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CATALYTIC MULTI-STAGE LIQUEFACTION OF COAL

SECOND QUARTERLY REPORT FOR THE PERIOD

1 JANUARY 1993 - 31 MARCH 1993

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WORK PERFORMED UNDER CONTRACT

DE-AC22-93PC92147

HYDROCARBON RESEARCH, INC.
100 OVERLOOK CENTER, SUITE 400
PRINCETON, NEW JERSEY 08540

AUGUST 1993

MASTER

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ABSTRACT

This quarterly report covers activities of Catalytic Multi-Stage Liquefaction of Coal during the period January 1 - March 31, 1993, at Hydrocarbon Research, Inc. in Lawrenceville and Princeton, New Jersey. This DOE contract period is from December 8, 1992 to December 7, 1994.

The overall objective of the program is to produce liquid fuels from direct coal liquefaction at a cost that is competitive with conventional fuels. Specifically, this continuous bench-scale program contains provisions to examine new ideas in areas such as: low temperature pretreatments, more effective catalysts, on-line hydrotreating, new coal feedstocks, other hydrogen sources, more concentrated coal feeds and other highly responsive process improvements while assessing the design and economics of bench-scale results.

The quarterly report covers work on Laboratory Scale Studies, Continuous Bench-Scale Operations, Technical Assessment and Project Management.

SUMMARY

Laboratory Scale and Bench-Scale studies commenced in this quarter. A new batch of Illinois #6 coal was qualified for use based on microautoclave testing. Microautoclave studies with a sodium lignosulfonate surfactant showed a significant improvement in conversion at the highest severity conditions and no effect at lower severities. A series of slurry viscosity determinations were made with different particle size distributions and solvents. An optimized particle size distribution showed negligible change in viscosity when compared to the standard distribution.

The first bench-scale (CMSL-1) run of the program studying the use of dispersed molybdenum and iron catalysts was completed in this quarter. With 300 ppm molybdenum, added excellent performance was observed with and without iron impregnated coal. A lower rate of catalyst deactivation was found with the molybdate in comparison to the dispersed iron addition.

In-Line hydrotreating was also studied in CMSL-1 and showed excellent performance (HDS of 76% and HDN of 74%).

A run plan for CMSL-2 to study the effect of coal, slurry concentration and surfactant on performance is presented.

INTRODUCTION

This is the second progress report of a two-year contract to evaluate novel process concepts for Direct Coal Liquefaction using the Catalytic Two-Stage process. The primary objective is:

- To produce liquid fuels from direct coal liquefaction at a cost that is competitive with conventional fuels.

The program consist of five tasks:

Task 1	Management Plan
Task 2	Laboratory Studies
Task 3	Bench-Scale Studies
Task 4	Technical Assessment
Task 5	Project Management/Administration/Environmental

TASK 2 - LABORATORY SCALE STUDIES

Coal Qualification Tests:

In preparation for Run CMSL-2, evaluation of lower oil/coal ratio, with a batch of Illinois No.6 coal (HRI-6107) microautoclave qualification tests were conducted. A standard sub-bituminous distillate (HRI-6002) from Wilsonville Pilot Plant was used to determine the coal conversion and 524°C⁺ residuum conversion of this new batch of coal. The standard conditions for coal evaluation were:

Solvent/coal ratio	= 8g/2g
Catalyst	= 2g
Temperature	= 399 & 427°C (750 & 800°F)
Time	= 30 min.
Pressure	= 13.8 MPa of H ₂

Results are shown in *Figure 1*. The THF coal conversion at the lower temperature (399°C) for the new batch of coal (HRI-6107) is comparable to another batch of Illinois #6 coal (HRI-6081) used in CC-16 (AO-60 Catalyst) run (84.6 W% as compared to 86.3 W%). However, at the higher temperature level, the new lot of Illinois No. 6 coal is significantly less reactive, 8W% lower in conversion, than HRI-6081.

Effect of Surfactant:

The use of surfactant to modify the physical properties of a system, hence alternating the hydrodynamics of coal liquefaction reactor was suggested by Jet Propulsion Laboratories, Calif. A test matrix was designed to determine the impact of sodium lignosulfonate on coal and residuum (524°C⁺) conversions of Illinois No. 6 coal (HRI-6107).

Preliminary results are shown in *Table 1 and Figure 2*. Except under the most severe condition tested (427°C and 30 min.), sodium lignosulfonate has no or marginal effects on the coal conversion of Illinois No. 6 coal in the presence of Shell 317 supported catalyst. At temperature of 399°C with 15 or 30 minutes of reaction time, the difference in coal conversions with and without the surfactant range from 0.7 to 1.8 W%. At the higher temperature (427°C) the conversions are the same for both cases for the shorter reaction time tests. However, the addition of the sodium lignosulfonate improves coal conversion by 6.9 W% for the 30 minute tests (Test CL-34 vs Test CL-24).

Residuum conversion data are not available at the time of reporting and will be reported in the next quarter.

TABLE 1**Effect of Surfactant on Conversion of Illinois No. 6 Coal**

Solvent/coal ratio: 4/1

Catalyst Shell 317 Ni/Mo Catalyst

Pressure: 13.8 MPa

Run No.	Temp. ° C	Time, min.	Surfactant	Coal Conv. (W%)
Thermal				
30	399	30	No	73.3
32	427	30	Yes	85.1
Catalytic				
23	427	15	No	87.4
33	427	15	Yes	87.4
24	427	30	No	84.9
34	427	30	Yes	91.8
25	399	15	No	76.6
35	399	15	Yes	79.4
26	399	30	No	84.6
36	399	30	Yes	83.9

Viscosity of Feed Slurry:

The viscosity of a feed slurry, solvent/coal ratio of 0.9, was determined at a range of temperatures (65 to 230°C) using a Brookfield viscometer.

A distillate (filtered L-760) and two pressure filter liquids (R76-E3 and R76-S/D) obtained from a previous run on Illinois No. 6 coal (CC-16, Run 227-76 E3) were used. The former distillate was generated from Black Thunder Coal and stored in Tank #4 and was used as start-up solvent for PDU 260-03. These liquids were chosen to cover the range of viscosities typically experience in a 15 day bench run.

The origin of these liquids are briefly described:

Solvent	API	IBP, °C	524°C ⁺ , W%
L-760 Filtered	10.1	318	None
R76-E3	1.3	308	39.0

The R76-E3 is a pressure filter liquid (PFL) obtained from the last yield periods of Bench Run CC-16 at relatively low severity conditions, while R76-S/D was PFL from the shutdown period (after coal feed was cut off) was the same bench run.

Results are illustrated in *Figure 3*. The R76-E3 PFL gives the most viscous coal/oil slurry. As anticipated, the least viscous slurry was from the start-up solvent. The impact of particle size distribution in the feed coal on viscosity of the feed slurry was briefly evaluated. The feed coal was separated into several size fractions and recombined according to the optimum particle size distribution proposed by Farris¹, see *Table 2*. In contrast to Farris method, the "optimum PSD" coal sample and R76-S/D slurry has higher viscosities than slurry from the original feed coal sample with the same PFL. The addition of 2 W% of sodium lignosulfonate, a surfactant, does not have significant effect the viscosity of the slurry.

¹. Sadler, L.Y. and Sim, K.G.; "Minimize Solid-Liquid Mixture Viscosity By Optimizing Particle Size Distribution", Chem. Eng. Prog., pp68-71, March 1991.

TABLE 2
Particle Size Distribution of Feed Coal

Sieve Size (Mesh)	Distribution (W%)	Optimum PSD (Farris Method)
+70	0.4	0
70-100	3.4	11.8
100-200	28.4	22.2
200-325	39.6	15.0
-325	28.2	51.0

Bench Run CMSL-2: Coal Qualification Tests

HRI-6081 vs HRI-6107

Shell 317 Catalyst, Solvent: HRI-6002, 30 min., 13.8 MPa H₂

FIGURE 2
Effect of Sodium Lignosulfonate on Conversion
Presulfurized Shell 317 Catalyst
Solvent: HRI-6002, Coal: Illinois No. Coal (HRI-6107)

FIGURE 3

Viscosity of Ill. No. 6 and PFL
Oil/Coal Ratio = 0.9

TASK 3 - BENCH-SCALE STUDIES

RUN CMSL-1 - Dispersed Moly Catalysts

Bench Run CMSL-1 "The Evaluation of Molybdenum Slurry Catalyst with an Iron Catalyst for Sub-Bituminous Coal" was conducted in this period.

Objective

The objective of this run was to evaluate the impact of molybdenum and iron additives on the liquefaction of Wyoming sub-bituminous coal (Black Thunder Mine) under a thermal/catalytic mode of operation. The first stage was a pre-heater stage to activate the catalyst, the second stage was a fully backmixed slurry reactor and the final (third) stage was an ebullated bed with a supported catalyst. A secondary objective of this run was to evaluate the effect of on-line hydrotreating on the product quality.

Operations

The iron catalyst precursor was impregnated on the coal matrix using an incipient wetness technique developed by Pittsburgh Energy Technology Center (described in U.S. Patent No. 5,096,570, March 17, 1992, "Method for Dispersing Catalyst onto Particulate Material" by A. Cugini and B. Utz) and previously scaled up by HRI for bench run CC-15. Black Thunder Mine coal was impregnated with hydrated iron oxide (FeOOH) at 5000 ppm of iron using this technique. The molybdenum catalyst was added as ammonium heptamolybdate at 300 ppm (Mo).

The iron and molybdenum catalyst precursors were first activated in a pre-treater stage (570°F) prior to the thermal stage. The catalytic stage was an ebullated bed reactor consisting of Shell 317 $1/32$ " extrudate. The hydrotreater was a 1 liter tubular reactor operated as a mixed-phase trickle bed and packed with Criterion 411 catalyst. The performance of this system was compared to bench run CC-15 which was with the iron impregnated catalyst and without the molybdenum catalyst or hydrotreater.

The two catalyst precursors were activated with H_2S (3 W% coal) at 572°F in the pre-treater stage. The test was conducted at a space velocity of 66 lbs coal/hr/ft³ catalyst for conditions 1 and 3 and at 75 lbs coal/hr/ft³ catalyst for condition 2. The slurry reactor temperature was maintained at 825°F (25°F higher than in bench run CC-15) and the ebullated bed reactor was maintained at 775°F (identical to bench run CC-15). The run conditions and results are given in Tables 3 & 4, attached.

The coal conversion was a constant 94 W% (maf coal) for conditions 1 and 3 and slightly lower for condition 2, at 93.4 W% (maf coal), which had a higher space velocity. The C_1 - C_3 gas yields for conditions 1 and 2 with the iron impregnated coal were consistent at an average of 9.1 W% (dry coal). The gas yield without the iron impregnated coal in condition 3 was 1.3% higher. The C_4 -975°F yield and 975°F+ conversion both showed the normal trend of decreasing as the catalyst deactivated. The product oils also demonstrated the expected trend of lower yields of the light distillate and higher yields of the heavy distillate and resid fractions as the catalyst deactivated. *Figure 4*, attached, shows the distillate yields, the resid conversions and the coal conversions. *Figure 5*, attached, shows the product oils divided into heavy distillate, light distillate and resid fractions.

Runs CMSL-1 and CC-15 were compared based on catalyst age rather than on a direct period by period comparison due to the difference in space velocities, 66 lbs/hr/ft³ for CMSL-1 and 42 lbs/hr/ft³ for CC-15. Coal conversion for CMSL-1 averaged 93.8% while coal conversion for CC-15 was 1% lower. This was due to the higher temperature that the slurry reactor was run at in CMSL-1. *Figure 6*, attached, shows the comparison of the resid conversions between CMSL-1 and CC-15. At equal catalyst ages the CMSL-1 run has a consistently higher resid conversion, from 2% to 4% higher. Also, for the CMSL-1 run, the resid conversion decreases at a slower rate than for CC-15. *Figure 7*, attached, shows a similar pattern for the 975°F+ yield. The CMSL-1 run generally has a higher distillate yield and a slower rate of decrease in the yield as the catalyst ages. *Figure 8*, attached, shows the resid content of the PFL for runs CMSL-1 and CC-15. For CC-15 the resid content continues to increase as the catalyst ages, but for CMSL-1 the resid content appears to be leveling off, or at least increasing at a much lower rate. All of this indicates that the rate of deactivation of the catalyst in CMSL-1 is lower than in CC-15.

Throughout the entire run the hydrotreater was on-line. At the end of the run an extension condition was added without the hydrotreater so as to give a basis of comparison to evaluate the hydrotreater. The mass and elemental balances around just the hydrotreater are given in *Tables 5 & 6*, attached. The total mass balance around just the hydrotreater was 103.1% and the oil balance (ignoring gases and water) was 98.4%. The hydrodenitrogenation around only the hydrotreater was 73.7% and the hydrosulfurization was 75.9%.

CONCLUSIONS

Run CMSL-1 (227-77) was successfully completed with two conditions on FEOOH impregnated coal with the molybdenum additive and with one condition with untreated coal and the molybdenum additive. Coal conversion for all three operating conditions ranged from 93.6 to 94.0 W% (maf) while 975°F+ resid conversion ranged from 89.1 to 91.6 W% (maf).

While the periods chosen from CMSL-1 and CC-15 are not ideal for comparisons, they still allow valid conclusions to be drawn. At comparable operating conditions the use of combined molybdenum/iron slurried catalysts in the first stage of the CMSL process resulted in an improved process performance for liquefaction of a sub-bituminous coal over that obtained from the use of either iron or molybdenum catalyst alone or the use of the supported Ni-Mo/Al₂O₃ catalyst in the first stage. CMSL-1 achieved a higher or equal level of resid conversion and a better or equal product distribution than either of the other two. This performance was achieved while maintaining a 46 to 66 wt% higher throughput.

The total mass balance around just the hydrotreater was 104.1% and the oil balance (ignoring gases and water) was 98.4%. The hydrodenitrogenation around only the hydrotreater was 73.7% and the hydrodesulfurization was 75.9%. The hydrotreater also improved the quality of the final product stream from a calculated H/C ratio (without hydrotreating) of 1.66 to an actual value of 1.74.

TABLE 3

**THERMAL/CATALYTIC COAL LIQUEFACTION WITH
MOLYBDENUM AND IRON SLURRY CATALYST PRECURSORS**

Wyoming Black Thunder Mine Coal
Shell S-317 1/32" Extrudate Catalyst
Ammonium Heptamolybdate Slurry Catalyst

Condition	1	2	3	ext
Periods	5	7	14	15a
Pressure, psig	2493	2503	2496	2498
Catalyst Age 2nd stage (lb coal / lb cat)	196	284	317	325
Temperature (°F)				
Pretreater	573	572	572	572
1st Stage	823	822	824	826
2nd Stage	775	775	775	776
Hydrotreater	659	660	661	592
Hot Separator	609	610	605	604
Atmospheric Still	505	502	497	498
Space Velocity				
Kg/h/M ³ cat.	1044	1205	1044	1044
lb/h/ft ³ cat.	65	75	65	65
Additives				
Molybdenum, ppm	300			
Iron, ppm	5000	5000	0	0
H ₂ S, W% mf coal	3%			
Performance (W% MAF coal)				
C ₁ -C ₃ (W% dry coal)	8.93	9.33	10.43	10.19
C ₄ -975°F Yield	64.5	63.5	60.7	65.2
975°F+ Conversion	91.6	90.5	89.1	87.9
Coal Conversion	94.0	93.4	93.9	93.6
H ₂ Consumption	8.2	8.1	8.5	8.0

TABLE 4

THERMAL/CATALYTIC COAL LIQUEFACTION WITH
MOLYBDENUM AND IRON SLURRY CATALYST PRECURSORS

Wyoming Black Thunder Mine Coal
Shell S-317 1/32" Extrudate Catalyst
Ammonium Heptamolybdate Slurry Catalyst

Condition	1	2	3	ext
Periods	5	7	14	15a

Normalized Yields (W% dry fresh feed)

C ₁ -C ₃ in Gas	8.93	9.33	10.43	10.19
C ₄ -C ₇ in gas	3.87	3.98	4.73	4.61
IBP-390°F	16.64	14.53	16.15	15.59
390-500°F	10.87	10.17	10.35	9.66
500-650°F	24.22	23.30	18.72	19.57
650-850°F	4.38	6.52	6.71	10.33
850-975°F	0.76	1.24	1.03	2.17
975°F+ Resid	2.18	2.77	4.54	5.50
Unconverted Coal	5.71	6.17	5.84	6.04
Ash	5.87	5.87	5.13	5.13
Water	20.48	19.65	19.96	14.47
CO _x	1.63	1.98	3.72	3.77
NH ₃	2.84	2.75	0.90	0.68
H ₂ S	-0.18	-0.21	0.30	0.30
Total	108.19	108.06	108.50	108.01

TABLE 5
HYDROTREATER MASS BALANCE

INPUT STREAMS	HOT SEP. OVERHEAD	INJECTED ASOH RECYCLED	WATER TO COLD SEP.	TOTAL
TOTAL FLOW (gm/hr)	885.3	236.3	315	1436.6
<u>COMPONENT FLOWS (gm/hr)</u>				
H ₂	173.1			173.1
C ₁ -C ₃	92.7			92.7
C ₄ -C ₇	41.8			41.8
CO _x	34.2			34.2
H ₂ S & NH ₃	24.5			24.5
TOTAL GASES	366.3			366.3
H ₂ O	234.2		315	549.2
IBP-390°F	120.8	23.6		144.4
390-500°F	57.3	47.3		104.6
500-650°F	83.7	130.0		213.7
650°F+	23.0	35.4		58.4
TOTAL LIQUIDS	519.0	236.3		755.3
PROD STREAMS	COLD SEP. OVERHEAD	COLD SEP. BOTTOMS	TOTAL	
TOTAL FLOW (gm/hr)	369.6	1111.2	1480.8	
<u>COMPONENT FLOWS</u>				
H ₂	173.7		173.7	
C ₁ -C ₃	101.8		101.8	
C ₄ -C ₇	46.1		46.1	
CO _x	36.3		36.3	
H ₂ S & NH ₃	11.7		11.7	
TOTAL GASES	369.6		369.6	
H ₂ O		598.6	598.6	
IBP-390°F		146.1	146.1	
390-500°F		101.5	101.5	
500-650°F		213.2	213.2	
650°F+		51.8	51.8	
TOTAL LIQUIDS		1111.2	1111.2	
TOTAL INPUTS		1436.6		
TOTAL PRODUCTS		1480.8		
MASS BALANCE %		103.1%		

* Estimated from period 15A with adjustments for coal SV.

TABLE 6

HYDROTREATER ELEMENTAL BALANCE

STREAM	<u>INPUTS</u>		<u>PRODUCTS</u>
	HOT SEP. OVERHEAD	INJECTED ASOH RECYCLED	COLD SEP. BOTTOMS
OIL COMPONENT (gm/hr)	284.8	236.3	512.6
<u>ELEMENTAL ANALYSIS</u>			
CARBON (W%)	86.41	87.21	84.23
HYDROGEN (W%)	12.57	11.32	12.49
NITROGEN (PPM)	3356	3600	926.7
SULFUR (PPM)	688	130.4	106.7
OXYGEN (PPM) (by difference)	6156	1100	1766
H/C RATIO	1.74	1.56	1.72

HYDROTREATER PERFORMANCE

HYDRODENITROGENATION	73.7%
HYDRODESULFURIZATION	75.9%

RUN 227-77 (CMSL-1) PROCESS PERFORMANCE (WT% OF MAF COAL)

FIGURE 4

RUN 227-77 (CMSL-1)
PRODUCT OIL FRACTIONS (WT% DRY COAL)

FIGURE 5

COMPARISON CC-15 & CMSL-1 975+F CONVERSION VS CATALYST AGE

FIGURE 6

■ CMSL-1 + CC-15

COMPARISON CC-15 & CMSL-1 C4-975F YIELD VS CATALYST AGE

FIGURE 7

■ CMSL-1 + CC-15

COMPARISON CC-15 & CMSL-1 RESID CONTENT OF PFL VS CATALYST AGE

FIGURE 8

TASK 3 - A BENCH SCALE STUDIES

RUN CMSL-2 - COAL SLURRY CONCENTRATION

Objective

To evaluate the impact of lower solvent/coal, from the current practice of 1.2 to 0.8, ratio on the liquefaction of Illinois No. 6 coal under CTSL mode of operations.

Technical Objectives:

1. To evaluate the impact of solvent/coal ratio, in the range of 1.1 to 0.8, on the performance of liquefying Illinois No. 6 coal under CTSL mode of operation.
2. To evaluate effects of reaction severity at high coal loading conditions
3. To evaluate effects of the addition of sodium lignosulfonate (2 W% of dry coal) on the conversions and liquid yield at a low solvent/coal ratio of 0.8 (under a sub-contract from JPL).

Run Plan

A run plan consisting of five conditions is shown in *Table 7*. This bench-run is being currently carried out. The results from CMSL-2 will be presented in the next Quarterly Report.

TABLE 7

CMSL-2 EFFECT OF OIL/COAL RATIO

Run Plan

Illinois No. 6 Coal		Shell S-317 Ni/Mo Catalyst			
Condition	1	2	3	4	5
Periods	1-5	6-9	10-12	13-15	16-19B (JPL)
Temperature, deg F					
1st Stage Reactor	750	750	775	775	775
2nd Stage Reactor	800	800	810	810	810
Hot Separator	600	600	600	600	600
Atmospheric Still	625	635	635	635	635
Catalyst Type					
1st Stage (Supported)	S-317	S-317	S-317	S-317	S-317
2nd Stage (Supported)	S-317	S-317	S-317	S-317	S-317
Solvent/Coal ratio	1.1	0.8	0.8	0.8	0.8
Space Velocity					
lb dry coal/h/ft ³ cat.	46	46	46	69	69
Additive**, W% dry coal	0	0	0	0	2.0

* subjected to the results from the off-line slurry pumping test.
to be conducted before the bench run.

** Sodium Lignolsulfonate

JPL: Jet Propulsion Lab. Sub-contract to HRI
(Evaluation of Surfactant)

TASK 4 - TECHNICAL ASSESSMENT

Two screening studies were planned in this period, one to examine the economics of using dispersed catalyst vs. supported catalyst in the first stage and a second to examine the cost impact of using syngas in the first stage and hydrogen in the second stage. Results should be available in the next quarter.

TASK 5 - PROJECT MANAGEMENT

Paper

A paper entitled " Catalytic Multi-Stage Liquefaction of Black Thunder Mine Coal using both Supported and Slurry Catalysts" is being prepared for presentation at the Chicago 206 National ACS Meeting from August 22-27, 1993.

Meetings

A planning and kick off meeting for the Catalytic Multi-Stage program was held on January 14, 1993 at PETC.

END

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12/22/93