FE-496-138
UC-90d

SOLVENT REFINED COAL (SRC) PROCESS

Monthly Report for the
Period July 1977

The Pittsburg & Midway Coal Mining Co.
Merriam, Kansas 66202

Date Published September 1977

PREPARED FOR THE UNITED STATES
ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION

Under Contract No. EX-76-C-01-496

ABSTRACT

This report summarizes the progress of the Solvent Refined Coal (SRC) Project by The Pittsburg & Midway Coal Mining Co., at the SRC Pilot Plant at Fort Lewis, Washington, and the P&M Laboratory in Merriam, Kansas for the Energy Research and Development Administration for the month of July 1977. During July, the Fort Lewis Pilot Plant processed a blend of Kentucky 9 and 14 coal during 25 days of operation in the SRC II mode. One SRC II material balance run was completed during this period. Three SRC II runs to explore the effect of varying pressure were made at the Merriam Laboratory.

I. Summary

One SRC-II material balance run was completed in July. A total liquid yield of 33% was obtained with SRC yield about 20%. The run was made at conditions of 25% coal, 0.8 hour residence time, 860°F and 1900 psig. Total slurry feed rate was 8026 lb/hr.

Problems with the old vacuum flash preheater eventually forced a plant shutdown on July 14 because of a plugged preheater coil. The new vacuum flash system was returned to service with a new centrifugal bottoms recirculation pump and the material balance run was completed. Several large vessels were eliminated from the material balance envelope by use of the new flash system, improving the accuracy of liquid yield measurements.

The old vacuum flash preheater coil was cut at the 15th and at the 17th turns from the inlet and was finally cleared when the plug was broken through between the 17th and 18th turns. Water from steaming and hydroblasting during this procedure apparently overloaded the waste treatment area upsetting this facility.

The increased coal feed rates led to a host of problems with the eductor coal mixing system. A number of coal outages were taken to clean the scrubber and two major plugs occurred when slurry recirculation problems reduced motive fluid flow. The vapor removal system of the new mix tank was modified and this unit was returned to operation near the end of the month.

Tests conducted on the new vacuum flash system showed it could be operated continuously to produce a vacuum bottoms product with a 400°F average fusion point.

Engineering projects for the month included installation of a vapor eductor to eliminate back pressure in the new slurry mix tank system and X-ray inspection of the plugged vacuum flash preheater coil.

A definitive estimate of $705,000 was submitted for engineering on the EPRI filter installation. It is anticipated that the total project will cost $1.05 million.

Three SRC II runs were made at the Merriam Laboratory to explore the effect of varying pressure on product yields. Pressures of 1700, 1900, and 2100 psig were used with other conditions held constant. Yields varied in the expected manner with oil yield increasing and SRC yield decreasing with increasing pressure.

II. Pilot Plant Operations, Engineering and Maintenance

A. Coal Receiving and Preparation - (Area 01)

Five hundred twenty-two tons of Kentucky 9 and 14 coal were processed during 25 days of operation in July. All operation was in the SRC-II mode. One material balance run was completed at feed rates of approximately 2095 lb/hr raw coal, 5749 lb/hr recycle slurry, and 304 lb/hr solvent seal flush to the slurry pumps.

Numerous coal outages were attributed to operation of the eductor coal mixing system. At higher coal rates frequent cleaning of the scrubber was required to prevent a major plug. Two major outages occurred when slurry circulating pump problems reduced motive fluid flow. The new mix tank system was returned to service after modification of the vapor removal system to prevent back pressure buildup in the mix tank. The new system performed well after this modification.

B. Slurry Preheating and Dissolving - (Area 02)

Area 02 processed coal slurry for a total of 25 days. All operation was in the SRC-II mode except for four hours of SRC-I operation on July 3. All operation was with a full dissolver. Operating targets were 800°F slurry preheater outlet, hydrogen quench to maintain a dissolver temperature of 860°F, and a 1900 psi dissolver pressure.

The operation of #3 separator caused two severe upsets. With both upsets, light oil flow to the stripper was lost which resulted in heavy stripper bottoms and area 01 problems. The July 8 upset was caused by a mistake in flow line up. The July 22 upset was caused by the failure of both #3 separator oil pumps. The oil flow to the stripper is now maintained by operating #3 separator at a higher pressure than the stripper; no pumps are utilized.

Area 02 operation was severely upset July 12 when both quench water pumps failed. Coal feed was out for 6 1/2 hours in order to control temperatures during pump repairs. Immediately after coal slurry processing resumed, instrumentation problems caused a slurry preheater flame failure. Coal feed was out for two hours and forty minutes for heater repairs.

C. Mineral Separation and Drying - (Area 03)

Filter "B" gas loop was circulated at normal operating temperature (500°F) during the month to provide an additional load for the Dowtherm system. This was necessary as a result of the inadequate turn-down ratio on the gas fired heater.

The filter feed flash and filter feed surge vessels were utilized for reclaiming solvent to the process as well as for additional surge capacity during periods of 04 area upsets.

The oil-water separation system operated normally during the month. However, problems occurred with the high pressure water booster pumps on July 12, resulting in a coal outage. The packing failed on "A" pump and the safety valve on "B" pump relieved prematurely which resulted in insufficient quench for the 02 area.

D. Solvent Recovery - (Area 04)

The original vacuum flash drum system was reassembled on July 1, after hydroblasting the transfer line from the heater to the vacuum flash drum. Intermittent plugging problems were encountered at numerous points in the system during the first fifteen days of operation. On July 15, the heater coil plugged, resulting in a shutdown of the system. Attempts were unsuccessful to flush or steam out the plug in the heater coil. The heater coil was X-rayed to locate the plug and the fifteenth and seventeenth coils were cut for hydroblasting. A considerable amount of coke was found between the eighth and eighteenth coils. In addition, flow control valves 220 A and B, pressure control valves 216A and B, level control valve 219A, and all piping in the recirculation loop were inspected for plugs. Plugs that were located and corrected included CS-25, the CS-5 manifold, 216A, 219A, and 220A. Attempts are still being made to decoke the heater coil.

A centrifugal recirculation pump was installed in the new vacuum flash drum system to replace one of the original gear pumps. The new system was returned to operation on July 15 using the centrifugal pump and operated well the remainder of the month. The only problems encountered with this sytem were a ruptured Dowtherm jacket on the recirculation line block valve and a mechanical seal failure in the centrifugal recirculation pump. After initially processing levels accumulated in the filter feed surge vessel, feed was introduced to the system directly from area 02.

On July 28 and 29, it was demonstrated that the new vacuum flash system could be operated to maintain a 400°F plus melt point.

The fractionation columns were operated in an intermittent mode with feed from liquids collected in the wash solvent accumulator. The addition of an oxidation inhibitor to the middle distillate product stream was begun in July.

Vacuum flash condensate drum liquid was used for seal flush during material balance runs and to supplement the feed to the original vacuum flash system.

E. Gas Recovery and Recompression - (Area 05)

Gas Recovery Unit

Naphtha unit operation was severely upset on July 20, 21, and 22 as a result of DEA carryover to the naphtha absorber on two separate occasions. The resulting mixture was allowed to settle and the DEA

solution drained off. Some problems were encountered maintaining the recycle hydrogen purity during these upsets.

Circulating pump vibration continued to cause pump oiler failures. Pump suction snubbers were installed and discharge snubbers will be installed.

Gas Recompression Unit

Other than a valve failure in the fresh H_2 compressor, no compressor problems were encountered. On July 27, coal feed was stopped for 4 1/2 hours while the third stage valves were repaired. As a preventive measure, the second stage pressure safety valve was also replaced.

F. Product Solidification and Storage - (Area 08)

Solidified SRC was routed from the cooling belt into fiber drums. Each drum was weighed and dumped on the off spec pile. On July 28 and 29, an SRC product with a 400°F plus melt point was produced. Product cooling was inadequate with a 200°F temperature at the discharge of the belt. It was necessary to remove the product chopper and gravimetric feeder to minimize plugging in the transfer system.

A bearing on the cooling belt drive roller was replaced on July 12. In addition, plugging problems were encountered with the vapor removal system filters.

G. Waste Treatment - (Area 09.1)

The waste treatment area operated with one major upset during the month. As a result of excessive phenols in the influent, coupled with a significant biomass loss, the plant effluent was high in phenols during the last half of the month. The effluent was treated with hydrogen peroxide and ferric sulfate.

H. Tank Farm - (Area 09.2)

Storage tanks 007, 043, 044 and 045 were inerted with high purity nitrogen in order to properly store SRC-II liquid products. In addition, the original Varec level indicators were replaced with differential pressure level indicators.

The new 1300 barrel storage tanks are currently being erected for additional storage of SRC-II liquids.

I. Gas Systems - (Area 09.5)

Stretford Unit

Maintenance work was completed and the unit was returned to service during the last week of this month. It appears to be operating satisfactorily at the present time.

J. Dowtherm Unit - (Area 09.8)

The Dowtherm heater control system operates at a critical stage during SRC-II operation when the heat duty is reduced. It is a low load condition, causing the heater to trip off frequently.

III. Process Analytical Data

A. Coal Receiving and Preparation (Area 01) and Preheating and Dissolving (Area 02)

The average analysis of raw coal was 8.32% ash and 6.00% moisture. The average analysis of the dehumidified pulverized coal during the month was:

Carbon	72.07%	-40 mesh	99.40%
Hydrogen	5.25%	-100 mesh	98.77%
Nitrogen	1.48%	-140 mesh	96.06%
Sulfur	3.32%	-200 mesh	78.39%
Oxygen (by difference)	8.18%		
Moisture	0.29%		
Ash	9.41%	Total Iron	1.39%

The analysis of forms of sulfur done on weekly composites of pulverized coal was:

	7-10-77	7-16-77	7-23-77	7-31-77
Pyritic Sulfur	1.46%	1.29%	1.34%	1.49%
Sulfate Sulfur	0.16%	0.17%	0.11%	0.08%
Organic Sulfur	1.80%	1.71%	1.75%	1.67%
Total Sulfur	3.42%	3.17%	3.20%	3.24%

The slurry from the slurry blend tank averaged 12.51% ash and 34.90% pyridine insoluble materials.

The average analysis of the recycle stripper bottoms was:

Water	0.26%
Naphtha	0.11%
Middle Distillate	6.29%
Heavy Distillate	34.62%
Vacuum Bottoms	58.72%
Pyridine Insolubles (as rec'd)	21.42%
Ash in Phridine Insolubles	62.99%
Total Iron (as rec'd)	2.56%

B. Solvent Recovery - Area 04)

Typical analyses of liquid products based on ASTM D-86 distillation data were:

Laboratory Distillation Fractions	Pilot Plant Products		
	Naphtha	Middle Distillate	Heavy Distillate
Naphtha up to 380°F	76%	10%	0%
Middle Distillate 380 to 550°F	24%	87%	6%
Heavy Distillate 550 to 850°F	0%	3%	94%

C. Product Solidification - (Area 08)

The plant vacuum bottoms produced this month averaged 24.21% ash with a fusion point of 336°F. Elemental analyses typically were: 67.63% carbon, 3.88% hydrogen, 1.56% nitrogen, and 2.72% sulfur.

D. Process Waste Disposal - (Area 09.1)

During the first half of the month, typical analyses of waste water units were:

	Bio-Unit Feed	Bio-Unit Effluent	Plant Effluent (Composite)
pH	7.1	7.2	7.4
Total Suspended Solids, ppm	95.5	25	1.7
Phenol, ppm	40	0.37	0.03
Chemical Oxygen Demand, ppm	705	104	13
Biological Oxygen Demand, ppm	160	41	4.5

During the last half of the month, the phenol content in the bio-unit feed rapidly increased to over 400 ppm. Since the micro-organisms in the bio-unit had not reached their optimum condition, they were unable to handle this sudden increase. During this upset, the plant effluent reached an average peak of 20 ppm phenol for approximately sixty hours. With dilutions and use of hydrogen peroxide and ferric sulfate additions to the plant effluent, the waste effluent lagoon (i.e. Harmer Marsh) phenol level was kept to an average value of less than 2 ppm.

IV. Process Evaluation

A. Material Balance Run

One material balance run was completed during the month of July. Run 77SR3 began at 0700 hours July 22 and ran for a total of 95 hours. However, coal feed was interrupted fourteen times during the run, reducing the actual running time to 88.67 hours. The results of 77SR3, shown in Tables 1 and 2, are based on an 88.67 hour run period. The effect of coal outages on the results of this run are felt to be negligible, since slurry compositions in the reaction area did not change significantly during the brief periods when the coal was not being fed.

Use of the new vacuum flash drum resulted in the elimination of the seal flush accumulator, vacuum flash feed accumulator and filter feed flash vessel from the material balance envelope, thus increasing the accuracy of the liquid balance. Average run conditions for Run 77SR3 are shown in Table 1. The run was made at a slurry feed rate 20% higher than the rates used in Runs 77SR1 and 77SR2. Other operating conditions were the same as in the previous runs.

Products yields from Run 77SR3 are listed in Table 2, and yield determination procedures are outlined below.

1. The yields of SRC, ash, and insoluble organic matter were based on the total weight of the plant produced vacuum bottoms, which was caught in drums and weighed during the run period. Averages of laboratory analyses of samples taken during the run were used to separate the vacuum bottoms yield into the components SRC, ash, and IOM.

2. Liquid yields were determined by measuring the accumulation of liquids in plant inventory during the run period.

3. Gas yields were determined from avaraged on-line flow rates and chromatographic analyses obtained during the run. A series of problems in this system resulted in the use of only the final six gas analyses in determining gas yield, but it is felt that those samples were representative of conditions during the run.

4. The water plus loss yield is the difference between the product yields discussed above and the coal feed rate.

B. Dissolver Density Instrumentation

During July a differential presssure indicator was connected across Dissolver A from the bottom hydrogen quench to the pressure indicator taps (340.5 inches apart). Initial measurements have been very encouraging and indicate that it may be feasible to monitor solids accumulation in the dissolver by density measurements from this instrument.

C. Vacuum Flash System Studies

In future runs, as coal rates are increased, slightly higher SRC yields are expected. However, if more distillate can be removed from the SRC in the vacuum flash system, it may be possible to offset the increase in vacuum bottoms yield due to reaction conditions by vacuum flashing at more severe conditions. Therefore, a study was undertaken to investigate the following:

1) The relationship between vacuum bottoms fusion point and the amount of distillate retained in the vacuum bottoms.

2) Maximum severity at which the vacuum flash system can be operated continuously, and the fusion point of the vacuum bottoms produced at those conditions.

During Material Balance Runs 77SR1 and 77 SR2, vacuum flash data were collected. During these runs the reaction conditions were well lined out and product yields were relatively constant. Data collected from these periods indicated that, at constant reaction conditions, the ash in the vacuum bottoms decreased as the fusion point of the vacuum bottoms decreased. Rearrangement of these results to show the effect of vacuum bottoms fusion temperature on the amount of solvent retained in the vacuum bottoms resulted in the mathematical expression:

$$\% \text{ Distillate in Vacuum Bottoms} = 0.189\ (\Delta T)$$

where ΔT is the difference between a reference temperature at which it is desirable to know the distillate retention and the actual fusion point of the vacuum bottoms.

In a second study on the vacuum flash system, operating conditions in the vacuum flash system were set at levels which would result in higher than normal fusion points. This study indicated that the new vacuum flash system could operate continuously at conditions severe enough to produce a vacuum bottoms product with a 400°F fusion point.

V. Special Projects

A. SRC-II Liquid Storage

Modifications to existing storage tanks to ensure the storage of SRC-II liquid products under an essentially leak-tight nitrogen blanket were completed. Construction of two new 55,000 gallon storage tanks was approximately 66% complete at the end of July.

B. EPRI Filter

A definitive estimate of $705,000 for engineering of Phase I was submitted. Definitive estimates are being prepared for an upgraded filter cake surge system and for an alternate "dry cake" letdown system, both of which are deemed necessary. It is anticipated that the final amount will be approximately $1.05 million.

C. Lummus Deashing Project

Engineering of the plot plan, process flow diagrams, piping and instrumentation diagrams and major equipment is now essentially complete. A definitive estimate is due August 31, 1977 and completion of the project is scheduled for August 31, 1978. Construction in the field could possibly begin November 1, 1977.

VI. Merriam Laboratory

Three SRC II runs made with Kentucky Nos. 9 and 14 coal (P&M Colonial Mine, lot 5) are reported this month. The purpose of these runs was to explore the effect of varying pressure. Conditions and results for these runs are summarized in Table 3. In all cases feed slurry composition was 30% coal and 70% unfiltered coal solution, nominal liquid residence time was 1 hr and nominal dissolver temperature was 455°C (851°F). Yields varied in the expected manner with oil yield increasing and SRC yield decreasing with increasing pressure. These results are summarized below. Hydrogen consumption, calculated by product analysis, increased with increasing pressure (Table 3).

Run	Pressure, psig	Yields, Wt % Dry Coal Basis	
		Total Distillate	SRC
GU 179R	1700	35.5	27.6
GU 180R	1900	38.3	24.8
GU 178R	2100	41.8	20.0

There was little variation in recycle solvent (heavy distillate fuel) compositions with varying pressure. Hydrogen content varied only between 8.13 and 8.19% and sulfur content between 0.20 and 0.24%. Although reported hydrogen contents increased with increasing pressure, the variations are not regarded as significant. Optimum desulfurization was observed at the highest pressure but again, the variations in composition may not be significant.

VII. Future Plans

In the next period the SRC Pilot Plant will continue to develop the SRC II Process on Kentucky 9 and 14 coal from the Colonial Mine. The Merriam Laboratory will make exploratory SRC II runs with other coals which may be processed at the pilot plant. Coals to be investigated include those from the Pittsburgh seam and Illinois No. 6 seam.

VIII. Travel

A. Fort Lewis

Trip No.	No. Attending	Destination	Purpose
1	2	Newark, NJ	Lummus Discussion
2	1	Denver, CO	Contract Discussion
3	2	Denver, CO	Syn. Fuels Meeting
4	1	Los Angeles, CA	Beckman Inst.School

B. Merriam

1	1	Denver, CO	GMRC Meeting
2	1	Denver, CO	Syn. Fuels Meeting
3	1	Blacksville, WVA	Coal Samples

TABLE 1

MATERIAL BALANCE RUN 77SR-3

RUN CONDITIONS

Raw Coal Feed:	2095 #/hr
Net Dehumidified Coal Feed:	1973 #/hr
Moisture Free Coal Feed:	1972 #/hr
Solvent Feed (as Seal Flush):	304 #/hr
Recycle Slurry Feed Rate:	5749 #/hr
Slurry Feed to Preheater:	8026 #/hr
Slurry Blend Composition:	
% Coal	24.6
% Solvent	31.1
% Vacuum Bottoms	27.8
% Ash	10.6
% IOM	5.9
Recycle Gas to Preheater:	611.4 #/hr
Recycle Quench Gas:	
Top	100.6 #/hr
Middle	105.6 #/hr
Bottom	0 #/hr
Recycle Purge Gas:	20.1 #/hr
Total Recycle Gas: (mass)	837.7 #/hr
Total Recycle Gas: (volume)	89937 SCFH
Recycle Hydrogen Purity % (mole):	92.2
Hydrogen to Slurry Ratio:	5.50 #H_2/100# Slurry
Slurry Heater Outlet Temperature:	806 ^{o}F
Dissolver Pressure:	1896 PSIG
Average Dissolver Temperature:	859 ^{o}F
Dissolver Temperatures:	

Feet From Inlet						
4	8	12	16	20	24	28
856	858	858	859	860	859	860

TABLE 2

MATERIAL BALANCE RUN 77SR-3

RESULTS

Component	Yield % MFC
H_2	-3.1
C_1	5.0
C_2	4.1
C_3	3.6
C_4	2.2
CO	2.3
CO_2	1.4
H_2S	1.5
H_2O Plus Losses	15.7
Naphtha (to 380°F)	5.7
Middle Distillate (380-550°F)	16.1
Heavy Distillate (550-850°F)	10.8
SRC*	20.2
IOM	5.2
Ash	9.3
TOTAL	100.0

*Average Fusion Point = 345°F

TABLE 3

SUMMARY OF SRC II PROCESS CONDITIONS AND YIELDS
(MERRIAM LABORATORY)

Kentucky No. 9 and 14 from the P&M Colonial Mine

	GU 179R	GU 180R	GU 178R
Conditions			
Nominal Residence Time, hr	1.06	1.04	1.03
Coal Feed Rate, lb/hr/ft^3	20.3	20.7	20.9
Hydrogen Feed Rate			
Wt % based on slurry	4.9	4.8	4.8
MSCF/ton of coal	61.9	60.8	60.1
Pressure, psig	1700	1900	2100
Yields, wt % based on dry coal			
H_2O	8.4	8.2	9.5
C_1-C_4	14.4	14.4	15.8
CO, CO_2, H_2S, NH_3	4.5	4.0	4.4
C_5+ (gas)	4.5	6.1	4.8
Naphtha, light oil (<249°C)	6.9	8.1	9.7
Heavy Distillate (>249°C)	24.1	24.1	27.3
Total Oil (C_5-heavy distillate)	35.5	38.3	41.8
SRC	27.6	24.8	20.0
Insoluble Organic Matter	6.0	6.5	5.0
Ash	9.0	9.0	9.2
H_2 Reacted			
gas balance	5.4	5.2	5.7
product analysis	4.1	4.3	4.9
MAF Conversion	93.4	92.9	94.5