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DIRECT LIQUEFACTION OF LOW-RANK COAL

*Quarterly Technical Progress Report
for the period April 1 - June 30, 1995*

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TABLE OF CONTENTS

LIST OF TABLES	i
EXECUTIVE SUMMARY	ii
1.0 INTRODUCTION	1
2.0 GOALS AND OBJECTIVES	1
3.0 ACCOMPLISHMENTS	2
3.1 Work Planned for This Quarter	2
3.2 Work Performed During This Quarter	2
3.2.1 Task 1 Operations	2
3.2.1.1 Test 3	2
3.2.1.2 Test 4	5
3.2.1.3 Test 5	5
3.2.1.4 Test 6	6
3.2.1.5 Test 7	6
3.2.1.6 Test 8	7
3.2.1.7 Test 9	7
3.2.1.8 Discussion	7
3.2.2 Preparation of Contractors' Review Conference Paper	9
3.2.3 Project Extension Request	9
3.3 Conclusions	9
4.0 FUTURE OBJECTIVES	10

LIST OF TABLES

1	Summary of Tests Performed from April Through June 1995	3
2	Run Conditions for Task 1, Tests 1 Through 9	4
3	Distillate Yields and Solvent Balances for Task 1, Tests 1 through 8	8

DIRECT LIQUEFACTION OF LOW-RANK COAL
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EXECUTIVE SUMMARY

A multistep direct liquefaction process specifically aimed at low-rank coals (LRCs) has been developed at the Energy & Environmental Research Center (EERC). The process consists of a preconversion treatment to prepare the coal for solubilization, solubilization of the coal in the solvent, and polishing using a phenolic solvent or solvent blend to complete solubilization of the remaining material. The product of these three steps can then be upgraded during a traditional hydrogenation step.

This project will address two research questions necessary for the further development and scaleup of this process: 1) determination of the recyclability of the solvent used during solubilization and 2) determination of the minimum severity required for effective hydrotreatment of the liquid product. The project will be performed as two tasks, the first consisting of ten recycle tests and the second consisting of twelve hydrotreatment tests performed at various conditions.

Several activities were performed during this quarter:

- A paper entitled "Solvent Recyclability in a Multistep Direct Liquefaction Process" was prepared for presentation at the Coal Liquefaction and Gas Conversion Contractors' Review Conference that will be held August 29-31, 1995.
- A no-cost project extension was requested and granted.
- Tests 3 through 8 in Task 1 were completed during this quarter. The pretreatment, solubilization, and polishing steps of Test 9 were completed.

During the Task 1 testing, the following were noted:

- The process remained both operationally and chemically constant.
- The Karl Fischer water analysis is not precise enough for use in yield structure calculations. Adequate data can be obtained by averaging the Karl Fischer water analysis results and the results of the physical separation that is performed during the distillation step.
- This process produces adequate quantities of excess solvent for recycle.

DIRECT LIQUEFACTION OF LOW-RANK COAL

1.0 INTRODUCTION

Direct liquefaction research at the Energy & Environmental Research Center (EERC) has, for a number of years, concentrated on developing a direct liquefaction process specifically for low-rank coals (LRCs) through the use of hydrogen-donating solvents and solvents similar to coal-derived liquids, the water-gas shift reaction, and lower-severity reaction conditions. The underlying assumption of all of the research was that advantage could be taken of the reactivity and specific qualities of LRCs to produce a tetrahydrofuran (THF)-soluble material that might be easier to upgrade than the soluble residuum produced during direct liquefaction of high-rank coals. A multistep approach was taken to produce the THF-soluble material, consisting of 1) preconversion treatment to prepare the coal for solubilization, 2) solubilization of the coal in the solvent, and 3) polishing to complete solubilization of the remaining material. The product of these three steps can then be upgraded during a traditional hydrogenation step.

To provide a preliminary comparison between the EERC process and existing direct liquefaction processes, product slurry produced during solubilization (Step 2) and polishing (Step 3) steps (for example, without the Step 1 pretreatment) was catalytically hydrotreated to equilibrium based upon hydrogen uptake. The hydrotreatment was performed in this manner to define the practical upper limit of the product's hydrotreatability. The results were positive, and further tests were performed incorporating the pretreatment step (Step 1). Steps 1 through 3 (pretreatment, solubilization, and polishing) were performed in an integrated fashion. The products were catalytically hydrotreated to demonstrate the maximum hydrotreatability of the solubilized slurry and to provide products that could be compared to the products of existing processes.

The results of the EERC's research indicated that additional studies to more fully develop this process were justified. Two areas were targeted for further research: 1) determination of the recyclability of the solvent used during solubilization and 2) determination of the minimum severity required for hydrotreatment of the liquid product. This project addresses these two areas.

2.0 GOALS AND OBJECTIVES

The project goals are to determine the recyclability of the solvent used during solubilization and to determine the minimum hydrotreatment severity required to upgrade the liquid product of the multistep EERC process.

The project will be performed as two tasks. The first task will consist of ten recycle tests. Lignite will be solubilized through the pretreatment, solubilization, and polishing steps. The product of these three steps will be combined with a vehicle solvent, and the resulting stream will be distilled to remove water, solubilization solvent, and oxygenated light coal-derived liquids. The overheads will be further distilled to separate the water and light oil streams. The light oil will then be recycled for use as the solubilization solvent in the next test sequence. The analyses of the

products of these tests will be used to characterize the recycle solvent stream and to calculate mass and material balances.

The second task will consist of a series of 12 hydrotreatment tests at various conditions. The tests will be performed according to a statistically designed experimental matrix to enable the identification and evaluation of the most effective low-severity hydrotreatment conditions. Analyses of the products of these tests will be used to characterize the hydrotreated product and to calculate mass and material balances.

3.0 ACCOMPLISHMENTS

3.1 Work Planned for This Quarter

Work originally scheduled for the quarter was to complete of the project. Because of scheduling changes made during the first quarter of the project, the Task 2 testing was rescheduled for performance during this quarter.

3.2 Work Performed During This Quarter

Three activities were performed during this quarter: 1) completion of Task 1, Tests 3 through 9, 2) preparation of a paper for the Direct Liquefaction Contractors' Review conference to be held in Pittsburgh, Pennsylvania, on August 29-31, 1995, and 3) request and approval for a no-cost project extension through November 30, 1995.

3.2.1 Task 1 Operations

Twenty runs were made during the April-June quarter as part of the Task 1 testing. These runs are summarized in Tables 1 and 2 and are discussed in more detail in the following paragraphs.

3.2.1.1 Test 3

Test 3 was performed as Runs N611, N612, and N613. (Run N611 was performed during the January-March quarter and is discussed here as part of Test 3.) During Run N611, approximately 0.342 kg of moisture- and ash-free (maf) Freedom lignite was slurried with 0.65 kg of the recycle solvent obtained from the Run N610 distillation. The autoclave was charged with slurry, CO, and H₂S, which served as the reaction promoter. The pretreatment was performed at 151°C and 1412 psig for 30 min. The conditions were then increased to 373°C and 3560 psig for 60 min. for the solubilization step. The unit was quenched and the product recovered and sampled. The overall material balance for the test was 99.0%.

The N611 product slurry was polished during Run N612. The product slurry was charged to a preheated autoclave containing 0.171 kg of recycle solvent and H₂. The polishing step was performed at average conditions of 434°C and 4575 psig for 20 min. The reaction was quenched and the product recovered. The polishing step overall material balance was 99.2%.

TABLE 1

Summary of Tests Performed from April Through June 1995

Run No.	Description	Mass Balance, %
N612	Task 1, Test 3 – Polishing step	99.2
N613	Task 1, Test 3 – Distillation of polished product	97.9
N614	Task 1, Test 4 – Pretreatment and solubilization steps	98.7
N615	Task 1, Test 4 – Polishing step	99.1
N616	Task 1, Test 4 – Distillation of polished product	98.1
N617	Task 1, Test 5 – Pretreatment and solubilization steps	99.3
N618	Task 1, Test 5 – Polishing step	99.0
N619	Task 1, Test 3 – Redistillation of bottoms from N613	97.9
N620	Task 1, Test 5 – Distillation of polished product	99.6
N621	Task 1, Test 6 – Pretreatment and solubilization steps	99.8
N622	Task 1, Test 6 – Polishing step	98.8
N623	Task 1, Test 6 – Distillation of polished product	97.2
N624	Task 1, Test 7 – Pretreatment and solubilization steps	99.1
N625	Task 1, Test 7 – Polishing step	98.8
N626	Task 1, Test 7 – Distillation of polished product	97.3
N627	Task 1, Test 8 – Pretreatment and solubilization steps	99.7
N628	Task 1, Test 8 – Polishing step	98.9
N629	Task 1, Test 8 – Distillation of polished product	97.4
N630	Task 1, Test 9 – Pretreatment and solubilization steps	99.8
N631	Task 1, Test 9 – Polishing step	98.2

The N612 polished product slurry was combined with 0.82 kg HAO61 heavy fraction and distilled during Run N613. Water was removed and the distillation performed at endpoint conditions of 1.10 psig and 219°C to separate the light oil fraction from the heavier fraction. The light oil fraction was recycled for use as the feed solvent for Test 4. A mass balance of 97.9% was attained for the Test 3 distillation step.

TABLE 2

Run Conditions for Task 1, Tests 1 Through 9

Processing Step	1	2	3	4	5	6	7	8	9
Pretreatment	N602	N607	N611	N614	N617	N621	N624	N627	N630
Temperature, °C	152	153	151	152	149	150	151	148	151
Pressure, psig	1402	1397	1412	1424	1458	1474	1461	1424	1477
Time, min	35	30	30	30	30	31	30	30	30
Gas	CO	CO	CO	CO	CO	CO	CO	CO	CO
Additive	H ₂ S	H ₂ S	H ₂ S	H ₂ S	H ₂ S	H ₂ S	H ₂ S	H ₂ S	H ₂ S
Solubilization	N602	N607	N611	N614	N617	N621	N624	N627	N630
Temperature, °C	377	377	373	372	372	374	374	374	374
Pressure, psig	3478	3520	3560	3440	3391	3493	3449	3340	3267
Time, min	60	60	60	60	60	60	60	60	60
Polishing	N603	N609	N612	N615	N618	N622	N625	N628	N631
Temperature, °C	441	440	434	437	438	442	443	443	440
Pressure, psig	4605	4540	4575	4294	4001	4204	4147	3987	3761
Time, min	20	20	20	20	20	20	20	20	20
Gas	H ₂	H ₂	H ₂	H ₂	H ₂	H ₂	H ₂	H ₂	H ₂
Additive	Feed Solvent	RS-606	RS-610	RS-613	RS-616	RS-620	RS-623	RS-626	RS-629
Distillation	N606	N610	N613	N616	N620	N623	N626	N629	- ^a
Temp., pot, °C	221	219	219	221	221	223	223	222	-
Pressure, psig	1.19	1.10	1.10	1.09	1.10	1.09	1.09	1.09	-

^a The Test 9 distillation was not performed during this quarter.

An unexpected electrical power outage occurred prior to Run N613. The equipment was preliminarily tested to be sure that it still operated, but the pressure transducer calibration was not tested. Because it was no longer in calibration, the distillation was not performed at the correct conditions, and approximately 20 wt% of the light organics remained in the bottoms. This changed the composition of the recycle organics used as the solvent in the Test 4 feed slurry. The bottoms from the N613 distillation were redistilled during N619, and the appropriate light material was collected for readdition to the system.

During analysis it was noted that some light organic material was removed with the aqueous phase during distillation. This material is phenolic in nature and makes up about 10 wt% to 16 wt% of the aqueous stream. The organics were gravity-separated for readdition to the system with the additional light organics collected during the N619 distillation.

3.2.1.2 Test 4

Runs N614–N616 made up Test 4. The pretreatment and solubilization steps were performed as Run N614. Approximately 0.300 kg of maf Freedom lignite was slurried with 0.590 kg of recycle solvent obtained from the N613 distillation. The slurry was cold-charged to the autoclave with CO and H₂S, the reaction promoter. The pretreatment was performed at 152°C and 1424 psig for 30 min. Solubilization was performed at 372°C and 3440 psig for 60 min. After the unit was quenched, the product was recovered and sampled. The overall material balance for these two integrated steps was 98.7%.

The N614 product slurry was charged to a preheated autoclave containing 0.151 kg recycle solvent and H₂ for Run N615. The average polishing conditions were 437°C and 4294 psig for 20 min. The reaction was quenched and product recovered. An overall material balance of 99.1% was achieved for the polishing step.

The polished product slurry from N615 was combined with 0.68 kg HAO61 heavy fraction and distilled during Run N616. Water was removed from the organics and the distillation performed at the end point conditions of 1.09 psig and 221°C pot temperature to separate the light oil fraction from the heavier fraction. The light oil fraction was recycled for use as the feed solvent for Test 5. An overall mass balance of 98.1% was achieved for the Test 4 distillation.

3.2.1.3 Test 5

Test 5 was performed as Runs N617, N618, and N620. During Run N617, approximately 0.249 kg maf Freedom lignite was slurried with 0.493 kg of the recycle solvent obtained from the Run N616 distillation. The autoclave was charged with slurry, CO, and H₂S, which served as the reaction promoter. The pretreatment was performed at 149°C and 1458 psig for 30 min. The conditions were then increased to 372°C and 3391 psig for 60 min. for the solubilization step. The unit was quenched and the product recovered and sampled. The overall material balance for the test was 99.3%.

The N617 product slurry was polished during Run N618. The product slurry was charged to a preheated autoclave containing 0.127 kg of recycle solvent and H₂. The polishing step was

performed at average conditions of 438°C and 4001 psig for 20 min. The reaction was quenched and the product recovered and sampled. The polishing step overall material balance was 99.0%.

The N618 polished product slurry was combined with 0.59 kg HAO61 heavy fraction and distilled during Run N620. Water was removed and the distillation performed at end point conditions of 1.10 psig and 221°C pot temperature to separate the light oil fraction from the heavier fraction. The light oil fraction was recycled for use as the feed solvent for Test 6.

3.2.1.4 Test 6

The additional light organics obtained from the N619 distillation (i.e., the redistillation of the N613 distillation bottoms) as well as the light oil removed from the aqueous phase separated during distillation were added to the N620 recycle solvent. Approximately 0.524 kg of this mixture was combined with 0.266 kg maf Freedom lignite to form the feed slurry for Run N621, the pretreatment and solubilization steps for Test 6. The slurry was cold-charged to the autoclave with CO and H₂S, the reaction promoter. The 30-min pretreatment step was performed at 150°C and 1474 psig. The solubilization step was performed at 374°C and 3493 psig for 60 min. An overall mass balance of 99.8% was achieved for this run.

The resulting product slurry was polished during Run N622. In this run, the slurry was hot-charged into an autoclave containing H₂ and 0.13 kg recycle solvent. The polishing step lasted 20 min and was performed at 442°C and 4204 psig. After the reaction was quenched, the product was recovered and sampled. The polishing step overall mass balance was 98.8%.

The polished product slurry was distilled during Run N623. Water was removed and the distillation was performed at end point conditions of 223°C pot temperature and 1.09 psig to separate the light oil fraction from the heavier fraction. The lighter organic fraction was recycled for use as the feed solvent for Test 7. A mass balance of 97.2% was calculated for the distillation step.

3.2.1.5 Test 7

Runs N624, N625, and N626 made up Test 7. The pretreatment and solubilization steps were performed as Run N624. Approximately 0.258 kg of maf Freedom lignite was slurried with 0.497 kg of recycle solvent obtained from the N623 distillation. The slurry was cold-charged to the autoclave with CO and H₂S, the reaction promoter. The pretreatment was performed at 151°C and 1461 psig for 30 min. Solubilization was performed at 374°C and 3449 psig for 60 min. After the unit was quenched, the product was recovered and sampled. The overall material balance for these two integrated steps was 99.1%.

The N624 product slurry was charged to a preheated autoclave containing 0.129 kg recycle solvent and H₂ for Run N625. The average polishing conditions were 443°C and 4147 psig for 20 min. The reaction was quenched and product recovered and sampled. An overall material balance of 98.8% was achieved for the polishing step.

The polished product slurry from N625 was combined with 0.57 kg HAO61 heavy fraction and distilled during Run N626. Water was removed from the organics and the distillation

performed at the end point conditions of 1.09 psig and 223°C pot temperature to separate the light oil fraction from the heavier fraction. The light oil fraction was recycled for use as the feed solvent for Test 8. An overall mass balance of 97.3% was achieved for the Test 7 distillation.

3.2.1.6 Test 8

Runs N627, N628, and N629 made up Test 8. The pretreatment and solubilization steps were performed as Run N627. Approximately 0.232 kg of moisture- and ash-free (maf) Freedom lignite was slurried with 0.456 kg of recycle solvent obtained from the N626 distillation. The slurry was cold-charged to the autoclave with CO and H₂S, the reaction promoter. The pretreatment was performed at 148°C and 1424 psig for 30 min. Solubilization was performed at 374°C and 3440 psig for 60 min. After the unit was quenched, the product was recovered and sampled. The overall material balance for these two integrated steps was 99.7%.

The N627 product slurry was charged to a preheated autoclave containing 0.114 kg recycle solvent and H₂ for Run N628. The average polishing conditions were 443°C and 3987 psig for 20 min. The reaction was quenched and product recovered and sampled. An overall material balance of 98.9% was achieved for the polishing step.

The polished product slurry from N628 was combined with 0.51 kg HAO61 heavy fraction and distilled during Run N629. Water was removed from the organics and the distillation performed at the end point conditions of 1.09 psig and 222°C pot temperature to separate the light oil fraction from the heavier fraction. The light oil fraction was recycled for use as the feed solvent for Test 9. An overall mass balance of 97.4% was achieved for the Test 8 distillation.

3.2.1.7 Test 9

The Test 9 pretreatment, solubilization, and polishing steps were performed as Runs N630 and N631. (The distillation step was not performed during this quarter.) During Run N630, approximately 0.214 kg maf Freedom lignite was slurried with 0.424 kg of the recycle solvent obtained from the Run N629 distillation. The autoclave was charged with slurry, CO, and H₂S, which served as the reaction promoter. The pretreatment was performed at 151°C and 1477 psig for 30 min. The conditions were then increased to 374°C and 3267 psig for 60 min. for the solubilization step. The unit was quenched and the product recovered and sampled. The overall material balance for the test was 99.8%.

The N630 product slurry was polished during Run N631. The product slurry was charged to a preheated autoclave containing 0.105 kg of recycle solvent and H₂. The polishing step was performed at average conditions of 440°C and 3761 psig for 20 min. The reaction was quenched and the product recovered and sampled. The polishing step overall material balance was 98.2%.

3.2.1.8 Discussion

The material balances for Tests 1 through 8 are presented in Table 3. These data show that recoveries for the gas and liquid fractions for each of the steps are similar. The liquid balance for the pretreatment/solubilization step ranged from 90.4% to 91.7%. The polishing step liquid

TABLE 3

Distillate Yields and Solvent Balances for Task 1, Tests 1 through 8

Test No.	Hydrotreatable Solubles, wt% maf ^a		Solvent Yield, wt% maf	Solvent Balance, %
	Liquid Basis ^b	Gas Basis ^c		
1	79.52	87.02	42.01	116.42
2	71.23	86.49	68.17	127.75
3	85.98	86.08	46.27	118.57
4	86.02	85.00	13.67	105.47
5	82.61	83.08	17.12	106.80
6	83.33	79.63	49.53	120.00
7	79.57	83.83	39.52	115.84
8	72.86	79.86	44.88	114.05
Average	80.14	83.87	40.15	116.12

^a Weight percentage of moisture- and ash-free coal fed to the system.^b Yield calculated from liquid stream mass balance data.^c Yield calculated by subtracting the gas yield from unity.

balance ranged from 95.9% to 97.7%. Overall mass balances for the pretreatment/solubilization step ranged from 96.0% to 99.8%, for the polishing step from 98.2% to 99.2%, and for the distillation step from 97.2% to 99.6%. The consistency of the mass balances for each step proves that the system is operationally stable. The fact that the overall mass balances are at least 96.0% indicates that adequate amounts of material were collected for analysis and subsequent tests and that the data, therefore, reliably describe the process.

The solvent balance from the Test 3 distillation was reduced because some of the distillable material was not collected. After redistillation of the original distillation bottoms, an actual Test 3 solvent balance was determined. This is the value shown in Table 3, which summarizes the solvent balance and yields of hydrotreatable solubles and distillate for Tests 1 through 8. As the table shows, solvent balances of more than 115% were attained for Tests 1 through 3. The lower balances shown by Tests 4 and 5 are probably due to the improper recycle solvent composition. The additional material removed during the redistillation of the Test 3 distillation bottoms was added to the recycle solvent stream used in the Test 6 feed slurry. As a result of the lower fraction of light distillate in Tests 4 and 5, the solvent recovery dropped from approximately 15% excess solvent to about 5% excess solvent. When the light distillate was recovered and added back in Test 6, the solvent recovery increased to the excess solvent levels noted for Tests 1–3. The average solvent recovery for all eight tests is 116.12%.

The yields of hydrotreatable soluble material noted in Table 3 were calculated two different ways: from the liquid stream mass balance data and by subtracting the gas yield from unity. The reliability of the data is high since the numbers are similar and exhibit a consistent 4% difference between values calculated by both methods. To determine a preliminary yield structure, the quantity of maf-derived material produced was determined for the individual tests. These quantities ranged from 62.0 wt% to 98.3 wt%. This is quite a large variance when compared to all of the other physical measurements, such as mass balances and most liquid-phase analytical data. The gas-phase mass and material balances are also very similar. Most of the data suggest that only subtle changes occurred between tests. All of the analytical data were examined for abnormally large variances. This examination suggested a problem with the Karl Fischer water analysis. During the previous reporting period activities, this was noted and a double check was performed using gas-based analysis and mass flow data. During the current reporting period, the water-free gas data were evaluated and are summarized in Table 3. The average yield of maf-derived material in the liquid phase determined by this method was 83.9%. The average for this comparable yield based on Karl Fischer water analysis of the product slurry was 88.87%, but with an error of $\pm 18.18\%$. The water content was also checked by determining the quantity of water present in the distillation step. The yield of maf-derived material present in the distillation bottoms was determined using this percentage of water, which averaged $74.4\% \pm 9.1\%$. The two water percentages were averaged resulting in a yield of maf-derived material of $80.14\% \pm 7.4\%$. This compares favorably with the $83.9\% \pm 3.7\%$ based on the gas data. As a result, the average water analysis was used for the preliminary estimates of the system yields. Final yields will be based on the yield structure determined by hydrotreatment stage tests and verified by elemental balances.

3.2.2 Preparation of Contractors' Review Conference Paper

A paper entitled "Solvent Recyclability in a Multistep Direct Liquefaction Process" was prepared for presentation at the Coal Liquefaction and Gas Conversion Contractors' Review Conference that will be held August 29-31, 1995.

3.2.3 Project Extension Request

A no-cost project extension until November 30, 1995, was requested and granted.

3.3 **Conclusions**

- The process remained both operationally and chemically constant, even though the recycle solvent composition was abruptly changed when some of the light solvent was not returned to the recycle stream.
- The process produces adequate quantities of excess solvent for recycle.
- The Karl Fischer water analysis is not precise enough for use in yield structure calculations. Adequate data can be obtained by averaging the Karl Fischer water analysis results and the results of the physical separation that is performed during the distillation step.

- This work was performed using a composite solvent that had not been previously used in the multistep process. Although the solvent was prepared using information gathered during previous process development work, it is possible that, as it recycles, the solvent will not remain hydrogen-enriched enough to be optimally effective in the pretreatment and solubilization steps. If not enough hydrogen is available, light organic material could be removed from the hydrotreated product stream and recycled to the pretreatment step.

4.0 FUTURE OBJECTIVES

- Tests 9 and 10 from Task 1 will be completed.
- The Task 2 tests will be performed.
- Quality assurance checks will be performed as outlined in the project quality assurance plan.
- The final technical report will be prepared.