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DOE/OR/03054-26-Suppl.
(DE84015097)

SRC-I QUARTERLY TECHNICAL REPORT

Supplement for the Period April—June 1983

June 1984

Work Performed Under Contract No. AC05-78OR03054

International Coal Refining Company
Allentown, Pennsylvania

Technical Information Center
Office of Scientific and Technical Information
United States Department of Energy

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DOE/OR/03054-26-Suppl.
(DE84015097)
Distribution Category UC-89

SRC-I
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EFFECTS OF STAGED VESSELS ON
DISSOLVER PERFORMANCE

R. Sivasubramanian* and E. N. Givens*

International Coal Refining Company (ICRC) has been conducting an ongoing research project entitled "Effects of Accumulated Solids and Staged Vessels on Dissolver Performance" (Program 12.1.7). The project's overall objective is to provide information required by the SRC-I Demonstration Plant team for the design of an optimized dissolver configuration.

This report presents the results of Task 3 of that project--"Inter-stage Conversion for Series Dissolvers." Task 3 was designed to:

- ° Determine conversion, yield, and hydrogen consumption after the first dissolver in a series operation.
- ° Expand the data base to both 20- and 60-min residence times.
- ° Develop process data for operating two dissolvers in series at staged temperatures in a coal liquefaction plant.
- ° Show the effect of operating at low hydrogen feed rates.

A process run was conducted in ICRC's coal process development unit (CPDU) during June and July 1982. Results from operating with Kentucky #9 Mulford coal at residence times from 20 to 60 min and temperatures from 780 to 840°F showed that oil yields were directly affected by reaction time, but were relatively insensitive to temperature. Both hydrocarbon gas yield and hydrogen consumption increased with temper-

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ature and residence times, whereas conversions to pyridine solubles were largely unaffected and preasphaltene yields decreased. Qualitatively, the data support a series reaction mechanism, although additional data are needed to confirm this.

Data on Mulford coal generated in series reactors under staged-temperature conditions, in which the first reactor was operated either at 780 or 810°F and the second at 840°F, agree with results observed previously under similar conditions on Lafayette coal. When the first reactor was operated at a lower temperature, the oil yields improved, compared to when both reactors operated at the same temperature. The hydrocarbon gas yields and hydrogen consumption were lower in the staged-temperature mode than in an isothermal mode.

Reducing the hydrogen feed rate to the first dissolver by partitioning the total hydrogen feed between the front end of the reaction train and the second dissolver may have a detrimental effect on product yields. Data at 20-min residence times, with a 50% hydrogen feed rate (15,000 scf of hydrogen per ton of coal), showed a 5% decrease in oil yield and a 2% decrease in pyridine conversion. If the reduced hydrogen feed rate allows the retrograde reactions to increase, this may result in an undesirable product slate.

INTRODUCTION

The 6,000-tpd SRC-I Demonstration Plant is designed to operate with two dissolvers. Direct scale-up of the SRC-I process from pilot plant operation at the Advanced Coal Liquefaction Facility in Wilsonville, Alabama and at Ft. Lewis, Washington implies the use of two vessels in parallel, since the pilot facilities use a single dissolver vessel. The use of multiple vessels in parallel offers some obvious advantages: more reliability, lower gas and liquid velocities, and higher solids loading. However, the option of having two vessels in series, in a single 6,000-tpd train, allows the independent control of many operating variables, in a manner that has not been addressed in previous SRC-I research. These variables can be controlled to optimize overall process yields.

Program 12.1.7 was designed to generate data in ICRC's CPDU that could be used to make a decision regarding dissolver configuration. Data developed earlier, under Task 2 of this program, showed that using dissolvers in series will favor high preasphaltene conversion and lower levels of insoluble organic matter (IOM), which will have an impact downstream of the dissolver (Sivasubramanian, 1982). Both of these advantages will help ensure that the critical solvent deashing (CSD) step in the SRC-I plant achieves maximum SRC recovery. In order for CSD to be most effective, the preasphaltene content of the feed to the CSD unit must be carefully controlled.

Other data from the staged-dissolver program of Task 2 showed that running the first and second dissolvers at staged temperatures (810 and 840°F, respectively) was definitely advantageous. Oil yields were higher with the staged-temperature sequence, in contrast to yields when one or both reactors were run at 840°F. Likewise, the staged-temperature runs also resulted in increased coal conversion. This kind of process advantage, due only to use of different temperatures in the two stages, will undoubtedly improve the process economics.

Lafayette coal was used in Task 2; however, it is one of the least reactive coals from the Western Kentucky basin. Because highly reactive Kentucky #9 coal such as that from the Mulford mine will most likely be chosen for the SRC-I Demonstration Plant, it was used in Task 3 to generate data that could be compared with the Task 2 data.

EXPERIMENTAL PLAN FOR TASK 3

Coal was processed through a single dissolver under the following conditions:

- 40% Kentucky #9 coal in Ft. Lewis process solvent
- 780, 810, and 840°F
- 20- and 40-min residence times
- 15,000 and 30,000 scf of hydrogen/ton of coal
- 2,000-psig total pressure

Further runs were conducted at the same pressure and slurry concentration and at 30,000 scf of hydrogen/ton of coal in two series dissolvers, at the following conditions:

Time in each dissolver (min)	Temp (°F) in dissolver	
	1st	2nd
20	780	840
20	810	840
20	840	840
30	780	780
30	810	810
30	840	840

At each condition, conversion, hydrogen consumption, process yields, and sulfur in SRC were determined.

Feed Materials

Coal. Washed Kentucky #9 Mulford coal was obtained directly from a coal preparation plant associated with the mine. The coal sample was ground to 95% minus 200-mesh particles and air-dried. It was screened through a 150-mesh sieve prior to use. Proximate and ultimate analyses are provided in Table 1.

Solvent. The feed solvent was a Ft. Lewis recycle solvent produced under SRC-I conditions. Table 2 lists analytical data. When fractionated into separate cuts by solvent separation, the solvent contained 96.2% oils and 3.8% asphaltenes. The hydrogen content was 8.2%.

Operation

The feed slurry (40 wt % Kentucky #9 coal and 60 wt % Ft. Lewis solvent) was prepared at room temperature in 40- to 60-kg batches immediately before use, and stored in a charge tank equipped with a turbine agitator and a recycle pump, to maintain a uniform suspension of the solids.

The feed slurry was pumped to the continuous stirred-tank reactor (CSTR) from a feed tank maintained at ambient temperature. The feed tank, equipped with a recycle pump to mix the slurry, was refilled each hour from the charge tank. Whenever the feed slurry composition was changed, the feed tank was drained and flushed several times with the new feed slurry. When reaction conditions were altered, at least 10 reactor volumes of slurry were processed through the reactor at the new conditions prior to collection of any product samples for analysis.

A continuous type of reactor was selected for these experiments so that the vapor composition in the reactor, including the partial pressure of light solvent components, would match that in an SRC-I dissolver. The stirred autoclave design of the CSTR ensures that the solids are well-mixed and do not accumulate in the reactor. Cold-flow experiments, conducted in a Plexiglas model, have confirmed that the flat-bladed turbine agitator of the CSTR keeps the solids well distributed throughout the reactor volume, so that the solids will not accumulate (Ying, 1982).

Because of their tendency to promote coking, baffles were not used in the CSTR. However, the thermowell reduces the size of the vortex, with a void fraction of 0.18. Because all sensible heat needed to bring the slurry to reaction temperature is provided by resistance heating of the reactor, the reactor wall is about 25°F hotter than the bulk slurry.

The product gas and slurry leaving the CSTR are quenched to 325°F before flowing to a gas/liquid separator at reactor pressure. The separator effluent slurry is throttled into the 40-psig product receiver at a rate that maintains a constant, small inventory in the separator.

Water, light hydrocarbons, and gases, which flash from the product slurry in the 40-psig receiver, are combined with the product gas stream from the separator. The combined gas stream is cooled to about 60°F to condense the water and light organics before it enters the gas chromatograph and dry test meter for measurement of product gas composition and volume.

Feed and Product Analysis

Two sets of product samples were withdrawn at each operating condition. This was done to ensure that the actual operating conditions

during sampling were close to the planned operating conditions. One set of product samples was analyzed further.

The feed and product slurries were solvent-separated into fractions: pentane-soluble material (oils), pentane-insoluble and benzene-soluble material (asphaltenes), benzene-insoluble and pyridine-soluble material (preasphaltenes), and pyridine-insoluble material. The latter contains insoluble organic matter (IOM) and mineral residue. It is important to note that the fractions from solvent separation are defined operationally, not chemically. The end points of each fraction often vary because of variations in the individual samples themselves, and also because of minute changes in details of the solvent separation procedures.

The experimental design called for analyzing each product sample twice, but only a few samples were actually analyzed because of time constraints. However, each fraction was further subjected to detailed elemental analysis. Product gases were analyzed by on-line gas chromatography for hydrocarbon gases through C_4 , and also for other nonhydrocarbon gases (H_2S , NH_3 , CO , CO_2 , etc.). The product water was determined indirectly by assuming that the water vapor exiting the process unit was in equilibrium with the condensed water.

Products sampled from the process unit included gases, light condensate, and the main receiver product slurry. The light condensate was a mixture of water and oil, the water making up about 80% of the mixture. The quantity of oil was extremely small based on the total main receiver product. Two of the organic condensate samples were subjected to elemental analysis, and the average of the two was used as the elemental analysis of the light oil condensate for this run.

RESULTS AND DISCUSSION

Effect of Time and Temperature

Process data were developed at 20-, 40-, and 60-min residence times at three temperatures, 780, 810, and 840°F. The 20- and 40-min data were taken in a single CSTR, and the 60-min data were obtained using two identical CSTRs in series. The effect that two series reactors have on

the yield distribution was taken into consideration in comparing the 60-min data to the 20- and 40-min data. Our results from Task 2 had already quantified the impact of operating two reactors in series (Sivasubramanian, 1982); the only effect was a change in preasphaltene concentration.

The earlier work during Task 2 had also shown that multiple analyses of product samples are needed to clearly distinguish the differences in yield structures at different operating conditions. Therefore, to produce statistically significant data, the experimental design for this task called for at least duplicate analyses of each product sample. However, the reduced scope of the original program cut down the time available for duplicate analyses such that only five samples were duplicated. Those samples had been completed at the time of the reduction in scope. From that point on, samples were analyzed only once.

The duplicate or multiple analyses from Task 2 were used to calculate variances for each individual fraction measured by solvent separation (oil, asphaltene, preasphaltene, and residue) by using a statistical technique known as pooled variance. These variances can be used to determine the statistically permissible maximum differences between two analyses of total product liquid. Then, knowing moisture and ash content of the feed coal, one can calculate the statistically permissible maximum differences in yield structure based on moisture-and-ash-free (MAF) coal yields. The maximum differences calculated during Task 2 for Lafayette coal were presented in an earlier report (Sivasubramanian, 1982). Since duplicate analyses were available for five samples generated from Mulford coal, new values for the variances (s^2) for individual fractions in the product analysis were calculated. Results indicated that the variances for the individual fractions were essentially similar to the earlier analysis on Lafayette coal. Hence, the values of the variances from Task 2 were also used here; however, since Mulford coal has a different moisture and ash content compared to Lafayette coal, new values were calculated for statistically permissible maximum differences in yield structure based on MAF coal (see Table 3). The following discussion on yield structures is based on these adjusted values.

The data for 20-, 40-, and 60-min reaction periods are listed in Tables 4, 5, and 6, respectively. Generally, conversion of coal to pyridine-soluble product is insensitive to residence time or temperature, except at a residence time of 60 min. The data for 60-min residence indicate a slight increase in conversion at the lower temperatures (780 and 810°F), which could be due to the use of two reactors in series. This phenomenon was not, however, observed previously when series data were developed at a residence time of about 30 min.

Figures 1 and 2 show the effect of reaction time and temperature on oil yields. Both figures reveal that the data point at 780°F and 40 min is inconsistent with the rest of the data. If that data point is ignored, Figure 1 clearly shows that oil yield increases with an increase in residence time, and Figure 2 shows oil yield is insensitive to temperature changes from 780 to 840°F at all three residence times studied.

The data on hydrocarbon gas yield and hydrogen consumption are internally consistent. Figure 3 shows that both higher temperatures (810 and 840°F) and longer residence times favor higher hydrocarbon gas yields. The data at 780°F are so closely bunched that a clear temperature pattern is difficult to discern.

Hydrogen consumption behaves similarly. Both higher temperature and longer reaction times favor increased hydrogen consumption (Figure 4). The 20-min data may be a little less sensitive to temperature than the longer residence times, possibly because solvent supplies almost all of the hydrogen demand during the early reaction periods.

Figures 5 and 6 show the effect of time and temperature on the yield of asphaltenes and preasphaltenes, respectively. Figure 6 shows that the yield of preasphaltenes decreases with an increase in reaction time. Also, the yield seems to decrease with an increase in temperature. Quantitatively, the variations in asphaltene yield with time and temperature are within the statistically permissible maximum differences. Qualitatively, the following trends were observed. At 780°F, increasing residence time seems to have no effect on asphaltene yield, but at 810 and 840°F the yield appears to decrease. Figure 5 clearly shows that the yield of asphaltenes does not increase with an increase in residence time.

In addition to a study of the effect of time and temperature on process yields, another major objective of this task was to generate enough information to decide whether a series or parallel model represents the data better. Because the uncertainties associated with the data are large due to the lack of multiple analyses, only a qualitative discussion can be presented here. Attempts were made to generate reaction rate constants and activation energies; these were also dropped due to the absence of acceptable uncertainty analysis.

Figure 7 illustrates the two reaction pathway models. The different implications of the models can best be appreciated by considering the yield structures they predict. Figures 8 and 9 present the yield structure predictions as a function of residence time for the two models. The predictions are based upon kinetic constants regressed from other data sets and are not intended to be quantitatively applicable to the current runs or Mulford coal. However, these predictions are useful for qualitatively describing the different trends that the two models imply.

Both models predict the same behavior for unreacted coal (IOM) and preasphaltene yields. The unreacted coal decreases rapidly with residence time at shorter residence times and then approaches an asymptote, while preasphaltene decreases with time and approaches its asymptote only at long residence times. The series model predicts that both asphaltene and distillate (oil) yields will go through a maximum and then decrease. Distillate reaches its maximum as it cracks to form hydrocarbon gas, and the rate of distillate production decreases due to the reduced concentration of asphaltenes at extended residence times. Note, however, that the region of maximum distillate yield can be quite broad, as it is in this case. The series model also predicts that hydrocarbon gas will increase linearly with residence. In contrast, the parallel model predicts that asphaltene, distillate, and hydrocarbon gas yields will asymptotically approach a maximum at long residence times.

Thus, the main qualitative differences between the models are that (1) the series model predicts a linearly increasing hydrocarbon gas yield, while the parallel model predicts a relatively constant and much

lower level of gas production, and (2) the series model indicates asphaltene yield reaches a maximum and then decreases, whereas the parallel model implies that asphaltene yield increases with residence time.

Figure 3 clearly shows that hydrocarbon gas yield increases almost linearly with time. While Figure 5 does not show that asphaltene passes through a maximum, nor even that asphaltene decreases, which would be predicted if the maximum asphaltene yield occurred before the 20-min residence time, the data clearly do not support a model that predicts increasing levels of asphaltene. Hence, the data seem to support the series model more than the parallel model. Additional data with multiple analyses are needed to confirm this trend.

Effect of Series Dissolvers

The Task 2 study on the effect of series dissolver operation on product yields indicated that operating the dissolvers in series decreased the preasphaltenes yield. However, conversion as well as oil, asphaltene, and gas yields were unaffected by reactor configuration. The earlier data were obtained from runs with a less reactive coal (Lafayette Kentucky #9), although a single data point was obtained using a highly reactive coal (Mulford) at 840°F and 40 min using the two different reactor configurations (series and parallel).

Table 7 shows that no noticeable differences exist between the yield structures obtained from two different reactor configurations. However, these data neither prove nor disprove the earlier conclusion. If anything, they support the conclusion that the yields of fractions other than preasphaltenes were unaffected by reactor configuration. Even in the earlier run, the data at 840°F showed that the decrease in preasphaltenes was barely outside the statistically permissible maximum differences. The earlier study was conducted at a residence time of 30 min, whereas this study was conducted at a residence time of 40 min. These data show the importance of conducting studies at multiple temperatures and residence times using different coals so that a definite trend can be established.

Effect of Staged Temperatures

One advantage of operating with both dissolvers in series is that the temperatures in each of the two dissolvers can be varied. In a previous run (SCL-51) under Task 2 of this program, we found an advantage for staging the temperatures in the dissolver. The operating conditions for Run SCL-51 are shown in Table 8 and results are in Table 9.

We have reexamined the effect of staged temperatures in a run with Mulford coal at a 40-min total residence time, which was equally divided between the two reactors. In this run, we also lowered the first reactor temperature to 700°F to study whether any advantages exist in operating the first dissolver at a very low temperature. The results are presented in Table 10.

Clearly, lowering the temperature in the first reactor increases the oil yield. The 840/840 oil yield is lower than the 780/840 and 810/840 data, although the difference is smaller than noted previously (see Table 7). This difference could be due to the more reactive coal or the differences in residence time (40 vs. 30 min).

However, the data do show a definite advantage in staging the temperatures in the series dissolvers. The data also indicate that the optimum temperature for the first dissolver should be above 700°F. As can be seen from Table 10, operating the first dissolver at 700°F lowered the oil yield, even though hydrogen consumption and conversion were identical to the data at 780°F. Apparently, the conversion of asphaltene to oils is low at this temperature.

The hydrocarbon gas yields measured after 20- and 40-min residence times (Tables 4 and 10, respectively) are consistently additive. For example, 20 min at 780°F gives 2.18% hydrocarbon gases, while 20 min at 840°F gives 6.68%. The staged run of 780/840°F for a total of 40 min gave 7.82%, vs. an additive number of 8.86%. Similar comparisons can be made for the other combination. After 20 min at 810°F, 4.57% hydrocarbon gases were formed, vs. 6.68% at 840°F. The 810/840°F run gave 9.55% vs. the additive number of 11.25%.

The increased oil yields and reduced hydrocarbon gas yields noted with staged temperatures both here and in Run SCL-51 (Table 7) provide

confidence that a definite advantage exists for operating with staged reactors that is not simply related to reactor mixing.

Segregation of Hydrogen Flow between Dissolvers

The data at 840/840°F shown in Table 10 simulate the total result of feeding all of the hydrogen through both reactors. However, data were also generated during Task 3 to show the effect of partitioning the hydrogen feed between the first and second dissolvers. We examined the effluent from the first reactor after only half of the hydrogen had been fed (Table 11), which is consistent with the Baseline design for demonstration plant dissolver operation.

At a decreased hydrogen flow to the first reactor, namely, 14,700 scf/ton of coal, the total oil yield (lights and oils) dropped from 14.0 to 9.0%. Even though this may not be statistically significant based on a single analysis, the liquid oil yield alone dropped from 12.8 to 4.9 wt %, which is significant. Also, note that the statistical analysis on oil yields applied mainly to the liquid oil obtained from product analysis. The yields of lights reported here are generated from the light materials trapped in the condenser. The corresponding hydrocarbon gas yield did not change significantly (from 6.7 to 7.4%). The decrease in gas flow rate changed the hydrogen partial pressure from 1,770 to 1,430 psig. The flow rate change will also change the residence time, due to vaporization of light materials. Both of these changes may have affected the yield of oils. Generally, these data show that staging hydrogen addition is detrimental. How significant this would be after going through the second reactor at full hydrogen pressure is unknown at this time.

CONCLUSIONS

Process data obtained at residence times from 20 to 60 min and temperatures from 780 to 840°F show that oil yields are directly affected by reaction time, but relatively insensitive to temperature. Hydrocarbon gas yield and hydrogen consumption parallel each other, in that both increase with temperature, especially above 800°F, and both

also increase with residence times, also at temperatures above 800°F. Coal conversion (to pyridine solubles), on the other hand, is not particularly affected by temperature or residence time in a regular manner. Preasphaltene yields decreased with an increase in temperature and residence time. Qualitatively, the data tend to support a series reaction mechanism. Additional data are needed to confirm this.

New runs on Mulford Kentucky #9 coal showed that lowering the temperature in the first of two reactors in series increased the oil yield. When the first reactor is at 780 or 810°F and the second at 840°F, the oil yield was about 2.5% higher and the hydrocarbon gas yield was lower than when operating at isothermal conditions with both reactors at 840°F.

An operation where fresh hydrogen flow would be split between the front end of the reaction train and the second dissolver was simulated by examining 20-min reaction times at half the normal hydrogen feed rates. The oil yield after the first reactor dropped by 5%, and conversion also appeared to decrease by 2%. If this lower hydrogen feed rate and presumably lower hydrogen partial pressure allow the retrograde reaction to accelerate, significant problems may occur. This observation strongly supports the need for additional data to verify whether this effect will occur.

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Table 1

Proximate and Ultimate Analysis of
Kentucky #9 Mulford Coal

		Proximate analysis (wt %)
	Moisture	2.15
	Volatile matter	36.91
	Fixed carbon	61.38
	Ash	9.48
		Ultimate analysis (wt %)
		Moisture- and ash-free basis
	Moisture-free basis	
Carbon	72.32	81.26
Hydrogen	5.19	5.83
Oxygen	8.01	9.00
Nitrogen	1.77	1.99
Sulfur	2.85	1.92
Organic	1.71	1.92
Pyritic	1.14	
Ash	9.68	

Table 2
Process Solvent Data

Solvent fractionation (wt %)	
Oils	96.18
Asphaltenes	3.82
Preasphaltenes	Nil

Elemental analysis (wt %)	
Carbon	87.83
Hydrogen	8.21
Nitrogen	0.85
Sulfur	0.35
Oxygen	2.92

Table 3
Statistically Permissible Maximum Differences
(95% Confidence Level, Based on MAF Yields)

Fraction	Difference (wt %)	
	1 analysis	2 analyses
Oils	7.32	5.19
Asphaltenes	9.34	6.61
Preasphaltenes	4.25	3.01
Residue	6.91	4.89

Table 4
Yield Data

Pressure: 2,000 psig
H₂ Feed Rate: 28,000 scf/ton of Coal
Coal: Mulford Kentucky #9
Solvent: Ft. Lewis Recycle Solvent

	<u>Temp (°F) and residence time (min)</u>		
	780	810	840
	20.5	20.8	20.5
Conversion (wt %)	85.6	86.96	87.38
H ₂ consumption, total (wt %)	0.78	1.07	1.20
From gas	0.60	0.89	0.89
From solvent	0.18	0.18	0.32
Yields (wt % MAF coal)			
Hydrocarbons	2.18	4.57	6.68
Other gases	1.88	3.18	3.14
Lights	1.19	0.69	1.22
Oils	15.80	14.31	12.81
Asphaltenes	29.44	30.04	31.51
Preasphaltenes	34.81	31.03	29.74
Residue	15.47	14.23	14.09
Water	0.32	3.14	2.27
S in SRC (wt %)	1.04	0.93	0.77
Solvent hydrogen (wt %)			
Start	8.21	8.21	8.21
Finish	8.11	8.10	8.03

Table 5
Yield Data

Pressure: 2,000 psig
H₂ Feed Rate: 28,000 scf/ton of Coal
Coal: Mulford Kentucky #9
Solvent: Ft. Lewis Recycle Solvent

	Temp (°F) and residence time (min)		
	780	810	840
	40.5	40.05	40.3
Conversion (wt %)	86.03	88.53	87.4
H ₂ consumption, total (wt %)	0.73	1.59	2.04
From gas	0.88	1.37	1.39
From solvent	-0.15	0.23	0.65
Yields (wt % MAF coal)			
Hydrocarbons	3.82	7.27	11.17
Other gases	4.19	3.56	4.08
Lights	0.94	1.54	2.19
Oils	13.86	17.56	18.86
Asphaltenes	29.56	29.91	20.80
Preasphaltenes	31.56	24.63	26.62
Residue	15.00	13.04	15.06
Water	2.03	4.07	3.65
S in SRC (wt %)	1.09	0.81	0.72
Solvent hydrogen (wt %)			
Start	8.21	8.21	8.21
Finish	8.30	8.08	7.84

Table 6
Yield Data

Pressure: 2,000 psig
H₂ Feed Rate: 28,000 scf/ton of Coal
Coal: Mulford Kentucky #9
Solvent: Ft. Lewis Recycle Solvent

	Temp (°F) and residence time (min)		
	780/780	810/810	840/840
	61.56	61.25	62.40
Conversion (wt %)	90.63	91.00	87.11
H ₂ consumption, total (wt %)	1.15	1.88	2.73
From gas	1.13	1.66	2.19
From solvent	0.02	0.22	0.54
Yields (wt % MAF coal)			
Hydrocarbons	3.66	8.97	15.30
Other gases	3.34	3.99	4.38
Lights	1.23	1.07	3.28
Oils	23.59	25.44	18.82
Asphaltenes	30.11	25.99	23.56
Preasphaltenes	25.87	24.32	18.45
Residue	11.09	11.09	15.64
Water	2.85	1.22	3.32
S in SRC (wt %)	0.91	0.65	0.51
Solvent hydrogen (wt %)			
Start	8.21	8.21	8.21
Finish	8.20	8.08	7.90

Table 7
Effect of Series Dissolvers

Pressure: 2,000 psig
 H_2 Feed Rate: 28,000 scf/ton of Coal
 Coal: Mulford Kentucky #9
 Solvent: Ft. Lewis Recycle Solvent
 Temperature: 840°F

	1 reactor	2 reactors
Residence time (min)	40.3	41.9
Conversion (wt %)	87.4	88.6
H_2 consumption (wt %)	2.04	2.05
Yields (wt % MAF coal)		
Hydrocarbons	11.17	11.48
Other gases	4.08	3.79
Oils	21.05	20.01
Asphaltenes	20.8	25.16
Preasphaltenes	26.6	24.98
Residue	15.04	13.44
Water	3.65	3.21
S in SRC (wt %)	0.72	0.61

Table 8

Operating Conditions for

Run No. SCL-51

Pressure: 2,000 psig

 H_2 Flow: 28,000 scf/ton of CoalFeed Composition: 40% Lafayette Kentucky #9 Coal,
60% Recycle Solvent

	Reactor I		Reactor II	
	Time (min)	Temp (°F)	Time (min)	Temp (°F)
I	13.3	780	15.1	840
II	12.1	810	13.7	840
III	12.5	840	14.2	810

Table 9

Effect of Staged Temperatures

Run: SCL-51
 Coal: Lafayette Kentucky #9
 Pressure: 2,000 psig
 Feed Solvent: 94% Oils

	wt % yields at temp (°F) and time (min)			
	780/840 13.3/15.1	810/840 12.1/13.7	840/840 12.4/14.1	840/810 12.5/14.2
Hydrocarbons	5.0	5.6	7.0	5.4
Other gases	1.7	2.2	2.5	2.0
Oils	12.1	22.7	17.2	19.6
Asphaltenes	23.3	23.0	24.0	22.3
Preasphaltenes	38.9	31.6	30.2	33.5
IOM	13.5	12.4	16.7	14.8
H ₂ consumption	1.42	1.74	1.55	1.37

Table 10

Effect of Staged Temperatures

Pressure: 2,000 psig
 H_2 Feed Rate: 28,000 scf/ton of Coal
 Coal: Mulford Kentucky #9
 Solvent: Ft. Lewis Recycle Solvent

	wt % yields at temp (°F) and time (min)			
	700/840	780/840	810/840	840/840
	41.12	41.07	41.37	41.09
Conversion (wt %)	89.18	89.6	89.4	88.6
H_2 consumption, total (wt %)	1.40	1.33	1.88	2.05
From gas	0.98	0.65	1.20	1.54
From solvent	0.42	0.68	0.67	0.52
Yields (wt % MAF coal)				
Hydrocarbons	7.25	7.82	9.55	11.48
Other gases	3.7	3.45	4.00	3.79
Lights	1.09	1.25	1.52	2.09
Oils	14.08	20.81	20.52	17.92
Asphaltenes	32.17	26.68	26.99	25.16
Preasphaltenes	27.39	28.71	23.24	24.98
Residue	12.49	12.10	12.76	13.44
Water	3.49	0.87	3.58	3.21
S in SRC (wt %)	0.84	0.73	0.67	0.61
Solvent hydrogen (wt %)				
Start	8.21	8.21	8.21	8.21
Finish	7.97	7.82	7.82	7.91

Table 11
Effect of Staged Hydrogen Addition

Pressure: 2,000 psig
Coal: Mulford Kentucky #9
Solvent: Ft. Lewis Recycle Solvent

	Gas rate (M scf/ton), temp (°F), and residence time (min)	
	29.4, 840, 20.5	14.7, 840, 20.62
Conversion (wt %)	87.38	85.46
H ₂ consumption, total (wt %)	1.20	1.19
From gas	0.89	1.00
From solvent	0.32	0.19
Yields (wt % MAF coal)		
Hydrocarbons	6.68	7.41
Other gases	3.14	3.54
Lights	1.22	4.15
Oils	12.81	4.89
Asphaltenes	31.51	27.52
Preasphaltenes	29.74	34.74
Residue	14.09	15.83
Water	2.27	3.21
S in SRC (wt %)	0.77	0.96
Solvent hydrogen (wt %)		
Start	8.21	8.21
Finish	8.03	8.10

Figure 1
Effect of Residence Time on Oil Yield

Figure 2
Effect of Temperature on Oil Yield

Figure 3
Effect of Temperature on Hydrocarbon Gas Yield

Figure 4
Effect of Temperature on Hydrogen Consumption

Figure 5
Effect of Time and Temperature on
Asphaltene Yield

Figure 6
Effect of Time and Temperature on
Preasphaltene Yield

Figure 7
Comparison of Series and Parallel Models

Series Model

Parallel Model

Figure 8

CPDU - SERIES MODEL PREDICTIONS FOR 840°F

Figure 9

CPDU - PARALLEL MODEL PREDICTIONS FOR 840°F

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LARGE-SCALE DISSOLVER COLD-FLOW MODELLING
FINAL REPORT

W. T. McDermott* and D. H. S. Ying*

APCI has completed studies directed specifically at providing fluid dynamic data for the design of the SRC-I Demonstration Plant dissolvers. Studies on gas/liquid contact for the air/water (two-stage) system were completed. Batch (zero liquid velocity) studies of gas holdup, mass transfer coefficient, and approximate effective bubble size were completed for each internal tested. Gas holdup studies with liquid flow were also completed for the cases of no internals and of gas spargers combined with the 2-ft-diam target plate. Finally, liquid axial dispersion studies were completed for the gas sparger/2-ft target plate configuration under batch conditions.

The results indicate an increase in the measured gas holdup for those instances in which gas flow was supplied by the sparger tubes. For all other internal configurations, the measured holdup was lower. Similarly, the measured volumetric mass transfer coefficient increased only in cases in which gas flow was supplied by the sparger tubes. The measured mass transfer coefficients were lower for all the other internal configurations. The measured liquid axial dispersion coefficients obtained for the spargers/2-ft plate configuration were an order of magnitude larger than those measured in a 1-ft-diam column in a separate research program. Bubbles were approximately 1/4-in. in effective diameter, and were accompanied by larger bubbles frequently approaching inlet diameter size. The smaller bubbles were invariant in size among the internal geometries tested; only the larger bubbles were affected by the internals.

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Gas/liquid contact studies in the air/water/sand (three-stage) system were completed for the gas spargers/2-ft-target plate configuration. Batch studies of gas holdup, mass transfer coefficient, approximate effective bubble size, axial solids dispersion, and liquid axial dispersion were performed. Sand particle sizes tested included 100-200 mesh and a mixture of 30-50 and 100-200 mesh.

The results indicate good dispersion with approximately uniform concentration of the 100-200-mesh-sand solid phase within the vessel, although sedimentation on the vessel bottom and in the horizontal inlet line was observed. A greater solids concentration gradient was present with the addition of 30-50-mesh sand to the system. Gas holdup values for the three-phase system were slightly lower than for the corresponding two-phase system values. However, measured volumetric mass transfer coefficients were not affected by the presence of solid particles.

INTRODUCTION

The overall objective of this project was to study gas/liquid mass transfer rate and solids removal in a cold-flow tubular column to provide data for the coal dissolver design of the SRC-I Demonstration Plant.

The specific objectives were:

- ° To study gas/liquid mass transfer rate for several internal designs using a two-phase system (air/water).
- ° To study the effect of solids on gas/liquid mass transfer rate.
- ° To test the design of a solids withdrawal system.

The gas/liquid mass transfer coefficient, $k_L a$, should be examined in an industrial-size dissolver column. Results from the Wilsonville Pilot Plant show that chemical reaction and not gas/liquid mass transfer is the rate-limiting step in hydrogen consumption. From the results of

this program, ICRC has found a correlation in the literature for a two-phase system. Predictions from the correlation indicate that the demonstration plant dissolver should not be gas/liquid mass transfer limited, although substantial reduction in the transfer coefficient in the presence of a solid phase was taken into account. Recent work by Kataska et. al. (1979) on gas/liquid mass transfer in an 18-ft-diam vessel also confirms the correlation for two-phase systems. However, the SRC-I Demonstration Plant dissolver design employs a 2-ft-diam inlet, which is more than 20 times larger than any inlet opening tested. Initially, large bubbles may occur. To what extent the entrance effect would influence the overall mass transfer coefficient is an important question in the dissolver design.

Wilsonville Pilot Plant experience has shown tremendous solids accumulation in the dissolver. A solids removal system is therefore required to control such loading in the dissolver. It is also preferable to remove the large particles, but not the fines, which may be catalytically active. 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.

During this program, studies of dissolver design without internals were completed; in addition, gas/liquid contact studies were completed for each internal (e.g., bubble cap, target plates, and gas spargers). Batch operation measurement of gas holdup, mass transfer coefficient, and approximate bubble size were obtained for each internal tested. Measurements of gas holdup and approximate bubble size with liquid flow were completed for the "no internals" configuration and the sparger/2-ft target plate configuration. In addition, batch operation measurements of the liquid axial dispersion coefficient were obtained for the sparger/2-ft target plate configuration. Finally, corresponding three-phase studies were performed for the sparger/2-ft target plate configuration.

EXPERIMENTAL PROCEDURE

Equipment

To ensure scale-up to an 11-ft-diam column (as proposed for the demonstration plant), ICRC tested the performance of a column having a diameter of 6 ft and a height of 25 ft. The piping of the system constructed for these studies was designed so that gas/liquid contact could be studied for both gas/liquid (two-phase) and gas/liquid/solid (three-phase) mixtures. Air, supplied by a compressor, was distributed through a manifold to instrument and operating lines, as well as to the process lines. One of the air process lines simulating the gas feed into the dissolver joined with the water/sand slurry line in a 1-ft-diam pipe before entering the column. The other air supply line fed directly into the column through a gas sparger system, or into the rear of the drain assembly. Nitrogen was sparged through the system to desaturate the oxygen content in the water so that the uptake rate of oxygen from air into the water could be measured to calculate the gas/liquid mass transfer rate.

The design of the 1-ft-diam column feed pipe simulated the interstage from the first dissolver to the second dissolver in series. A suitable slurry pumping and gas phase distributing system and a holding tank for air/slurry experiments were developed. The gas combined with the slurry in the horizontal section of the feed pipe located at an elevated level. This pipe turned 90°, and descended to a lower level. Part of this vertical feed pipe was Plexiglas to enable study of the down-flow pattern. The gas/slurry then flowed through another horizontal section before entering the column at the bottom. Part of this horizontal section was also made of Plexiglas so that the gas/slurry flow pattern could be observed before it entered the column.

Studies were made with and without vessel internal configurations. The internals included two crossed gas sparger pipes with 36 3/8-in. openings on each pipe; 1- and 2-ft-diam circular target plates, the latter with dividers; and a 4-ft-diam by 6-in-deep circular bubble cap with triangular teeth for phase separation.

Gas/Liquid Contact

Experiments to determine the extent of gas/liquid contacting were performed without internals in the column, and then with the several design configurations listed above. The gas/liquid contact system is depicted in Figure 1. Each set of experiments employing a particular design configuration was followed by a period during which equipment was modified to the next design configuration. Superficial gas velocities of up to 0.5 ft/sec and liquid velocities of up to 0.05 ft/sec were investigated, thereby covering the full range of SRC dissolver column design operating velocities.

Gas Holdup Measurement. For this measurement, the liquid displacement method was used. Measurements were made of the difference in liquid levels with and without air bubbling through the 1-ft inlet or tubular gas sparger system, as well as through the no-internals system. The ratio of this difference in volume to the aerated liquid volume represented the overall gas holdup. Liquid level measurements were made with a level sighting tube outside the column, as well as with a calibrated scale on the internal wall of the column.

In an alternative "delta h" measuring technique, the difference in height between the aerated vessel contents and the unaerated liquid level sighting tube was used to calculate the average density of material between the vessel surface and the two submerged levels shown in Figure 2. The technique was also applied to obtain the average holdup in the vessel segment located between the two submerged levels in Figure 2 (epsilon 5-20). (The term "epsilon 5-20" refers to holdup values obtained for the vessel segment located between the 5- and 20-ft nominal depths, while "epsilon 0-5" refers to the holdup between the surface and 5-ft nominal depth, and so on.)

Gas/Liquid Mass Transfer Measurement. Gas/liquid mass transfer coefficient measurements were made using dissolved oxygen probes to determine the rate of increase in oxygen saturation in the water during the sparging operation. Nitrogen was first sparged into the column at low flow rates to remove the dissolved oxygen from the water. Simultaneous oxygen readings at three vertical column levels were used to determine the degree of uniformity of oxygen saturation throughout the

column. The time rate of increase in oxygen saturation in the column was used to calculate the mass transfer coefficient.

Volumetric mass transfer coefficients were measured in the two-phase batch mode (zero liquid flow), with and without vessel internals. Volumetric mass transfer coefficients were also measured in the three-phase batch mode with 100-200-mesh sand using the spargers/2-ft plate internal. The method used in computing the coefficients has been described previously (McDermott and Ying, 1982). By using three dissolved oxygen probes positioned at different vessel depths, the assumption of a completely back-mixed liquid phase was confirmed as adequate for each of the internals tested. The results obtained were compared with the predictions of Akita and Yoshida (1973).

Liquid Axial Dispersion Measurement. In order to obtain measurements of the liquid axial dispersion coefficient, a quantity of tracer, either dye or salt solution, was introduced on the surface of the vessel during operation. Liquid samples were taken at approximately 1.4-sec intervals from a depth of 20 ft. The rate of migration of the tracer to the column bottom was a measure of the liquid axial dispersion. The time rate of increase in tracer concentration at a given depth was used to calculate the liquid axial dispersion coefficient, E_{ZL} , according to the procedure outlined by Khanq and Kothari (1980).

The dispersion coefficient was obtained from the tracer response curve by measuring the area between the response curve and the horizontal line of equilibrium tracer concentration. The area calculation was performed numerically using the trapezoidal rule. Approximately 31 tracer concentration values were used for each area calculation. One, two, or three runs were made for each dispersion coefficient, and the results were averaged.

Solids Concentration Measurements

Approximately 1 ton of 100-200-mesh sand was added to the system; a second was later added. After several experiments, 2 tons of 30-50 mesh sand were added so that the system contained equal amounts of both size sands. Solids concentrations were obtained during system operation by

withdrawing samples from ports located at depths of 0, 5.0, 9.7, 15.0, and 21.4 ft below the slurry outlet line. A sixth sample port was located in the vertical section of the vessel inlet drain assembly. The sampling port tubes could be traversed radially within the vessel. The samples were dried to determine the degree of uniformity of solids mixing within the vessel.

Solids Withdrawal System Tests

These experiments were designed to determine the effect of solids withdrawal rates upon system stability and weight percent solids withdrawal. For these tests, the following operating conditions were employed:

- Batch operation and internal liquid flow.
- Solids concentration of 16 wt % sand.
- Superficial gas velocities up to 0.5 ft/sec.
- Slurry withdrawal rates of 1.8-3.0% of slurry feed rate.

Experiments were performed using a system designed to withdraw solids from the upper face of the 2-ft target plate. The material was removed using a 1-in.-diam line, with flow induced by a hydrostatic pressure difference of about 25 ft of water. During these tests, the sparger gas flow was maintained at a value corresponding to 0.110 ft/sec. The inlet line gas flow was set to values corresponding to 0.100 and 0.200 ft/sec, and the inlet line slurry flow was set at 0.020 and 0.033 ft/sec. The withdrawal line flow rate was maintained at 7.5 gpm, which corresponded to 3.0% of the inlet slurry flow when the flow was 0.020 ft/sec, or 1.8% of the inlet slurry flow when the flow was 0.033 ft/sec.

The withdrawal system was turned on after the rest of the system had been operating for some time, and a no-withdrawal stability level had been achieved. Withdrawal samples were then obtained every 2 min as the system achieved a new stability level. (Withdrawn material was continuously returned to the holding tank to avoid progressive depletion of the system.)

EXPERIMENTAL RESULTS AND DISCUSSION

Gas Holdup

For the case of no internals and no liquid flow, the measured gas holdup values in the gas/liquid system obtained using the "delta h" technique were compared with those obtained using the liquid displacement method. The two techniques produced identical results.

Experimental gas holdup data obtained for the no-internals, two-phase case in the batch mode of operation are shown in Figure 3. Local gas holdup was expected to increase from the bottom to the top of the column due to hydrostatic expansion of individual bubbles. The measured values for epsilon 0-5 and 0-20 in Figure 3 were determined using the assumption that the mean surface level is at the liquid exit line. However, due to surface turbulence, the mean surface level was observed to lie somewhat below the exit level during batch operation. Consequently, the true epsilon 0-5 and 0-20 gas holdup values are lower than those shown in Figure 3. (Uncertainty in the mean surface level would not affect gas holdup values obtained between two submerged levels, such as epsilon 5-20.) As a result of the above observations, the true overall vessel gas holdup is believed to lie slightly below the values indicated for epsilon 0-20, but above the values indicated for epsilon 5-20. The prediction from the correlation of Akita and Yoshida (1973) shown in the figure would thus be slightly higher than the actual overall gas holdup value for the no-internals configuration; this is presumably due to the large rapidly rising gas slugs generated by the 1-ft-diam inlet.

The results of another research program (Ying et al., 1981) indicate that gas holdup measured in a 1-ft-diam vessel with a bubble-cap distributor plate is not affected by liquid flow having superficial velocities of up to 0.05 ft/sec. As indicated in Figure 4, values obtained for epsilon 5-20, which do not depend upon a knowledge of the true mean surface level, show a slight decrease in gas holdup with increasing liquid flow. This behavior could be characteristic of the no-internals vessel.

A comparison of batch operation gas holdup data obtained with the 1- and 2-ft-diam target plates, the 4-ft-diam bubble cap, and with the spargers supplying the gas flow is shown in Figure 5. The values of epsilon 5-20 differ from those obtained for no-internals, especially in the case of spargers supplying the gas flow. As can be seen, the epsilon 5-20 holdup values for sparger flow are significantly higher, a result of the better gas phase dispersion leading to smaller, more slowly rising bubbles in the vessel. With the sparger supplying gas flow, no large gas slugs were observed during operation.

Data obtained for the spargers/2-ft plate combination are compared with the no-internals case in Figure 6. Again, this internal combination results in higher gas holdup than that case without internals. In addition, the gas holdup values (Figure 6) are greater than those obtained for the 2-ft plate alone, but less than those obtained for the spargers alone (Figure 5).

Average gas holdup data were also obtained for vessel segments 5 to 10, 10 to 15, and 15 to 21.4 ft below the liquid exit line for the sparger/2-ft plate configuration. These experiments were run under batch conditions and with continuous liquid flow. Figure 7 clearly shows increases in local gas holdup from the bottom to the top of the vessel.

Gas holdup values were also obtained with the sparger/2-ft plate combination for the vessel volume located between 1.5 and 21.4 ft below the exit line (epsilon 1.5-20) (Figure 8). These values, both measured under batch conditions, most closely represent the true average gas holdup throughout the total vessel volume, irrespective of the true mean surface level position. Although the amount of gas flowing through the sparger differs by 30%, the resulting gas holdup is relatively unchanged.

When solids (sand) were added to the system, the measured gas holdup values decreased very slightly below those values measured for the two-phase system. First, low solids concentrations were studied by adding a small quantity of 100-200-mesh sand to the column with the combined sparger/2-ft plate internals. Vessel segment holdup values were obtained as for the two-phase system; Figure 9 shows the epsilon

5-20 holdup values obtained for a variety of gas and liquid flow conditions. The system solids concentration of 100-200-mesh sand was then increased approximately twofold. The holdup values were again observed to change only very slightly from the corresponding two-phase case. When an equal proportion of 30-50-mesh sand was added to the system, the overall solids concentration increased, but again there was little effect upon the measured gas void fractions.

Gas/Liquid Mass Transfer

The measured two-phase volumetric mass transfer coefficients obtained for each of the vessel internals tested are presented in Figure 10. For the no-internals configuration, the gas flow was introduced through the 1-ft inlet line, or through the 3-in. gas inlet port at the rear of the vessel drain assembly. When the 1-ft-diam target plate was inserted, the measured $k_L a$ values remained relatively unchanged, as shown in Figure 10. The data obtained with the 2-ft target plate and the 4-ft bubble cap in place were identical.

Experiments were performed with the tubular gas spargers in place and with all gas flow to the vessel supplied by the spargers. The results (Figure 10) indicate an increase in the measured volumetric mass transfer coefficients over those previously discussed. This increase can be attributed to the same improvement in gas phase dispersion and reduction in average effective bubble size that was observed to increase the measured gas phase holdup with the spargers in operation.

The gas spargers were also tested in combination with the 4-ft bubble cap and with the 2-ft target plate. Gas flow through the sparger was held constant at set values, while the gas introduced into the 1-ft inlet line was varied. The sparger/2-ft target plate experimental results are compared with the results for the spargers alone in Figure 11. In each case, the effect of sparger flow on vessel performance appears to predominate and to result in volumetric mass transfer coefficients nearly the same as those in the case of the sparger alone (Figure 10). The same behavior was observed for the spargers/4-ft bubble cap combination.

The presence of solids has a negligible effect on the gas/liquid mass transfer rate. Figure 12 compares the measured $k_L a$ values in the presence of 100-200-mesh sand particles with those of the gas/liquid system shown in Figure 11. Within experimental accuracy, the comparison suggests no appreciable difference with and without solid particles.

Comparison of the $k_L a$ data with the Akita/Yoshida correlation is also shown in Figure 12. The authors expressed the $k_L a$ in a dimensionless group or Sherwood number as a function of four other dimensionless quantities as follows:

$$N_{Sh}(a\theta) = \beta (N_{Sc})^{0.5} (N_{Bo})^{0.62} (N_{Ga})^{0.31} (\epsilon_g)^{1.1}$$

where

$$N_{Sh} = \text{Sherwood number, } \frac{k_L D}{D_L}$$

$$N_{Sc} = \text{Schmidt number, } \frac{u_L}{D_L}$$

$$N_{Bo} = \text{Bond number, } \frac{g D^2 \rho_L}{\sigma}$$

$$N_{Ga} = \text{Galileo number, } \frac{g D^3}{u_L^2}$$

ϵ_g = gas holdup or gas volume fraction

β = empirical constant, dimensionless

D = column diameter

D_L = liquid phase diffusivity

u_L = kinematic viscosity

ρ_L = liquid density

σ = surface tension

g = gravitation constant

In this correlation, the empirical constant (β) equals 0.60. ICRC's measured $k_L a$ values are appreciably lower than this predicted curve. 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 β values of 0.45 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. 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 design hydrogen consumption rate. Therefore, it is concluded that the SRC-I dissolver design is not gas/liquid mass transfer limited.

Liquid Axial Dispersion

Two sets of dispersion coefficient data were obtained for the case of tubular gas spargers combined with the 2-ft target plate. In the first set, the sparger contribution to the total flow was held at 0.110 ft/sec, while the 1-ft inlet line flow was varied. The averaged dispersion coefficients are shown in Table 1, along with the corresponding predicted values. For the second set of data (Table 2), the sparger contribution to the total flow was held constant at 0.146 ft/sec, while the 1-ft inlet line flow was varied. The measured liquid axial dispersion coefficients shown in the tables are an order of magnitude larger than those obtained with a 1-ft-diam vessel in a separate research program (Ying et al., 1981). Although the present data were obtained with a vessel of considerably larger proportions than those for which the available correlations were developed, the agreement between predicted and measured values is believed to be reasonable.

Liquid axial dispersion coefficients were also measured for the cases in which equal proportions of 30-50- and 100-200-mesh sand were present in the system. With a sparger gas flow corresponding to 0.110

ft/sec, and an inlet line gas flow corresponding to 0.25 ft/sec, the measured dispersion coefficient was $5.69 \text{ ft}^2/\text{sec}$, which is approximately the same as the measured two-phase liquid axial dispersion coefficient. Therefore, the correlations used in Tables 1 and 2 can be applied to scale-up to the 11-ft-diam demonstration plant dissolver. The results suggest a well-mixed system at demonstration plant design conditions.

Solids Concentration

Because no dependence of measured solids concentration upon radial position was detected under any of the experimental conditions tested, the solids concentrations at a given vessel level are reported as a single value. When only 100-200-mesh sand was present in the system, solids dispersion was essentially uniform under all conditions listed. With a small quantity of 100-200-mesh sand, the sparger flow was set at 0.110 ft/sec and the liquid flow was maintained at 0.01 ft/sec. As can be seen in a typical normalized concentration plot shown in Figure 13, the essentially flat concentration profiles indicate near-uniform dispersion of the solid phase for fine-mesh sand. Little change in this behavior was observed when the sparger flow was increased to 0.146 ft/sec, as shown in Figure 14. When two sparger flow rates were used (0.110 and 0.146 ft/sec), and when liquid flow was increased to 0.033 ft/sec, the solid phase dispersion was also unchanged (Figures 15 and 16). This insensitive response to the liquid flow rate is not surprising because of the almost homogeneously mixed system.

When the quantity of 100-200-mesh sand was increased, the solids also remained uniformly dispersed within the vessel. This can be seen in Figure 17 which shows data obtained at 0.033 ft/sec liquid velocity.

When an equal amount of 30-50-mesh sand was added to the system, the concentration profiles did, as expected, develop a distinct axial gradient. A typical concentration profile at a liquid velocity of 0.02 ft/sec is shown in Figure 18. In Figure 19, the liquid velocity was increased to 0.033 ft/sec, which in turn increased the dispersion of the solid phase, and, as expected, reduced the slopes of axial concentration gradients. It is interesting to note that eliminating gas flow in the 1-ft inlet line caused settling of the 30-50-mesh solid phase,

which resulted in a flatter axial concentration profile, as shown in Figure 20. Under this condition, only the well-dispersed 100-200-mesh solids remain in suspension.

Solids Withdrawal System

As shown in Figure 21, no discernable variation in the composition of withdrawn samples occurred over the period in which samples were taken. The variation in sample composition with inlet line slurry flow rate was also small. The greatest change in withdrawn slurry composition occurred when superficial gas velocity was changed, as shown in Table 3. At 0.210 ft/sec gas velocity, the withdrawn samples contained about 93 lb/ft³ solids, while at 0.310 ft/sec gas velocity, the withdrawn samples contained about 56 lb/ft³ solids. The higher gas velocities presumably generate increased turbulence, which reduced the average solids concentrations in the vicinity of the withdrawal point. This successful operation at high solids concentration suggests the applicability of this simple withdrawal design using hydrostatic head in the SRC-I dissolver.

Visual Data Records

Audiovisual data records were obtained at selected flow conditions for each of the internal geometries tested, i.e., for the 1-ft-diam vertical interstage pipe down-flow pattern, the 1-ft horizontal inlet line flow pattern, the lower port window bubble pattern, and the vessel liquid surface.

In continuous two-phase liquid flow experiments, the vertical down-flow line exhibited annular flow characteristics for each of the experimental flow conditions tested. The horizontal inlet line section just upstream of the drain assembly exhibited an unsteady flow pattern. In the batch mode of operation (no net liquid flow), the inlet line periodically filled with liquid. During this process, gas flow to the vessel temporarily ceased; the liquid then "surged" forward into the vessel, and gas flow resumed. The result was periodically interrupted gas flow to the vessel through the 1-ft inlet.

As gas flow rate was increased, the surging phenomenon ceased in the horizontal transparent section, and stratified two-phase flow was constantly maintained. (Of course, when gas flow was introduced through the 3-in. drain assembly inlet, the flow was steady and uninterrupted.) When net liquid flow was introduced, occasional traveling liquid slugs were formed in the horizontal transfer line under certain flow conditions, and this became superimposed upon the surging phenomenon described above. The result was a complex flow behavior having unsteady gas and liquid flow leading into the vessel inlet.

In the contact vessel, many finely dispersed bubbles approximately 1/8 to 1/4 in. in effective diameter persisted over the full range of flow conditions investigated. Superimposed upon this were the larger bubbles, which frequently achieved diameters approaching that of the inlet in the case of no internals. (The large slugs were not generated when gas was introduced through the 3-in. inlet line with no internals.) Each of these phenomena, along with surface behavior, were recorded using magnetic tape audiovisual equipment. The size of the finely dispersed bubbles was invariant among the internal configurations tested. The larger gas bubbles were completely eliminated only in the case in which 100% of the gas flow was contributed by the tubular gas spargers; this case yielded the best overall gas phase dispersion.

When no internals were in place, the gas phase rose from the vessel inlet in a dispersed column, which required approximately one vessel diameter to become distributed across the vessel cross-sectional area. This resulted in very little direct interfacial contact of clear liquid zones near the vessel bottom. Such zones were reduced in size or eliminated with the target plates or bubble cap in place, although these objects tended to create static zones above them.

The contact vessel surface was characterized by the presence of many finely dispersed bubbles combined with high turbulence generated by the larger, more rapidly rising gas bubbles. When surging behavior was observed in the 1-ft inlet line, the periodic interruption of gas flow to the vessel resulted in a corresponding temporary interruption of the surface turbulence. When 100% of the gas flow was supplied by the spargers, no large gas bubbles were present, and the surface was more

quiescent and characterized by the presence of only the smaller (1/8- to 1/4-in.) bubbles.

When solids were introduced into the system, a significant amount of sedimentation was observed in the 1-ft inlet line, even in the case of 100-200-mesh material. Settled solids typically occupied about 1/3 to 2/3 of the inlet line volume, and were never completely eliminated, even at the highest obtainable mixture flow rates. With no solids withdrawal, a large amount of settled material was observed on the 2-ft target plate during operation, and some settled sand was present around the periphery of the vessel bottom. Vessel surface turbulence and gas bubble behavior appeared to be about the same as for the two-phase system. The amount of inlet line sedimentation was recorded on video tape for selected experimental conditions and for both the fine-mesh and the mixed-mesh systems.

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Table 1
 Comparisor of Axial Dispersion Coefficients in Liquid Phase for the 6-foot Column
 2-Phase, Liquid Flow Rate = 0, Gas Spargers + 2-foot Target Plate
 Gas Sparger Contribution to the Total Flow = 0.110 ft/sec

Total gas velocity (ft/sec)	Predicted axial dispersion coefficient (ft ² /sec)						Experimental axial dispersion coefficient (ft ² /sec)
	Kato & Nishiwaki (1972)	Towel & Ackerman (1965)	Deckwer et al. (1974)	Hikita & Kikukawa (1974)	Baird & Rice (1975)	Ying	
0.110	7.28	6.00	4.30	2.59	5.82	3.94	2.41
0.160	7.59	7.23	4.81	2.99	6.59	4.51	6.22
0.210	7.87	8.28	5.22	3.36	7.22	4.98	4.37
0.260	8.14	9.22	5.56	3.71	7.75	5.38	5.31
0.310	8.41	10.07	5.86	4.04	8.22	5.72	7.58
0.360	8.65	10.85	6.13	4.36	8.64	6.04	6.12

Table 2

Comparison of Axial Dispersion Coefficients in Liquid Phase for the 6-foot Column
 2-Phase, Liquid Flow Rate = 0, Gas Spargers + 2-foot Target Plate
 Gas Sparger Contribution to the Total Flow = 0.146 ft/sec

Total gas velocity (ft/sec)	Predicted axial dispersion coefficient (ft ² /sec)					Ying	Experimental axial dispersion coefficient (ft ² /sec)
	Kato & Nishiwaki (1972)	Towel & Ackerman (1965)	Deckwer et al. (1974)	Hikita & Kikukawa (1974)	Baird & Rice (1975)		
0.146	7.50	6.91	4.68	2.88	6.39	4.37	2.98
0.196	7.79	8.00	5.11	3.26	7.05	4.86	4.87
0.246	8.07	8.97	5.47	3.61	7.61	5.27	3.90
0.296	8.33	9.84	5.78	3.95	8.09	5.63	6.31
0.346	8.59	10.63	6.06	4.27	8.52	5.96	10.21

Table 3
Solids Withdrawal Data

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

^a0.11 ft/sec gas through spargers.

Figure 1
Flow Diagram of the Gas/Liquid Contact System

Figure 2
The "Delta h" Holdup Measurement Technique

Figure 3
Measured Gas Void Fraction
No Internals; Batch Operation;
Gas Flow Through 12-in. Line

Figure 4
Measured Gas Void Fraction (epsilon 5-20)
No Internals; Continuous Liquid Flow;
Process Flow Through 12-in. Line

Figure 5
Measured Gas Void Fraction (epsilon 5-20)
Comparison of Internals; Batch Operation

Figure 6
Measured Gas Void Fraction (epsilon 5-20)
Comparison of Internals; Batch Operation

Figure 7
Measured Gas Void Fraction
Spargers + 2-ft Plate; Batch Operation;
Spargers Contribution to Total Gas Flow = 0.110 ft/sec

Figure 8
Measured Gas Void Fraction (epsilon 1.5-20)
Spargers + 2-ft Plate; Batch Operation

Figure 9
Measured Gas Void Fraction (epsilon 5-20)
Three-Phase Flow Using 100-200 Mesh Solids

Figure 10
Gas/Liquid Volumetric Mass Transfer Coefficient

Figure 11
Gas/Liquid Volumetric Mass Transfer Coefficient

Figure 12
Gas/Liquid Volumetric Mass Transfer Coefficient

Figure 13
 Measured Axial Solids Distribution
 Spargers + 2-ft Plate; 100-200 Mesh Solids;
 Liquid Velocity = 0.01 ft/sec; Sparger Contribution to
 Total Gas Flow = 0.110 ft/sec; Vessel Surface Solids
 Concentration = 1.39 lb/ft³

Figure 14
 Measured Axial Solids Distribution
 Spargers + 2-ft Plate; 100-200 Mesh Solids;
 Liquid Velocity = 0.01 ft/sec; Sparger Contribution
 to Total Gas Flow = 0.146 ft/sec; Vessel Surface
 Solids Concentration = 1.45 lb/ft³

Figure 15
 Measured Axial Solids Distribution
 Spargers + 2-ft Plate; 100-200 Mesh Solids;
 Liquid Velocity = 0.033 ft/sec; Sparger Contribution
 to Total Gas Flow = 0.110 ft/sec; Vessel Surface
 Solids Concentration = 2.38 lb/ft³

Figure 16
 Measured Axial Solids Distribution
 Spargers + 2-ft Plate; 100-200 Mesh Solids;
 Liquid Velocity = 0.033 ft/sec; Sparger Contribution
 to Total Gas Flow = 0.146 ft/sec; Vessel Surface
 Solids Concentration = 1.63 lb/ft³

Figure 17
 Measured Axial Solids Distribution
 Spargers + 2-ft Plate; 100-200 Mesh Solids;
 Liquid Velocity = 0.033 ft/sec; Sparger Contribution
 to Total Gas Flow = 0.110 ft/sec; Vessel Surface
 Solids Concentration = 3.93 lb/ft³

Figure 18
 Measured Axial Solids Distribution
 Spargers + 2-ft Plate; 100-200 and 30-50 Mesh Solids;
 Liquid Velocity = 0.02 ft/sec; Sparger Contribution
 to Total Gas Flow = 0.110 ft/sec; Vessel Surface
 Solids Concentration = 3.18 lb/ft³

Figure 19
 Measured Axial Solids Distribution
 Spargers + 2-ft Plate; 100-200 and 30-50 Mesh Solids;
 Liquid Velocity = 0.033 ft/sec; Sparger Contribution
 to Total Gas Flow = 0.110 ft/sec; Vessel Surface
 Solids Concentration = 4.65 lb/ft³

Figure 20
 Measured Axial Solids Distribution
 Spargers + 2-ft Plate; 100-200 and 30-50 Mesh Solids;
 Liquid Velocity = 0.02 ft/sec; Sparger Contribution
 to Total Gas Flow = 0.110 ft/sec; Vessel Surface
 Solids Concentration = 2.14 lb/ft³

Figure 21
6-Foot-Diameter Column
 $V_L = 0.02 \text{ ft/sec}$