymptotic to 1.0, indicating almost completely uniform mixing and distribution of solids.

The solid axial distribution profiles increase in flatness with an increase in liquid viscosity because of the increased drag on the particles, which decreases their terminal velocity. This is suspected to be the main cause of the decrease in solids accumulation because the liquid axial dispersion coefficient was only mildly affected by the liquid properties, as discussed before. The change is significant for solids--with a change of viscosity from 1.0 to 4.1 cP, the nondimensional solids concentration changed from 6.5 to 2.25, as shown in Figure 52. This implies that a reasonably good knowledge of the viscosity of the solvent and products in the dissolver is essential, having fixed other flow conditions, to determine whether there might be an unreasonably steep solids profile distribution that may cause solids to accumulate in the column.

Increasing the particle size increased the steepness of the solids distribution profile, primarily because of an increase in the terminal velocity of the larger particles. The importance of this effect as it relates to the SRC-I Demonstration Plant is that even with slurry feeds containing a small percentage of larger particles, caution should be taken to ensure that solids do not build up in the bottom of the dissolver. Hence, a solids removal system should be considered, as will be discussed later. Figures 53-55 exemplify the dramatic effect of varying solids distributions of different sized particles with approximately the same slurry feed rate.

The effect of the presence or absence of a distributor was investigated in detail in the 12-in. column. Differences were found to be minimal. Figures 56 and 57 show nondimensionalized concentration plots of the -140- and 60/80-mesh sand, respectively. That there seems to be very little difference is not surprising--as seen before in the 12-in.

column, there was no noticeable difference in the gas holdup between the above-mentioned cases and no noticeable differences in the axial dispersion coefficient (E_{zL}). This is expected to have a strong bearing on the solids axial mixing coefficient and, ultimately, on the solids distribution.

Solids dispersion tests were performed in the 6-ft column using the spargers and the 2-ft target plate. However, the effect of distributors was not studied in the 5-in. or 6-ft columns (distributors being defined as perforated plates spanning the whole cross-section of the column).

An increase in the column diameter decreases the accumulation of solids in general. This is not unexpected, because increasing the diameter size increases the axial dispersion coefficient, which tends to homogenize the slurry mixture concentration. Figures 58-60 show that effect.

The effect of column diameter on the finer particles profile seems negligible, but that for the large particles shows a dramatic change, especially between the 12-in. and 6-ft columns. The slight increase in solids concentration profile steepness between the 12- and 5-in. columns is due to the fact that the feed slurry rates are not exactly the same in both cases.

Solids Dispersion Modeling. Predicting the average concentration in a bubble column is important because this might be one of the factors affecting the chemical reaction of coal in the dissolver (the catalytic effect of solids).

The details of the model derivation are discussed in Ying et al. (1982). The objective here was to determine the solids profile given the feed concentration, the functional value of V_p/E_{zP} , V_1 , and gas holdup (ϵ_g). Using numerical methods, one can then predict an axial solids profile. Whether the model could correctly predict the experimental values is a function of the reliability of the different parameters input to the model. V_p/E_{zP} was obtained as a linearly interpolated function for two values of V_p/E_{zP} obtained at two different average solid concentrations in the batch mode. The model was compared with 40 experimental runs, in the 12-in. column, at different flow conditions for the three different liquids tested (see Appendix A).

Figures 61-63 show that agreement between the model's predictions and experimental data from the 12-in. column was satisfactory. Unfortunately, values of V_p/E_{zp} were not provided for the 6-ft column, which marks a significant scale-up in size from the 1-ft column. The studies in the 6-ft column revealed very flat profiles of solids distribution for 100-200-mesh sand. However, when 30-50-mesh sand was added to the fine particles, the percentage of fine-to-coarse sand was not measured at the different sampling ports. Hence, the concentrations of either size in the feed to the column were unknown. As stated previously, knowledge of the feed concentration is extremely important, because the variation in solid profiles is dependent on the value of the feed concentration, as well as V_p/E_{zp} .

At this point, insufficient information is available to effectively model the solids distribution in the 6-ft column. The profiles seen from previous figures for the distribution of fine particles are much flatter than those for coarser particles. The ratio of maximum to minimum concentration is about 3.0-4.0, for the coarse particles for the 6-ft column, while those for the 12-in. column are as high as 7.0. Thus, it is expected that, with a scaled-up dissolver for the SRC-I plant as large as 11 ft, the profile will be even flatter than that for the 6-ft column. The E_{zL} for the 11-ft dissolver was estimated using correlations at about $17.0 \text{ ft}^2/\text{sec}$, which is more than twofold higher than the 6-ft column. The design of the SRC-I dissolver inlet conditions requires an inlet coal concentration of about 38% by wt. To estimate the total concentration of solids at the bottom of the SRC-I column, the following assumptions were used:

1. Ninety percent of the coal is expected to be dissolved by the time it enters the dissolver. This leaves from the 38% by wt coal ($25 \text{ lb}/\text{ft}^3$) about $2.5 \text{ lb}/\text{ft}^3$.
2. Out of the 10% undissolved solids, 80% is assumed to consist of 100/200-mesh particles and the remaining 20% to be 60/80-mesh.
3. The ratio of solids in the bottom of the 11-ft dissolver to that at the top will be 3:1, similar to that in the 6-ft column at a slurry velocity (V_L) of $0.036 \text{ ft}/\text{sec}$ and a gas velocity (V_g) of $0.36 \text{ ft}/\text{sec}$.

4. A flat solids concentration profile exists for the 100/200-mesh particles axially.
5. The total solids concentration of the different particle sizes is the cumulative sum of the values of the solid profiles of different particle sizes at each axial point along the dissolver. This fact has been shown to be true in Ying et al. (1982).

Hence, using all these assumptions one gets 1.5 lb/ft^3 of large particles (60/80-mesh) at the bottom of the column, which is basically $3 \times 0.5 \text{ lb/ft}^3$ (feed concentration of large undissolved particles). This value is added to the 2.0 lb/ft^3 of 100/200-mesh particles at the bottom, to give a total of 3.5 lb/ft^3 total undissolved solids concentration.

Solids Withdrawal

Solids withdrawal data were obtained in the 5-in., 12-in., and 6-ft columns. Solids withdrawal seems to favor removing large particles rather than the fines. The withdrawal system was operated in the once-through mode for the 5- and 12-in. columns, while that for the 6-ft column was operated in the recycle mode. Hence, the concentration of the profiles was progressively decreasing in the smaller columns, while that of the 6-ft column was allowed to reach steady state by recycling the flow from the withdrawal port back to the column.

Different withdrawal rates were tested in the 5- and 12-in. columns, ranging from 6 to 30% of the slurry feed rate. Increasing the withdrawal rate affected the solids concentration profile significantly. In all these withdrawal tests, profiles for larger particles, with higher terminal velocities, were affected more than the fines. This is not surprising, since the distribution profiles for larger particles, which are usually steeper, have been depleted more than those for fine particles (see Figure 64). It is interesting to note the faster rate of depletion of the 20/30-mesh sand, compared to that of the 60/80-mesh sand for the same time interval after withdrawal started. This occurred because the larger particles have a tendency to settle to the bottom. The weight percentage of solids withdrawn was found to be a function

only of the gas superficial velocity, as shown in Table 15. A variation of slurry velocity between 0.02 and 0.033 ft/sec did not produce any change in the solids concentration withdrawal rate using the 2-ft target plate.

A nozzle has been included, as part of the Baseline Design Package, protruding into the dissolver at an angle to help alleviate any future problems with solids settling. The nozzle is capped at this time but could be used as part of a withdrawal system in case a coarser grade coal is used such as #20 mesh instead of the #100-200 mesh that is called for in the current design. Presently a blowdown system is used to bleed off any excess solids that might occasionally deposit in the dished bottoms. The cold flow tests in the 6 ft column have shown some deposits in the bottom of the dissolver using the #100-200 mesh sand which is heavier than coal (i.e., 2.4:1.4 specific gravity).

SCALE-UP CONSIDERATIONS FOR THE DEMONSTRATION PLANT

The overall aim of the various cold-flow studies was to see how size and flow conditions affect the data needed for scale-up to the demonstration plant size. Some of the factors that affect this scale-up are:

- ° Gas holdup and residence time
- ° Liquid axial dispersion number (Pe, Peclet number)
- ° Gas/liquid mass-transfer coefficient

ICRC's Baseline Design calls for two dissolvers in series, with the following geometry and flow conditions: 11-ft diameter; 67-ft high; estimated gas holdup of 17%; first dissolver, superficial liquid velocity (V_L) = 0.06 ft/sec and superficial gas velocity (V_g) = 0.2 ft/sec; second dissolver, V_L = 0.06 ft/sec and V_g = 0.28 ft/sec.

The increase in the gas velocity in the second dissolver is due to the flow of quench gas introduced in between the dissolvers. The resulting approximate residence times are 16 and 17 min, respectively, in the first and second dissolvers.

Table 15
Solids Withdrawal Data for 6-ft Column

Gas velocity ^a (ft/sec)	Slurry velocity (ft/sec)	Concentration of solids withdrawn (lb/ft ³)
0.10	0.020	92
0.20	0.020	57
0.10	0.033	94
0.20	0.033	55

^a0.10 ft/sec gas through spargers. 3% of slurry feed is withdrawn.

The maximum holdup found in the cold-flow data was about 22% in tetralin/nitrogen in the 12-in. column at $V_g = 0.42$ ft/sec, which is reasonable with the assumptions of 17% gas holdup in the SRC-I Demonstration Plant.

However, as previously mentioned in the section on gas holdup, Exxon (1980) found that gas holdup at process conditions (840°F, 2,000 psi) in its 2-ft EDS pilot plant was as high as 40-45%. This has also been seen in smaller diameter pilot plant dissolvers using the EDS process, as illustrated in Figure 22. This raises some concern as to the applicability and extrapolation of results from the cold-flow data to process conditions at high pressure and temperature. An increase of gas holdup will definitely lower the liquid residence time and will affect the yield structure ultimately.

Tests should be run on two- and three-phase mixtures with simulated gas densities close to that of hydrogen at process conditions to evaluate the effect of gas density on the holdup measurements at elevated pressures. If the gas holdup is around 40% under flow conditions, the residence times would decrease accordingly from 16 and 17 min to 11.56 and 12.28 min. This would probably be serious enough to change the slate of products significantly, because of a shorter contact time between the hydrogen gas and coal slurry.

A convenient way to measure the performance of two reactors in relation to their yield is to quantify the effect of backmixing as opposed to the effect of convective slurry velocity. The nondimensional parameter used is the Peclet number, $Pe = V_L L / E_{ZL}$, where E_{ZL} = liquid axial dispersion (ft²/sec); V_L = superficial liquid velocity (ft/sec); and L = height of dissolver (ft). The relation using the Peclet number to quantify the performance of a certain vessel reactor basically calculates the equivalent number of stirred tanks in series and is given by:

$$\text{no. of tanks} = 0.5 / \left[\left(\frac{1}{Pe} \right) - \left(\frac{1}{Pe} \right)^2 (-e^{-Pe}) \right] \quad (8)$$

The discussion on axial liquid dispersion coefficients, concluded that the correlation for Baird and Rice (1975) describes reasonably well the effect of superficial gas velocity and vessel diameter on the liquid dispersion coefficient. The correlation is as follows:

$$E_{ZL} = 0.350(V_g)(Dg/V_g^2)^{0.33} \quad (9)$$

where E_{ZL} = liquid axial dispersion coefficient (ft²/sec)
 D = vessel diameter (ft)
 V_g = superficial gas velocity (ft/sec)
 g = acceleration of gravity (32.17 ft/sec²)

Extrapolating this correlation to the SRC-I conditions results in Peclet numbers of 3.96 and 4.45 for dissolvers one and two, respectively, and of 1.086 and 1.077 for the equivalent stirred tanks. These numbers are indicative of a system that is well backmixed.

Backmixing is desired to prevent severe axial temperature gradients in the dissolver, as is the case with plug flow, because the reaction is highly exothermic.

The correlation used here is a function of gas velocity and gas holdup, and was developed at ambient conditions. At this stage, the specific effect of increased gas holdup on the dispersion number for the same average superficial gas velocity is not known. However, intuitively the trend is to effect an increase in the dispersion coefficient with an increase in the holdup, primarily because of an increase in the bubble population and a probable decrease in bubble size. The overall effect is to decrease the number of equivalent stirred tanks in series.

The gas/liquid mass-transfer rate $k_L a$ was not found to be the limiting process in the SRC-I Demonstration Plant dissolver. This is based on mass-transfer measurements in the 6-ft column which indicate that the case with the highest transfer rate, i.e., zero concentration in bulk fluid, is about ten times higher than the design mass-transfer value of the actual dissolver, i.e., 230 hr⁻¹ (under process conditions). The effect of a possible increase in the gas holdup under high pressure will increase the value of $k_L a$, as it is proportional to $\epsilon_g^{1.1}$. The cold-flow tests have shown, importantly, that in scale-up to the demonstration plant, low mass-transfer rates do not seem to be a limiting factor.

Gas velocity did not affect the solids distribution profile at values higher than the minimum velocity required for complete suspen-

sion. The pronounced effect of this critical velocity was shown in the case of air/water experiments to be about 0.2 ft/sec, because water has a lower viscosity than tetralin or glycol. (Critical velocity is defined as the minimum superficial gas velocity required for a given average solids concentration, to achieve full suspension of these solids.) The design dictates gas velocities of 0.2 and 0.28 ft/sec (in the first and second dissolvers, respectively).

Increasing the slurry velocity flattens out the solids concentration profile. As discussed previously (in the section on solids dispersion), the worst possible solids concentration at the bottom of the column, assuming 2.5 lb/ft³ of solids is fed into the column, is 3.5 lb/ft³, which includes the fine and coarse particles. The analogy uses the profile of the 6-ft column and a liquid velocity of 0.03 ft/sec. Knowing that the design velocity of the SRC-I slurry into the dissolver is 0.06 ft/sec and that the dissolver has a larger diameter and thus a higher axial dispersion coefficient, one expects the value of 3.5 lb/ft³ to be a worst-case situation.

Hence, the trend in increasing the slurry velocity is to help homogenize the slurry mixture, as previously concluded. However, at turndown velocities, with a decrease of gas and slurry flow rates to about 50% of their rated values, the tendency would be to decrease the solids axial dispersion coefficient and the slurry velocity, which increases the steepness of the profile of the solids inside the dissolver. This then further indicates the need for a solids blowdown system to help decrease the possibility of settled solids. Because the Baseline dissolver does not include internals, the solids will probably settle around the dished bottom inlet, as this area is rather flat and is a "dead region," characterized by a recirculation zone of the slurry. This flow pattern is characteristic of a jet coming into a sudden expansion. Perhaps the withdrawal outlets for this blowdown system could be placed diametrically opposite to each other with respect to the bottom inlet, which would allow slurry to be withdrawn by pressure differential to a point downstream of the dissolvers.

Another alternative provided from an engineering point of view is the fact that in the actual case most of the coal particles would be

dissolved by the time they reach the dissolver. Only a small percentage of inorganic matter will stay in a solid state and may settle in the bottom. Purging or blowdown will then be performed after shutdown at regular intervals.

Because the mineral solids in the coal slurry seem to have a catalytic effect on the conversion of asphaltenes from preasphaltenes and since hydrogen consumption increases with an increase in reactor solids (Skinner, 1983), a reasonable concentration of these solids is desirable in the dissolver. The previous discussion on solids axial distribution indicates that this aim can be achieved. The solids profile is expected to be reasonably homogeneous, with the highest concentration of 3.5 lb/ft³ at the bottom of the column and an exit concentration of 2.5 lb/ft³, as required in the design.

However, turndown velocities may affect preasphaltene to asphaltene conversions because the concentration in the bottom of the dissolver is much higher than that at the top. This will give a nonhomogeneous solids distribution.

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