**Coal Technology Program
Progress Report for July 1976**

MASTER

OAK RIDGE NATIONAL LABORATORY

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Printed in the United States of America: Available from
National Technical Information Service
U.S. Department of Commerce
5285 Port Royal Road, Springfield, Virginia 22161
Price: Printed Copy \$4.00; Microfiche \$2.25

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Contract No. W-7405-eng-26

COAL TECHNOLOGY PROGRAM
PROGRESS REPORT FOR JULY 1976

Date Published: September 1976

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OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee 37830
operated by
UNION CARBIDE CORPORATION
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ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION

MASTER

PREVIOUS REPORTS IN THIS SERIES

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ORNL/TM-5045, Progress Report for September 1974
ORNL/TM-5046, Progress Report for October 1974
ORNL/TM-4787, Progress Report for November 1974
ORNL/TM-4796, Progress Report for December 1974
ORNL/TM-4850, Progress Report for January 1975
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COAL TECHNOLOGY PROGRAM PROGRESS REPORT FOR JULY 1976

ABSTRACT

This report - the twenty-fourth of a series - is a compendium of monthly progress reports for the ORNL research and development projects that are in support of the increased utilization of coal as a source of clean energy. The projects reported this month include those for research in hydrocarbonization and residue carbonization, solids-liquid separation, in situ gasification, analytical chemistry, engineering studies and evaluations, coal-fueled MIUS, and materials.

1. SUMMARY

J. P. Nichols

Highlights of our progress in July are summarized below:

° During July the bench-scale hydrocarbonization system was modified to permit recirculating fluidized-bed operation. Experimentation will resume in August. Three residue carbonization runs were performed in July. All runs were terminated prematurely by plugs in the off-gas system, but two could be termed partially successful since no large agglomerates formed in the reactor.

° In solids-liquid separations work, a sealed filtrate weighing unit was installed in the bench-scale apparatus that will allow continuous print-outs of filtration rates at operating pressures downstream of the filter up to 150 lb/in.² Settling runs of 530°F with process solvents from the P & M-SRC pilot plant have shown excellent results. Twenty percent light process oil gave settling rates which were more than double those obtained with SRC-unfiltered oil (UFO) alone.

° In our supporting work for in situ gasification, we have discovered that high-temperature (>873°K) pyrolysis tests with large blocks of coal produce significantly greater quantities of gas than that produced by pyrolysis of powdered coal. This is thought to be the result of extensive steam-char reactions that take place as the steam generated at the core of the large block diffuses through the high temperature char bank to the block surface.

° In our analytical chemistry work stabilities of gas mixtures have been studied to determine the relative validity of dynamic and static gas analyses.

° Work was completed this month on the capital and operating cost estimates and the economic analyses in our conceptual design studies of commercial-scale Synthoil and Hydrocarbonization plants.

° The majority of the Incoloy 800 tubing for the Coal-Fueled MIUS furnace was received. The furnace bid period was extended to August 12 and regenerator specifications were under internal review. Work continued on analysis of the turbine electronic control system, design of building modifications, endurance testing of coal feed systems and a prototype feed nozzle, and supplemental studies.

° In our materials engineering work, we completed the assessment effort for the pressure vessel and piping technology project, continued to develop inspection techniques for wear and process-resistant-coatings, and continued conceptual design studies of prestressed concrete pressure vessels for gasifier applications.

2. HYDROCARBONIZATION/RESIDUE CARBONIZATION RESEARCH

H. D. Cochran, Jr.

2.1 Bench-Scale Hydrocarbonization

H. D. Cochran, G. L. Yoder, P. R. Westmoreland, and R. L. Andrews

2.1.1 Work Accomplished

Fabrication and Installation. Reactor internals, piping, valving, and instrumentation were installed during July to permit recirculating fluidized-bed operation. A 3/4-in.-diam, 29-in.-long draft tube was centered in the reactor 1-in. above the distributor plate. As in the atmospheric hydrocarbonization experiment, a cup-shaped deflector was mounted 10-in. above the top of the draft tube.¹ Hot hydrogen and pneumatically transported coal are fed upward into the bottom of the draft tube. An updated flowsheet will be presented in the August monthly report.

Operation. Data analysis continued during the hydrocarbonizer shutdown. Partial material balances and gas compositions for Runs HC-5 and HC-6 (Tables 2.1 through 2.5) were calculated. As further chemical analyses are made and are critically reviewed, final material balance for HC-1 through HC-6 will be issued.

Material balance calculations on hydrogen consumption in Runs HC-1 through HC-4 indicated that, within the precision of measurement, the hydrogen consumption was 0.0% of the weight (MAF) of coal fed; that is, there was neither significant consumption nor significant production of hydrogen. The precision of these calculations, $\pm 0.7\%$, is consistent with the precision of hydrogen flow measurement in the experiment. This result, 0.0% consumption at 300 psig and 910° - 1075°F, agrees with the prediction² based on correlation of published results.

2.1.2 Work Forecast

Sufficient coal remains for a shakedown experiment in the recirculating fluidized-bed reactor, the experiment being planned for early August. Upon receipt, fresh Wyodak coal will be ground, dried and sieved for subsequent experiments.

2.2 Residue Carbonization

H. D. Cochran, J. B. Gibson, and J. C. Rose

2.2.1 Work Accomplished

Three residue carbonization runs, RC-10, RC-11, and RC-12, and two

agglomeration tests were performed in July. The first residue carbonization run was at 1230°F, the second suffered from problems in the product recovery section and did not operate for a significant time, and the third was at 1200°F. Both agglomeration tests were at 1110°F.

Runs RC-10 and RC-12 could be termed successful from the standpoint of no large agglomerates in the reactor. Both runs were terminated after plugs developed in the 1/2 in. pipe leading from the reactor to the cyclone. Run RC-10 lasted 1 3/4 hr and RC-12 about 1 hr. In both runs, the draft tube had a 1 in. diameter, while in all previous runs the draft tube was 3/4 in. It is believed that the larger draft tube is significant in the solution to the agglomeration problem in the reactor, but the extra gas required for operation probably contributes to the greater solids carryover and plugging downstream. In these runs, the feed nozzle was flush with the distributor plate, the feed rate was 2 lb/hr of a 4:1 mixture of Wyodak char to residue, and the initial bed was 3.3 lb of Wyodak char. Particle size in the feed was -40 to +200 mesh with an average size of 165 microns. The fluidizing gas was argon with a velocity of 0.42 ft/sec (39 times minimum fluidizing velocity of the feed particles) in the downcomer region and 4.2 ft/sec in the draft tube. Average particle size of the product char was 430 microns in both runs.

Both of the agglomeration tests were performed with mixtures of Wyodak char and residue in the ratios of 2:1 and 9:1. Agglomeration in both tests was slight and the agglomerates were softer and more porous than those formed in tests with residue alone. The total weight of agglomerates recovered was less than the weight of the residue in the feed mixture for both cases.

2.2.2 Work Forecast

More runs are planned with the residue carbonizer in order to determine conditions which allow a longer operation time (less solids carryover) and a higher solids feed rate. Redesign of the product recovery section and the reactor are being considered in order to achieve better operation.

2.3 References for Section 2

1. J. P. Nichols, Coal Technology Program Quarterly Progress Report for the Period Ending March 31, 1976, ORNL-5159 (July 1976), p. 15.
2. J. M. Holmes, et al, Hydrocarbonization Research Phase I Report: Review and Evaluation of Hydrocarbonization Data, ORNL/TM-4835 (August 1975), p. 80.

Table 2.1. Preliminary material balance for Experiment HC-5.

Average bed temperature: 965°F
 Total pressure: 300 psig
 H₂ feed rate: 0.36 scf/lb maf coal

Coal feed rate
 As-received: 15.5 lb/hr
 Moisture-and-ash-free: 12.9 lb/hr

	Mass, g	Carbon fraction	Ash fraction	Sulfur fraction	Percent of carbon fed	Percent of ash fed	Percent of sulfur fed
<u>Input</u>							
1) Coal	54625	.6107	.0634	.00375	100.0%	100.0%	100.0%
2) Scrubber water	18900	0	0	0	0	0	0
<u>Output</u>							
3) Char recovered	25850	.8030	.1044	.0034	62.2	77.9	43
4) Scrubber top phase	122.6	.7848	.0413	.0026	.289	.146	.15
5) Scrubber middle (aqueous) phase	32470	-- ^a	--	--	-- ^a	--	--
6) Scrubber bottom phase	2429.9	.8316	.0005	.0020	6.06	.351	2.4
7) Cold trap top phase	167.0	.4436	.0032	.0028	.22	.015	.23
8) Cold trap middle (aqueous) phase	307.4	-- ^a	--	--	-- ^a	--	--
9) Cold trap bottom phase	C	--	--	--	--	--	--
10) Miscellaneous tars	440.	.8316 ^b	.0005 ^b	.0020 ^b	1.10	.006	.43
11) Gases	12304	.572	--	--	21.10	--	53 ^c
<u>Rinses</u>							
12) Scrubber - water	66010	-- ^a	--	--	-- ^a	--	--
13) Scrubber and cold trap - trichloroethane	43920	.00503	.00052	.000041	.662	.65	.89
Overall balances					91.7 ^a	79.1	100

^aIncomplete; water analysis not yet available.

^bData estimated from analysis of similar product.

^cCalculated by difference.

Table 2.2. HC-5 effluent gas composition at steady state
at 9:42 p.m. June 7, 1976

Component	Effluent flow rate (scfm)	Composition (%)	H ₂ and N ₂ -free composition
CH ₄	0.348	6.33	43.9
C ₂	0.111	2.02	14.0
C ₃	0.056	1.02	7.1
CO	0.132	2.40	16.6
CO ₂	0.143	2.60	18.0
H ₂ S ^a	0.003	0.05	0.3
H ₂	4.71	85.6	----
N ₂	0.000	0.0	----

^a Near low limit of sensitivity; accuracy questionable.

Table 2.3. Preliminary material balance for Experiment HC-6.

Average bed temperature: 1030°F
 Total pressure: 150 psig
 H₂ feed rate: 0.18 scf/lb maf coal

Coal feed rate
 As-received: 16.3 lb/hr
 Moisture-and-ash-free: 13.6 lb/hr

	Mass, g	Carbon fraction	Ash fraction	Sulfur fraction	Percent of carbon fed	Percent of ash fed	Percent of sulfur fed
<u>Input</u>							
1) Coal	31900	.6025	.05665	.00325	100.0%	100.0%	100.0%
2) Scrubber water	24140	0	0	0	0	0	0
<u>Output</u>							
3) Char recovered	15530	.7992	.103	.00273	64.6	86.3	40.9
4) Scrubber top phase	Trace	--	--	--	--	--	--
5) Scrubber middle (aqueous) phase	28900	-- ^a	--	--	-- ^a	--	--
6) Scrubber bottom phase	1490	.8322	.0034	.0012	6.44	.28	1.8
7) Cold trap top phase	133.7	.6837	.0463	.0040	.456	.343	.52
8) Cold trap middle (aqueous) phase	75.2	-- ^a	--	--	-- ^a	--	--
9) Cold trap bottom phase	0	--	--	--	--	--	--
10) Miscellaneous tars	413	.8322 ^b	.0034 ^b	.0012 ^b	1.79	.077	.48
11) Gases	7201	.450	--	--	19.19	--	54 ^c
<u>Rinses</u>							
12) Scrubber - water	50635	-- ^a	--	--	-- ^a	--	--
13) Scrubber and cold trap - trichloroethane	61280	.00577	.000449	.000046	1.84	1.52	2.7
Overall balances					94.3 ^a	88.6	100

^aIncomplete; water analysis not yet available.

^bData estimated from analysis of similar product.

^cCalculated by difference.

Table 2.4. HC-6 effluent gas composition at steady state
at 4:46 p.m., June 22, 1976

Component	Effluent flow rate (scfm)	Composition (%)	H ₂ and N ₂ -free composition
CH ₄	0.269	7.34	40.8
C ₂	0.082	2.24	12.5
C ₃	0.037	1.01	5.6
CO	0.106	2.89	16.1
CO ₂	0.160	4.36	24.2
H ₂ S ^a	0.005	0.14	0.8
H ₂	2.97	81.0	
N ₂	0.037	1.01	

^aNear low limit of sensitivity; accuracy questionable.

Table 2.5. Summary of preliminary material balances
for experiments HC-5 and HC-6

Experiment	HC-5	HC-6
Average bed temperature, °F	965	1030
Reactor pressure, psig	300	150
Carbon balance (% of carbon fed)		
Char	63.2	66.2
Liquids	7.39 ^a	8.93 ^a
Gases	21.1	19.2
Overall	91.7 ^a	94.3 ^a
Overall ash balance (% of ash fed)	79.1	88.6
Sulfur balance (% of sulfur fed)		
Char	44	42
Liquids	3.4	4.4
Gases	53 ^b	54 ^b
Overall mass balance (%)	100.8	95.9
Oil yield (weight % of maf coal)	6.7	8.0

^aIncomplete pending carbon analysis of aqueous samples.

^bCalculated by difference from 100%.

3. SUPPORTING RESEARCH AND DEVELOPMENT IN SEPARATIONS TECHNOLOGY

B. R. Rodgers and D. A. McWhirter

3.1 Characterization Tests

3.1.1 Ageing

Analyses of SRC-Filtered oil (FO) after 8 months ageing under various storage conditions shows that the FO continues to be stable. However, the unfiltered oil (UFO) analyses (4 months ageing) showed that storage at 140°F lowers the percentage of carbon and hydrogen with a corresponding gain in oxygen and solids content. Since the FO did not indicate oxygen or solids gain, the solids in the UFO may act as a catalyst toward oxidation. Further analyses may provide information to elucidate this mechanism.

3.1.2 Distillation

SRC process derived solvents were subjected to distillation to separate wide boiling point cuts for settling experiments. Process solvent (slurry oil) had 38% boiling in the 500-600°F range and 58% boiled > 600°F. Wash solvent boiled 30% in the 300-400°F range and 67% in the 400-500°F range, while light oil boiled 73% in the 200-300°F range and 21% in the 300-400°F range. After settling experiments are performed to determine the more promising materials, narrower boiling points cuts will be examined as settling accelerators.

The density of wash solvent was found to be 0.958 gm/cc, and the light oil density was 0.786 gm/cc at 25°C.

3.2 Bench-Scale Tests

3.2.1 Settling of SRC-UFO/Solvent Mixtures

Settling tests with UFO containing various solvents at a 20% concentration, including two process solvents from the SRC process at Ft. Lewis, Washington, were carried out in the modified bench-scale settler. Rates were considerably improved over UFO alone for all solvents, but particularly for the light oil process solvent and decane. Some typical rates were: toluene = 11 in./hr, light oil = 15 in./hr and decane = 22 in./hr. However, a considerable increase in the solids content of the mixture occurred with decane addition, and the increased solids load on the process may not be desirable. For instance, in a commercial process the underflow would typically feed a carbonizer or gasifier and the cost of treatment of additional solids in this step would have to be examined. To quantify the solids increase with the addition of solvent, 1.2 μ Millipore vacuum

filtrations were run using mixtures of UFO with both toluene and decane. The results, after drying the collected solids to a constant temperature at 170°C, showed that for the 20 wt % solvent mixture, the solids content of the neat UFO (6.8 wt %) was increased to 17.8 wt % of UFO by decane, but only slightly to 8.3 wt % of UFO by toluene.

3.2.2 Modification of the Bench-Scale Filter

Due to the high filtrate vapor losses during filtration at elevated temperatures, a high pressure filtrate receiver was designed, constructed, and installed on the bench-scale facility. The heart of the weighing system is a Statham strain gauge and Universal transducer. The unit has been calibrated and shakedown runs will begin next month. Initial experiments are planned to verify the lab-scale coal precoat results.

4. EXPERIMENTAL ENGINEERING SUPPORT OF AN IN SITU GASIFICATION PROCESS

R. C. Forrester III

4.1 Large-Block Pyrolysis Studies

R. C. Forrester III and G. D. Owen

Five kilogram blocks of Wyodak subbituminous coal have been pyrolyzed at 1173°K using heating rates of 3°K/min and 0.3°K/min. At the conclusion of both experiments, exposure of the remaining char to air resulted in self-heating to temperatures as high as 488°K although ignition did not occur.

Alteration of the data reduction code was also completed and detailed analyses of the gases produced by previous pyrolysis tests have been calculated. One of the more interesting results of these calculations is the discovery that high-temperature pyrolysis tests ($> 873^{\circ}\text{K}$) with large blocks produce significantly greater quantities of gas than that produced by powder pyrolysis using similar heating rates and the same temperature maximum (Fig. 4.1). Observed levels of gas production are thought to be the result of extensive steam-char reactions which take place as the steam generated at the core of the block diffuses through the high-temperature char bank to the block surface. Later experiments with dried blocks will test this thesis.

4.2 ERDA's UCG Program

R. C. Forrester III

A summary of results produced by the large block pyrolysis experiments is being prepared for presentation at the 2nd Annual Underground Coal Gasification Symposium which is to be held at the Lakeview Inn and Country Club, Morgantown, West Virginia, August 10-12, 1976. The paper is entitled, "Two Dimensional Studies of Coal Pyrolysis: Preliminary Results".

Fig. 4.1. Total gas production as functions of maximum pyrolysis temperature and heating rate.

*See, for example, Mazumdar and Chatterjee, "Mechanism of Coal Pyrolysis in relation to industrial practice," Fuel, 52(1), 11 (1973).

5. ANALYTICAL CHEMISTRY

W. R. Laing

5.1 Stabilities of Gas Mixtures Relevant to
Coal Conversion Technologies

A. D. Horton and B. R. Clark

A continuing study is being carried out in collaboration with members of the Environmental Sciences Division (S. McLaughlin and D. Shriner) to develop a model exposure system for subjecting plants to a simulated synfuel stack gas emission. Uptake of model gases and the effects upon plants will be studied for single gases and gas mixtures.

Measurements of gas concentrations under dynamic operating conditions with the exposure chamber have been completed. Static tests are being carried out to determine what, if any, chemical interactions occur in the model gas mixtures. To achieve this, the exposure system was operated dynamically until the gas mixtures reached steady state levels. Then the blowers and gas supplies were cut off simultaneously, and samples of the stagnant gas in the chamber were taken at intervals of 10, 40, and 85 minutes. Only the hydrocarbons in the sample were determined.

Table 5.1 shows the change in gas chromatographic peak heights versus time during the test. A possible explanation of the increase in concentration is the migration of the heavier gases (benzene, xylene, and phenol) to the bottom of the chamber. (Samples are withdrawn from a tube at the bottom of the chamber.) However, this does not explain the marked increase in the lighter gases, methane, and ethylene, nor does it explain why xylene concentration increased only slightly and phenol remained constant. Hydrogen sulfide and carbonyl sulfide were present in the mixture, and these will be determined at a later date.

A better test of the static mixture may be obtained by trapping the sample in a glass bulb when the chamber is operating in the dynamic mode. Changes in the components can then be determined hourly over a longer period of time with a minimum of effects due to migration, stratification or natural room air currents.

Table 5.1 Peak Heights, mm

	<u>Under Constant</u> <u>Dynamic Conditions</u>	<u>After Stagnation</u>			<u>Starting Concen-</u> <u>tration, ppm</u>
		<u>10 min</u>	<u>40 min</u>	<u>85 min</u>	
CH ₄	191	239	258	242	4.5
C ₂ H ₄	49	62	107	102	2.3
C ₆ H ₆	116	127	173	184	2.5
C ₆ H ₄ (CH ₃) ₂	48	50	59	53	1
C ₆ H ₅ OH	9.5	11	11	11	0.8

An attempt was made to determine the stability of a real coal conversion by-product gas mixture stored in glass. Unfortunately the samples were collected in such a manner that most sulfur components were absent and, therefore, expected reactions with sulfur compounds could not occur.

Three samples of gas, obtained from Pittsburgh Energy Research Center and described as "tail gas", were received July 9, 1976, about 4:00 p.m. The samples were collected in 250 ml gas sample bulbs by water displacement. Each sample was analyzed for sulfur components, gaseous hydrocarbons and CO and CO₂. A 1/8-in. x 15-ft FFAP column was used for sulfur analyses, a 1/4-in. x 8-ft Silica gel - 3% squalene column was used for gaseous hydrocarbons, and the nickel from Amberlyst 15 column was used for the determination of CO and CO₂.

No sulfur components were found at the lower limit of detection (<0.1 ppm as H₂S); carbon dioxide was below the limit of detection (<0.05%). The lack of measurable quantities of sulfur compounds is easily explained by the manner in which the samples were collected, i.e., by water displacement from the sampling bulbs. The high solubilities of such compounds would lead to severe losses.

Table 5.2 shows the results obtained for gaseous hydrocarbons in samples collected at 10:00 a.m., 11:00 a.m., and 12:30 p.m. Analyses were repeated July 12, 1976.

Table 5.2. PERC Tail Gas Samples

Source Samples at: Analysis Made on:	<u>Volume Percent</u>				
	10:00 a.m. <u>7-9-76</u>	10:00 a.m. <u>7-12-76</u>	11:00 a.m. <u>7-12-76</u>	12:30 p.m. <u>7-9-76</u>	12:30 p.m. <u>7-12-76</u>
CH ₄	12.1	12.4	13.3	14.5	14.2
C ₂ H ₆	3.4	3.5	3.7	4.2	4.1
C ₃ H ₈	2.5	2.6	2.7	3.0	3.0
iso C ₄ H ₁₀	0.16	0.15	0.16	0.18	0.16
n C ₄ H ₁₀	1.00	1.02	1.04	1.27	1.10
C ₃ H ₆	0.10	0.09	0.09	0.15	0.11
iso C ₅ H ₁₂	0.14	0.14	0.15	0.18	0.16
n C ₅ H ₁₂	0.24	0.23	0.26	0.31	0.28
CO	0.11		0.10		0.13
CO ₂	<0.05		<0.05		<0.05
Sulfur Compounds	All Samples <0.1 ppm				

5.2 ANALYTICAL SERVICES

W. R. Laing and L. J. Brady

A total of 503 samples was submitted for analysis as compared to 210 for the previous month. Equipment from the SRC solids-liquid separation tests is back in operation. This resulted in the increase in samples submitted for test this month.

Twelve samples from hydrogenation tests were analyzed. Proximate and ultimate analyses were made on nine of these samples which were either coal or char samples. The ash ranged from 5.7 to 14.4 percent, and carbon, on a moisture and ash free basis, ranged from 72.2 to 91.8 percent. The calorific value ranged from 12,300 to 15,000 Btu per pound.

It appears that the difficulty which was causing the timer on the integrator to malfunction has now been corrected. It will now be possible to do simulated boiling range tests on those samples that have been on the backlog.

One set of gas samples was analyzed for CO₂ content on the Perkin Elmer Model 3920-B gas chromatograph.

6. ENGINEERING EVALUATIONS OF THE SYNTHOIL AND HYDROCARBONIZATION PROCESSES

J. M. Holmes, R. Salmon, and E. G. St. Clair

6.1 Synthoil Process

R. Salmon, E. G. St. Clair, M. S. Edwards,
W. G. Ulrich, and D. A. Dyslin

Work was completed on the capital and operating cost estimates for the Synthoil facility. The economic analysis of the facility also was completed. Product price was calculated as a function of coal price and rate of return on equity, for various debt-equity ratios. The results are being prepared for inclusion in the final draft report. Work on the report is expected to be completed during the month of August, 1976.

In the area of cost estimation, the following work was performed this month:

1. the design and cost estimation of Unit 51, the industrial water system, was completed;
2. the costs used for a power plant (required for steam generation) with stack gas cleanup were verified through vendor contacts and open literature sources;
3. all other off-site facilities were also cost estimated;
4. the capital cost for the entire Synthoil facility was finalized; and
5. the factor method used to develop capital costs was verified by an in-house estimation method.

Equipment lists were completed for all process units and are now in final form. Revisions were made to assure that consistent nomenclature for materials of construction was used, in addition to other minor changes and corrections.

6.2 Hydrocarbonization Process

J. M. Holmes, D. A. Dyslin, M. S. Edwards,
D. S. Joy, G. R. Peterson, and C. B. Smith

The design and cost evaluation of a hydrocarbonization facility for the production of clean fuel equivalent to a nominal 100,000 bbl/day of fuel oil continued. The capital cost estimate of the facility, including

the cost of the fluid bed combustor (FBC), was completed. Information concerning the capital cost of the FBC modules developed by Foster Wheeler Corporation in their design study was expanded to include the cost of a building to enclose the FBC modules, equipment pads, structural steel, a fly ash conveying system, coal-limestone handling equipment, and other equipment items required for the FBC unit. The total capital cost for the FBC unit was estimated at \$245,332,000, including a package boiler for startup purposes.

Operating cost estimates for the complete hydrocarbonization facility were also completed, and the economic feasibility is being determined using a discounted cash flow procedure in which coal price, debt/equity ratios, and rate of return on equity are varied over a given range.

An alternative to the basic flowsheet where the coal tar and oil produced during hydrocarbonization is marketed directly at its fuel value without hydrotreating is being evaluated. Results indicate that there is a significant reduction in capital cost of the facility which results in a lower overall cost for the product mix. However, further study to establish realistic market values for the crude tar and oil products is recommended.

A project schedule based on a 60-month design and construction period for the facility was prepared. The schedule was arranged so that the design, procurement and construction of critical units such as the hydrocarbonization and tar hydrotreating units would continue throughout the period. However, in order to maintain the peak construction manpower under 3000 workers, the construction of several units was delayed until the latter part of the project.

7. COAL-FUELED MIUS

A. P. Fraas and W. R. Mixon

This project for analysis, design, and demonstration of a concept utilizing a fluidized-bed coal combustion system as a heat source for a gas turbine generator suitable for applications in Modular Integrated Utility Systems (MIUS) is carried out under the ORNL-HUD-MIUS Program within the Energy Division. Work is supported by the U.S. Department of Housing and Urban Development under HUD Interagency Agreement No. IAA-H-40-72 and by the Energy Research and Development Administration, Office of Fossil Energy (formerly Office of Coal Research, Department of the Interior), under ERDA Contract No. E(49-18)-1742. The project consists of four phases: I - Conceptual Preliminary Evaluation; II - Conceptual Design; III - Detailed Design and Construction; and IV - Shakedown, Performance, and Endurance Tests.

7.1 Furnace Procurement

The furnace bid deadline date was extended to August 12 at the request of vendors who could not meet the original deadline. One bid was received and bids are expected from at least three other firms.

Most of the Incoloy 800 tubing to be included in the furnace tube bundles was received.

7.2 Turbine-Generator Unit

Analysis of the electronic control system continued in preparation for design of turbine instrumentation and controls. Procurement of recuperator modules and placement of the turbine modification contract are awaiting receipt of additional funds.

7.3 Regenerator

Specifications for the flue-gas combustion-air regenerator are currently under internal review.

7.4 Site Preparation

Work continued on the design of modifications of Building 9401-1 and on drawings for relocation of existing equipment.

7.5 Cold Flow Tests of a Fluidized Bed

The small cold flow model was filled with a special batch of smaller mean particle size limestone for heat transfer and fluidizing tests.

7.6 Coal Metering and Feed System

Endurance tests were continued on the flow splitter feed system and on a prototype coal feed nozzle. No problems have been observed during cumulative operating times of 155 hr on the feed system and 75 hr on the nozzle.

The Vibra-screw coal feeder was tested and excessive air leakage along the screw was found for a pressure differential as low as 1 to 2 psi. The screw feeder provides a constant coal feed rate without the cyclic variation that has been observed with the rotary feeder, but will not provide an air seal at the pressure differential required for driving the coal transport air ejectors. Consideration is being given to using the screw feeder in series with the rotary feeder.

7.7 Ash Handling System

Work began on design of the ash handling system. Recommendations for preferred types of components were obtained in discussions with several manufacturers, and a flowsheet incorporating the recommended components was prepared. The option of having the ash handling system designed and supplied by a systems engineering firm was explored, but a preliminary estimate indicated that this approach would be much more expensive than ORNL design and procurement.

7.8 Supplemental Studies

A preliminary ORNL analysis on coal, limestone, and ash handling for a coal-fueled MIUS was completed. No serious social or environmental problems were found; in fact, the effects of municipal solid waste collection and disposal practices are far greater than that for handling fuel and wastes of the coal-fueled MIUS. Costs for coal-related solids handling amount to less than 4% of owning and operating costs of the MIUS installation and should not place a serious burden on the MIUS concept.

The corrosion test specimens are ready for welding to form the tubes to be installed in the Fluidyne furnace.

The instrumented tube and other components for the heat transfer test have been shipped to Fluidyne. The heat transfer tests are now scheduled to begin about August 23.

Internal review of the report on the reliability of coal-fired gas turbine systems was completed and the revised draft was submitted for program review.

The analysis of system performance with alternate cycles was completed and a draft report was near completion.

8. MATERIALS

W. R. Martin and D. A. Canonico

The materials engineering and supporting technology reported herein are in support of activities directed by Materials and Power Generation, Division of Fossil Energy Research. Other related work not funded directly by this division of ERDA/FE is included also.

Summary

The assessment effort for the Pressure Vessel and Piping Technology Program was completed. Sections of the report are being retyped. Those sections completed have been transmitted to Fossil Energy Research for review.

Inspection techniques for wear and process-resistant coatings are being developed. Computer calculations were completed to indicate inspection parameters for thermal examination of coating bond quality. Because existing liquid penetrant emulsifiers are unacceptable for ceramic coatings, various solvents have been tested which better remove excess penetrant and thereby reduce background indications caused by porosity.

Conceptional design work continued in our Prestressed Concrete Pressure Vessel Program. Emphasis was given to selecting a top-head closure design to be used in HYGAS and Synthane gasifiers. Other design work is continuing on a study of various circumferential tendon arrangements, manway penetrations, and those efforts to finalize the conceptional design of each gasifier.

8.1 Pressure Vessel and Piping Technology Assessment

D. A. Canonico, R. H. Cooper, R. K. Nanstad, G. C. Robinson, and G. M. Slaughter

A review of the piping and pressure vessel needs for coal conversion systems is in progress. The program will identify those areas where additional material property data needs are required in order to assure that the pressure boundary components in conversion systems can be designed, fabricated, and operated in a safe and reliable manner.

Sections of the report have been reviewed, edited, and have been transmitted to Fossil Energy Research for their review.

8.2 Inspection Techniques for Wear- and Process-Resistant Coatings

R. W. McClung and G. W. Scott

8.2.1 Specimens and Standards

Crack Specimens. The first artificial-crack specimen for liquid penetrant experiments has been coated with ZrO_2 and tested with penetrant in the as-coated (i.e., no stress history) condition. It will be stressed in a tensile machine to various loads, first in the elastic deformation region for the substrate, and later, if necessary, in the plastic deformation region for the substrate. After each stress test, the stress will be removed and the specimen will be inspected visually and with penetrant for the formation of cracks.

Coating Thickness Standards. Twenty-four standard blanks have been machined and dimensionally inspected. One blank is being used to check out the spray fixturing. Spraying of blanks will begin soon.

8.2.2 Thermal Testing (W. A. Simpson)

We are continuing to investigate the applicability of thermal evaluation techniques to the detection of nonbonds between coating and the substrate. The problem has been simulated on a computer using a generalized heat conduction code, and a number of runs with different heat inputs have been obtained. As expected, the calculations show that the maximum thermal contrast develops very quickly (within 20 msec) and the rates of introduction of heat to the surface will probably have to be of the order of $0.1 \text{ BTU} \cdot \text{in.}^{-2} \cdot \text{sec}^{-1}$ or greater. This value may vary somewhat depending on the area of the unbonded region, but it yields a maximum thermal contrast of about 12°F for a region having a diameter of 0.100 in. and for a coating thickness of 0.010 in.

We are investigating the possibility of modifying the computer code to allow for a localized moving heat source which would then scan the sample in a raster-like pattern.

8.2.3 Liquid Penetrant Testing (S. D. Snyder)

We have continued process experiments on application of penetrants to porous ceramic coatings. Various solvents have been substituted for the standard penetrant emulsifiers to speed up the removal of excess penetrant from surface porosity. More aggressive, less viscous solvents remove the penetrant more rapidly, but are more difficult to control, i.e., timing of specimen exposure to the solvent is more critical. The optimum choice will be a compromise between speed and control, and may also be affected by factors such as contrast between various types of indications (i.e., cracks versus background porosity).

8.2.4 High Voltage (Corona Discharge) Probe Testing

Two additional probe designs have been evaluated, and a salvaged DC voltage source has been substituted for the AC (tesla coil) source initially used. When the DC source is set at a voltage somewhat less than that required to break down the air column in an artificial defect, a change in current flow is observed when the probe passes over such a defect. Apparently, an air gap between the probe and substrate conducts more current than a gap which is partly dielectric material and partly air. If this current effect can be exploited, it may allow a test method which does not require an arc, thereby diminishing the arc's undesirable characteristics (i.e., noise, ozone production).

8.3 Iron and Nickel Carbonyl Formation and Prevention

J. Brynestad and J. H. DeVan

The furnace assembly for the reaction rate apparatus has been finished and calibrated at 260, 200, and 150°C. The overall temperature deviation within the autoclave (5 in. ID x 26 in. length) was less than $\pm 2^\circ\text{C}$.

The system design has been approved by the High Pressure Vessel Review Committee. We are still awaiting the delivery of pressure relief valves, but we expect to start preliminary experiments in mid-August.

8.4 Failure Analysis of Materials and Components

D. A. Canonico, D. P. Edmonds, and T. K. Roche

This project is devoted to the posttest examination of components that have been removed from service in coal liquefaction processes. The goal is the avoidance of failures in commercial coal conversion systems.

The type 316 stainless steel heater tube from Project Lignite is being examined to determine the possible sources of the stress associated with the stress-corrosion failure. In assessing the probability lifetime of substitute heater materials, such as I-800, it is important to know if there were high residual, membrane, or cyclic loads.

The final report from the posttest examination of the pressure let-down valve from the Synthoil Plant at PERC is being prepared.

8.5 Prestressed Concrete Pressure Vessel Studies

W. L. Greenstreet

The objective is to investigate the potential use of prestressed concrete pressure vessels (PCPV) for coal conversion processes, to identify major problem areas, and to define and outline a test program (or programs) for feature and concept demonstration. Conceptual designs of pressure vessel and liner combinations for commercial size systems are to be developed and studies as vehicles for assessment and guidance.

Conceptual design work was continued as the main activity for both the HYGAS and Synthane gasifiers. Emphasis was given to selecting a top head closure design to be used for both vessels, to refractory design investigations, and to temperature distribution studies for the refractory-liner-vessel interface region. The latter will have important bearing on vessel dimensions and details of design.

Under Task I, a flat-plate head, which will be held down by toggle bolts, was selected for the HYGAS and Synthane vessels. Contributing to this selection was development of suitable vertical tendon arrangements for satisfactory load transfer from the toggle bolts and location of the manway for access to the upper region of the Synthane vessel in the cylindrical portion.

Under Task II, a study of various circumferential tendon arrangements was carried out, along with investigations of tendon strengths, preloads, and preloading techniques. The most suitable circumferential prestressing arrangement is based on wire winding, with gaps being left in the winding at penetration locations. Axial prestressing arrangements for the two vessels were also developed.

Under Task III, manway penetrations are being given more detailed considerations. Involved are basic decisions related to the location of the vessel closure plate, that is, location at the liner diameter or at the outside diameter of the structural concrete. The details of the designs affect the discontinuity and local stresses in both the liner and the structural concrete.

Relatively detailed finite element analyses are being conducted to finalize vessel dimensions and to examine stresses in the head regions. These analyses will be continued concurrently with the detailed heat transfer analyses now under way.

The liner and refractory studies under Task IV are now addressed to improving original design numbers and to providing the details necessary for conceptual design finalization. Specific refractory material selections are being made, directly applicable properties data are being obtained, and methods for support are being detailed. The insulating concrete requirements for the hot liner concept have been established and a materials examination made.

The thermal analyses will give data on types and thicknesses of materials and on cooling circuit requirements. The latter include cooling tube spacing and temperature increases and flow rates for the water coolant. Both hot and cold liner concepts are under study.

Thermal stress computations and fatigue behavior examinations are to be conducted. These considerations are especially important for the hot liner concept.

8.6 Other Related Work

W. W. Harris, R. H. Cooper, Jr., and J. H. DeVan

8.6.1 Fluidized Bed Materials Support Activities

Fireside Corrosion in a Fluidized Bed Coal Combustor. Surveillance coupons of four candidate iron-nickel-chromium alloys exposed in the Combustion Power Company, Inc., Menlo Park, California, atmospheric fluidized bed combustor have been examined. The four alloys, type 316 stainless steel, type 310 stainless steel, Incoloy 800, and Incoloy 825 were exposed in the gas plenum immediately above the bed for 500 and 1000 hr at 1575 to 1590°F at atmospheric pressure. Additives to the fluidized bed were dolomite and clay.

The specimens were examined by scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS), electron spectroscopy chemical analysis (ESCA), electron probe microanalysis (EPMA), optical metallography, and x-ray diffraction. These examinations showed no indications of sulphidation or carburization effects in any of the materials examined. ESCA results confirmed that the binding energies of all metallic components in the surface scale existed in oxidized forms. X-ray diffraction indicated that the scale and near-surface regions on all coupons consisted of a FCC matrix, CaSO_4 , spinels of slightly varying spacings, Cr_2O_3 structures. The 316 stainless steel specimens showed relatively heavy oxide scales, while oxidation of the higher alloy materials was primarily intergranular. A topical report covering these observations has been written and is being reviewed.

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