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Coal Technology Program
Progress Report for March 1976

OAK RIDGE NATIONAL LABORATORY
OPERATED BY UNION CARBIDE CORPORATION FOR THE ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION

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PROGRESS REPORT FOR MARCH 1976

MAY 1976

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

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COAL TECHNOLOGY PROGRAM PROGRESS REPORT FOR MARCH 1976

ABSTRACT

This report — the twentieth 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 hydrocarbonization research, solid-liquid separations, in situ gasification, analytical chemistry, engineering evaluations of the Synthoil and Hydrocarbonization processes, coal-fueled MIUS, materials, and engineering evaluations of nuclear process heat for coal conversion.

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1. SUMMARY

J. P. Nichols

Highlights of our progress in March are summarized below:

° In the Hydrocarbonization Research program, shakedown tests of the bench-scale system were successfully completed with hydrogen. Wyodak coal was fed to the reactor at a rate of 9.9 lb/hr where it was hydrocarbonized at 1050°F under 20-atm hydrogen pressure. The system is scheduled for routine operation in the near future.

° The Supporting Research and Development in Separations Technology laboratory results include laboratory settling tests, bench-scale settling tests, and sample ageing tests. Two of ten compounds tested with the laboratory-scale apparatus were effective in increasing settling rates of solids in Solvent Refined Coal unfiltered oil. Bench-scale tests concentrated on one of the more promising inorganic additives from the laboratory-scale tests. Twelve runs at various concentrations failed to show any improvements in the settling rate over the untreated SRC-UFO.

° Experimental Engineering Support of an In Situ Gasification Process laboratory examination of the pyrophoricity phenomenon observed with chars produced by low-temperature block pyrolyses is continuing. These experiments have confirmed early indications that coal obtained from the LERC UCG field test site produces a less reactive char upon pyrolysis than those taken from the Roland-Smith seams near Gillette, Wyoming.

° The Analytical Chemistry participation comprised of the removal and concentration of organic components in by-product waters from fossil fuel conversion processes. A sephadex gel is being used to achieve hydrophilic-lipophilic separations in organic mixtures as a step in the analysis of fossil fuel related materials.

° Engineering Evaluations of the Synthoil and Hydrocarbonization Processes continued with the Synthoil process flow diagrams, heat and material balances, and utilities requirements being completed. A preliminary version of a computer code for cost estimation by the factor method is now operational.

The Hydrocarbonization overall utility requirements were estimated and balances for steam, fuel gas, water, and electric systems were obtained. A preliminary cost estimate of $24.59 million for the hydrotreater section was made, and agrees closely with the Hydrocarbon Research, Incorporated, estimate of $24.35 million. The Fluidized Bed Combustion Company completed their first-month design effort and prepared a solids and gas flow schematic diagram, a preliminary plant layout, and an equipment arrangement drawing for one fluidized bed combustion module.

° The Materials effort continued in the development of inspection techniques for the wear- and process-resistant coatings. The x-ray fluorescence data obtained on specimen ANL-C5 were correlated to variations in the composition of the coating. Additional coated samples were prepared consisting of NiCr and ZrO_2.

A wet chemical method for quantitative analysis of iron carbonyl in synthesis gas was tested and perfected, resulting in a detection limit of 3 ppb.

An evaluation of the 316 stainless steel heater coil from Project Lignite was begun.

° In Coal-Fueled MIUS, orders were placed for the Incoloy 800 tubing and a smaller quantity of Inconel 600 tubing for the tube matrix in the fluidized bed. Drawings and specifications for the furnace bid package were completed and are undergoing final review. Recommended gas turbine modifications for operation under the MIUS unit design conditions were received from AiResearch. Tests are continuing on the mixing rate of coal in a fluidized bed and on the coal feed system.

° Engineering Evaluations of Nuclear Process Heat for Coal Conversion engineering feasibility review of General Atomic's proposal to ERDA for a bench-scale test program on thermochemical water splitting for hydrogen production was completed.

2. HYDROCARBONIZATION RESEARCH

H. D. Cochran, Jr.

Summary

Several shakedown tests of the bench-scale system were completed successfully with hydrogen including one in which a small amount of Wyodak coal was smoothly and successfully fed to the reactor at 9.9 lb/hr where it was hydrocarbonized at 1050°F under 20-atm hydrogen pressure. The system is ready to begin routine operation.

2.1 Experimental Development

P. R. Westmoreland, J. B. Gibson,
R. L. Andrews, and J. C. Rose

Ambient Mock-Ups

There was no effort in this area to be reported during March.

High-Temperature Studies

A final carbon balance of 103.1% was determined for Run AHC-11, batch atmospheric hydrocarbonization in a recirculating fluidized bed at 1020°F. Liquid yield was 11.1% compared to 12.2% and 11.2% in Run AHC-8 and Run AHC-9. However, about half the coal charged in Run AHC-11 fell below the distributor plate and so was incompletely hydrocarbonized. Volatile content of char from Run AHC-8 was 14.8% and of char from Run AHC-9 was 16.3%, while composite "char" from Run AHC-11 had a volatile content of 26.7%. Volatile content of feed coal for each run was 46.4%. Thus, on the basis of coal completely hydrocarbonized in the recirculating fluidized bed, liquid yield appears to have been higher than yields from batch, uniformly fluidized bed hydrocarbonization.

2.2 Bench-Scale System

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

Design and Review

Design of modifications to the bench-scale system have continued in three important areas: (1) specifications for the hydrogen booster compressor, (2) design of the 80-atm reactor, and (3) design of the 80-atm preheater. Detailed design specifications for the hydrogen booster compressor have been completed and are undergoing internal review. The specifications will be released for bids within 2 weeks.

The most challenging design problem associated with the 80-atm reactor concerns the design of penetrations through the bottom flange of the reactor. The reactor consists of a 10-in. Sch 80 cold wall pressure shell with internal insulation and a heated reactor core. The flange penetrations for two hot hydrogen supply lines, a coal feed line, a product gas effluent line, a char overflow line, electrical connections for the internal heaters, and instrument taps for thermocouples and pressure signals present difficult layout problems within the limited space and problems of thermal stresses from steep temperature gradients. A preliminary layout of the bottom flange assembly showing the coaxial penetration for two hot gas supply lines and the coal feed line has been completed. Heat transfer and thermal stress analysis of this assembly are currently underway.

As a result of recent visits to PERC and Coalcon experimental facilities, we are considering the use of a coiled tube in a molten lead bath in the 80-atm preheater design. Preliminary calculations have been made on the following basis: two hydrogen streams each at 35 scfm at 80 atm are to be heated from ambient temperature to 1350°F. It has been estimated that 5/8-in. tubing BWG 12 of seamless 316 SST will yield a Reynolds number of about 10^4 with a tube-side convective heat transfer coefficient of >50 Btu/hr·ft^2·°F. The wall thickness of 0.109 in. will be adequate for a 1300-psi rating at 1300°F. Approximately 50 ft of tubing will be required to heat each hydrogen stream. Two helical coils of 2-ft diam with 1 1/2-in. centers can be accommodated in a 2.5-ft ID by 2.5 ft high externally heated lead bath. Approximately 70 kW of electric power is available to heat the lead batch at 1300°F. Because lead has a vapor pressure of about 2×10^{-4} psi at 1300°F, containment of the lead batch is not difficult. Further details of design will be reported later.

Specifications for the 80-atm hydrogen booster compressor will be let out for bids; analysis of the cold-wall reactor design will continue; and detailed design of the 80-atm preheater will be completed.

Fabrication and Installation

There was no effort in this area to be reported during March. An alternative feeder valve (essentially a plug valve modification of the rotating ball valve concept) and capillary flow restrictors to control inert gas purge of reactor pressure taps are being fabricated. A two-stage refrigeration unit rated at 8500 Btu/hr (at -80°F) will be installed to replace dry ice cooling of the cold trap refrigerant.

Operation

Progress toward routine operation continued during March with the initial introduction of hydrogen into the system. It was further marked by successful feeding and hydrocarbonization of a small amount of coal in a shakedown test. Ground and sized coal for full-scale runs was received from Morgantown Energy Research Center (MERC) late in the month.

Hydrogen from a tube trailer was introduced to the pressurized, heated system in three flow-through shakedown tests. These tests pointed out several minor problems for correction, flowmeter calibrations, etc. In mid-March, a successful shakedown test was completed in which 4 lb of -50 +140 mesh coal were successfully fed at 9.9 lb/hr and hydrocarbonized at 1050°F and 20 atm. Evolved gases were analyzed by the on-line gas chromatograph and liquid product was collected.

In late March, 274 lb of nominally -50 +140 mesh coal, prepared by MERC, was delivered to us for use in our bench-scale experimental program. The subbituminous coal was taken from the Roland and Smith seams at the Wyodak Resources Development Company mine, Gillette, Wyoming, as was the coal used in shakedown tests. Our measurements showed 2% (by weight) larger than 50 mesh and about 15% smaller than 140 mesh; particles greater than 35 mesh were sieved out after the coal arrived at ORNL.

Attempts were made to begin routine, full-scale experiments, but were interrupted by failures of the coal feed system. The first experiment was stopped when the stem of the ball valve feeder broke because the ball locked against a brass seat. Coal had been fed and hydrocarbonized for about 20 min. Later, a second run was interrupted because a 1/4-in. piece of coal lodged in the transport line, but coal was also packing within the body of the valve. Solutions are being implemented to prevent these problems from reoccurring.

Experiments with Wyodak coal will continue. Five experiments are planned: a 10-atm experiment at 1050°F; 20-atm experiments at 950, 1050, and 1150°F; and a 20-atm, 1050°F experiment with a recirculating fluidized bed.

2.3 Residue Carbonization

H. D. Cochran, Jr., and J. B. Gibson

Residue carbonization experiments with H-Coal vacuum tower bottoms were begun last month. Vacuum tower bottoms were used in a successful feeding test and in two carbonization experiments. In the feeding test finely ground residue, with a size range of -45 to +140 mesh, was fed from the feed hopper through the pneumatic transport feed line and into a vacuum flask at a feed rate of 5 lb/hr. No problems were encountered during this test. The first residue carbonization experiment, RC-1, was plagued with feeding problems and was aborted. The second experimental run, RC-2, was aborted when a plug developed in the gas exit from the condenser. Residue had been fed to the system for several minutes at a rate of 12.5 lb/hr when the run was aborted. The reactor temperature was 1200°F during the run, and an inert ceramic material had been used as a bed starter for the recirculating bed.

Inspection of the residue carbonization system after RC-2 revealed plugs in the condenser gas exit, in several pressure taps, in the bottom 2 1/2 in. of the draft tube, and in the feed line between its point of entry into the reactor to the feed nozzle. The feed nozzle was embedded in the plug at the bottom of the draft tube. Numerous agglomerates were found in the reactor. Most of the agglomerates were hollow and were 1/4 to 1/2 in. in the longest dimension. The largest agglomerate found, other than the plugs already mentioned, was 1 3/4 in. in the longest dimension.

We have been advised by ERDA/FE that solvent extraction underflows of CSF and SRC products, to be prepared at the Cresap, West Virginia, pilot plant for our testing, will not be available for several months. This will necessitate some revision of our planned experimental program. A suitable revision will be submitted to ERDA/FE for review during the next month.

An experimental run with H-Coal vacuum tower bottoms is planned for early April. In this run, the feed rate will be about 2 1/2 lb/hr, and the recirculation rate about double the rate of RC-2. If this run proves successful, additional runs will be made under similar flow conditions but with varying temperatures; otherwise, much of the reactor internals and feed injection system will be redesigned to minimize agglomeration. Two changes being considered at this time are removing a bend in the residue feed line and placing the feed nozzle below the draft tube instead of inside the draft tube.

A small-scale batch reactor, about 1 in. OD, is being designed to study the agglomeration tendencies of residue at various temperatures.

3. SUPPORTING RESEARCH AND DEVELOPMENT IN SEPARATIONS TECHNOLOGY

B. R. Rodgers

Summary

Results this month include laboratory-scale settling tests, bench-scale settling tests, and sample ageing tests. Two of ten compounds tested with the laboratory-scale apparatus were effective in increasing settling rates of solids in Solvent Refined Coal (SRC) Unfiltered Oil (UFO). Bench-scale tests concentrated on one of the more promising inorganic additives from the laboratory-scale tests. Twelve runs at various concentrations failed to show any improvement in settling rate over the untreated SRC-UFO. After 6 months ageing, only minor physical and chemical property changes occurred in SRC-UFO samples stored under different conditions.

3.1 Additive Agglomeration Studies: Laboratory-Scale

S. Katz

Tests of additives to improve settling continued using the system described previously.[1] Ten compounds were selected for test because they could be economical to use as additives, and they bore some chemical similarity to compounds which appeared to give favorable results from previous tests. Only two of the test compounds appear to affect settling favorably. These results combined with those obtained previously suggest that certain families of compounds operate through physical and chemical mechanisms to promote settling.

3.2 Additive Agglomeration Studies: Bench-Scale

B. R. Rodgers and D. A. McWhirter

Twelve settling experiments with the bench-scale apparatus were completed this month. An additive that the laboratory-scale tests had shown to be promising was added to SRC-UFO prior to settling at 530°F; standard procedures described previously[2] were followed. There were no significant improvements in the settling rates of the mixture over the untreated SRC-UFO.

3.3 Characterization Studies: Ageing Tests

B. R. Rodgers and D. A. McWhirter

Only small changes were detected in the physical and chemical properties of samples from the SRC process that had been stored under various conditions[3] for a 6-month period. However, there were slight increases in the viscosity, acetone insolubles, and calorific values, and slight decreases in the hydrogen, nitrogen, and sulfur contents, and the densities.

3.4 References for Section 3

1. J. P. Nichols (Program Director), Coal Technology Program Quarterly Progress Report for the Period Ending September 30, 1975, ORNL/TM-5093 (December 1975).

2. J. P. Nichols (Program Director), Coal Technology Program Monthly Progress Report for February 1976, ORNL/TM-5321 (March 1976).

3. J. P. Nichols (Program Director), Coal Technology Program Quarterly Progress Report for the Period Ending December 31, 1975, ORNL/TM-5120 (March 1976).

4. EXPERIMENTAL ENGINEERING SUPPORT OF AN IN SITU GASIFICATION PROCESS

R. C. Forrester, III

Summary

Examination of the pyrophoricity phenomenon observed with chars produced by low-temperature block pyrolyses has continued. These experiments have confirmed early indications that coal obtained from the LERC UCG field test site produces a less reactive char upon pyrolysis than those taken from the Roland-Smith seams near Gillette, Wyoming.

4.1 Large-Block Pyrolysis Studies

R. C. Forrester, III, F. H. Wilson, and G. D. Owen

Table 4.1 briefly describes the most recent pyrolysis experiments which, in every case, produced a pyrophoric char.

Table 4.1. Block pyrolysis: Recent test conditions

Description[a]	Block Diameter (inches)	Heating Rate (C°/min)	Maximum Temperature (°C)
1. Wyodak	6	0.3	500
2. LERC site	3	3	500
3. LERC site	3	0.3	500
4. Wyodak	3	0.3	600
5. Wyodak	3	3	600

[a]Wyodak samples taken from the Roland seam; LERC samples taken from Hanna No. 1 seam.

Preliminary indications suggested that the maximum pyrolysis temperature plays an important role in this phenomenon (lower temperatures producing the pyrophoric materials.) However, chars produced by pyrolysis of the smaller core samples from the LERC test site, while decidedly pyrophoric, were not sufficiently reactive to cause self-ignition upon exposure to air. A temperature rise of 10 to 16 Centigrade degrees was observed, followed by cooling to ambient temperature. Tests with small Wyodak cores were made to determine whether this reduced reactivity was a property of the Hanna coal or a result of surface/volume heat transport

effects. The small Wyodak samples were also highly pyrophoric, leading us to the conclusion that the Hanna coal is simply less reactive.

4.2 ERDA's Underground Coal Gasification Program

R. C. Forrester, III

The Coal Technology Program at ORNL was represented at the ERDA/FE FY 1977 Planning Conference on In Situ Technology (Oil Shale and Coal) which was held in Washington, D.C., on March 25. Welcoming remarks by Dr. P. C. White, Assistant Administrator for Fossil Energy, were followed with brief presentations by participating energy research centers and national laboratories. Field tests of UCG processes under development at LLL, LERC, and MERC were described, as were the associated support programs at ORNL and elsewhere. Afternoon discussions centered upon overall program direction for the coming fiscal year and upon expected funding levels.

5. ANALYTICAL CHEMISTRY

W. D. Shults

Work reported here is carried out within the Analytical Chemistry Division. Sponsoring agencies are ERDA, EPA, and NSF/RANN, the latter agency providing support through the Ecology and Analysis of Trace Contaminants Program for which R. I. Van Hook serves as Principal Investigator.

Summary

The removal and concentration of organic components in by-product waters from fossil fuel conversion processes continues as a principal activity. A Sephadex gel is being used to achieve hydrophilic-lipophilic separations in organic mixtures as a step in the analysis of fossil fuel related materials.

5.1 Analysis of Aqueous By-Products from Coal Conversion Technologies and Oil Shale Retorting

B. R. Clark, C. H. Ho, and M. R. Guerin

In the previous report were listed the major organic compounds of by-product water from simulated in-situ retorted shale oil; these were tentatively identified by co-chromatography as carboxylic acids. Recently, the structures of these compounds were confirmed by GC-MS data obtained by W. T. Rainey and his research group. TMS derivatives of the acids were prepared for this purpose since the simple acids do not yield very distinguishing mass spectra.

For the purpose of surveying the extent of recovery of carboxylic acids from shale by-product water, we have tried different common extraction methods such as organic solvent extraction with and without pH adjustment, activated carbon adsorption and back extraction, and lyophilization. Using GC-analysis (carbowax as stationary phase), the various methods were compared. Acetic acid was not efficiently removed by any of the methods. The series of acids, pentanoic through nonanoic, were nearly 100% removed by ether extractions with the pH adjusted to 7 and then to 1. Propionic and butyric acids were 75 to 85% extracted by ether. Activated carbon desorption followed by chloroform desorption removed acids (except acetic) with a 60 to 99% removal efficiency. Lyophilization was not efficient (<40% at best) because of losses in the vapor phase. Neither was chloroform or benzene extraction efficient at ambient pH.

5.2 Hydrophilic-Lipophilic Separations on Fossil Fuel Related Materials

A. R. Jones

A procedure for the preliminary fractionation of crude tobacco smoke condensate has been developed by workers at the Research Institute of the Cigarette Industry in Germany. Its applicability to shale oil and coal-derived oils is being investigated. The first step is essentially a partitioning of lipophilic and hydrophilic compounds between hexane and an 85/15 vol % mixture of methanol and water which is carried on a column of Sephadex-LH-20, a well characterized, dextron-derived gel.

Crude shale oil was dehydrated by azeotropic distillation with benzene. The benzene solution was then filtered through a 1-cm pad of powdered cellulose and 5 and 2 micron nucleopore membranes. There was little insoluble material. The benzene was removed on a rotary evaporator at 52°/50 mm.

A 17.5-g aliquot of the dehydrated and filtered oil was dissolved in benzene and the 50 ml of solution was added with a syringe to a 5 x 100-cm column containing 500 g of Sephadex-LH-20 which had been swollen with 85% methanol and equilibrated with hexane. The column was washed with 3700 ml of hexane (more than four times the quantity required to elute benz(a)pyrene). In the absence of a suitable detector, the eluate was collected in 100-200 ml fractions and these were evaporated on the rotary evaporator in tared flasks to constant weight.

The lipophilic fraction was 92.5% of the original sample. The hydrophilic fraction was eluted with methanol and with acetone. Total recovery was 100%. A second aliquot of 17.9 g was separated with a recovery of 101%. The lipophilic fraction amounted to 94.7% in the run. Over 90% of the lipophilic fraction was eluted in the 500 ml of eluate immediately following the interparticle volume of solvent. The first step of the procedure was thus shown to be loss-free and reasonably reproducible. The fractions are being further characterized.

5.3 Analytical Services

W. R. Laing and L. J. Brady

A total of 522 samples were submitted for analysis during the past month. The determination of ash content of samples derived from the solids liquid separation tests continued to be the major effort. A rack which permits the flash burning of six samples at one time rather than one at a time has helped to make this step in the analysis more efficient.

Eight samples of SRC liquid were submitted for the following tests: viscosity, specific gravity, acetone insolubles, cresol insoluble, carbon, hydrogen, nitrogen, sulfur, calorific value, forms of sulfur, and simulated distillation tests.

Ten samples of coal were tested for calorific value, sulfur, carbon, and hydrogen.

6. ENGINEERING EVALUATIONS OF THE SYNTHOIL AND HYDROCARBONIZATION PROCESSES

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

Summary

Synthoil. Process flow diagrams and heat and material balances were completed for all process units, and overall utilities requirements were calculated. A preliminary version of a computer code for cost estimation by the factor method is now operational.

Hydrocarbonization. The overall utilities requirements for the process were estimated, and balances for the steam, fuel gas, water and electric systems were reached. Major emphasis continued on equipment selection, sizing, and cost estimation. A preliminary cost estimate of $24.59 million for the tar hydrotreater section was made, and agrees very well with the Hydrocarbon Research, Inc. estimate of $24.35 million. The Fluidized Bed Combustion Company completed their first-month design effort and prepared a solids and gas flow schematic diagram, a preliminary plant layout, and an equipment arrangement drawing for one fluidized bed combustion module.

6.1 Synthoil Process

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

Process flow diagrams and heat and material balances were completed for all process units with the exception of the ammonia plant and the oxygen plant. These are proprietary units for which simplified flow diagrams, material balances, and utilities requirements will be presented.

The overall utilities requirements were calculated. A utilities system balance was reached for steam, water, electricity, and fuel gas. Utilities systems flow diagrams were completed for the water and steam systems.

Work continued on the preliminary cost estimate. A preliminary version of a computer code for cost estimation by the factor method is now operational.

The various sections of the flowsheet progressed as follows:

(1) Coal Handling and Preparation: The coal preparation flowsheet was revised to provide coal feed for an electric plant which will provide power for the Synthoil plant.

(2) Gasification and Low-Temperature Carbonization: The flowsheets, material balances, and equipment lists for the gasification unit (Unit 18) and the low-temperature carbonization unit (Unit 26) were finalized, and utility requirements for both units were tabulated. Work continued on developing equipment costs for these units.

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 100,000 bbl/day of fuel oil continued. Major emphasis was placed upon equipment selection, sizing, and cost estimation. The overall utilities requirements for the process were estimated and a utilities summary is being prepared. A utilities system balance for the steam, fuel gas, water, and electricity was reached. The various units of the plant progressed as follows:

(1) Hydrocarbonization: Cost estimation of the hydrocarbonization vessels continued, and cost factors for installation, piping, instrumentation, insulation, and contractor's overhead are being developed. Consideration is being given to the use of the treated water from the sour water unit as part of the makeup slurry water for the char slurry handling system. Information on equipment needed for the char-handling system was obtained from vendors. Data on the optimum particle size (0.094 in. top size) for slurry pipeline transport was provided by Bechtel Corporation, designers of the Black Mesa coal slurry pipeline. The Willis Company supplied information on a choke they manufacture for lowering the pressure of the char slurry from 125 psia to atmospheric pressure for sizing and storage purposes. The limestone handling system was revised to reflect a higher rate of flow and a larger storage capacity.

(2) Cryogenic Separation Unit: The cryogenic separation system material balance was revised to reflect the effects of methanation on the ethane and heavier hydrocarbons in the feed stream to cryogenic separation. The methanator in the make-gas treating unit will convert almost all of these heavier hydrocarbons to methane, which means that a separate stripper to separate these heavier hydrocarbons in the cryogenic system will not be needed. The additional methane produced by the methanator will be substituted for the heavy hydrocarbon feed stream to the reformer, so the overall plant material balance is unaltered.

(3) Tar Hydrotreater: The process calculations for the hydrotreating section were completed, and all equipment sized. A preliminary cost estimate of $24.59 million was made for this section and agrees very well with the $24.35 million estimate made by Hydrocarbonization Research, Inc.

(4) Fluidized Bed Combustion Unit: Fluidized Bed Combustion Company (FBCC) completed their first-month design effort on the FBC unit for the hydrocarbonization plant. FBCC has prepared a solids and gas flow schematic, a preliminary plant layout, and an equipment arrangement drawing for one module.

The six FBC modules and associated equipment will cover an area of 300 ft by 420 ft. The module height is 140 ft with the steam drum and outlets at a height of approximately 165 ft. The hydrocarbonization plant layout is to be modified to improve the FBC interconnection with the remainder of the plant.

(5) Water Treatment: The flowsheet and material balance for the hydrocarbonization facility water supply and treatment plant were completed. The daily water requirements for the overall facility amount to 69.1×10^6 gal. The major portion of the daily needs (43.6×10^6 gal) will be used for transporting the low sulfur char product and will be given only a minimal sedimentation treatment to remove suspended solids; whereas the process water (25.5×10^6 gal) will be clarified and subsequently treated as boiler feedwater or as cooling tower makeup water.

The equipment sizing and equipment lists were prepared and the equipment cost data procured.

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. 1AA-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.

Summary

Orders were placed for tubing to be used in the matrix of the fluidized-bed furnace and preparations for procurement of the furnace continued. Recommended gas turbine modifications for operation under the MIUS unit design conditions were received from AiResearch. Tests continued on the mixing rate of coal in a fluidized bed and on the coal feed system.

Furnace Procurement

The order has been placed for the Incoloy 800 tubing and a smaller quantity of Inconel 600 tubing for the tube matrix in the fluidized bed. Negotiations are continuing on procurement of a small number of stainless steel tubes that are also to be included in one of the tube bundles.

Two additional vendors were visited to determine their interest and capability for fabrication of the fluidized-bed furnace. Drawings and specifications to be included in the furnace bid package were completed and are undergoing final review.

Turbine-Generator Unit

The AiResearch analysis of ways to adapt the Model 831-200 gas turbine to the MIUS application was completed and their final report was published. The conclusions reached in the study were that the Model 831-200 engine could be modified for closed cycle operation and that a speed of about 80% of the original design speed would be optimum for the closed cycle system. The recommended modifications include replacement of the shaft seals and bearings and the thrust bearing as well as other alterations. Specifications for engine modification in accordance with AiResearch recommendations for closed cycle operation were prepared and arrangements for completion of the work was initiated.

Cold Flow Tests of a Fluidized Bed

A third test on the mixing rate of coal in a fluidized limestone bed in the 4 ft square cold flow model was completed. The fluidizing velocity for this test was about 2.2 ft/sec, which is equivalent to about 50% of full power air flow for the hot furnace. The mixing rate at this air velocity was considerably higher than that previously found at a velocity just above the minimum operating flow rate.

Following the third test, modifications to the outlet air duct and cleanup system were initiated to reduce back pressure in the model in order to operate up to the maximum test velocity of 4 ft/sec.

Coal Metering and Feed System

Testing of the flow splitter type of system for dividing the coal feed stream into 4 equal parts continued with emphasis on endurance running at full design power coal feed rate. The system has been operated for a total of 35 hr and no operating problems have been encountered.

Supplemental Studies

Work was initiated on six supplemental tasks in support of the conceptual design phase of the coal-fueled MIUS. The tasks include analytical studies and experimental testing as summarized below:

1. materials testing of air-cooled tubes in an atmospheric fluidized-bed combustor,
2. economic analysis of the coal-fueled MIUS concept,
3. analysis of reliability, operating and maintenance, and back-up power requirements,
4. heat transfer tests using air-cooled tubes in an atmospheric fluidized-bed combustor.
5. economic trade off studies of applicable coal-fueled MIUS components and against other types of power cycles, and
6. analysis of the solid materials handling aspects of the coal-fueled MIUS concept.

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 on the Pressure Vessel and Piping Technology Program included a visit to Coalcon, where arrangements to discuss Synthoil at PERC and a visit to the Synthane Plant were planned. Preparations of several sections of the report continued.

In our development of inspection techniques for wear and process-resistant coatings, the x-ray fluorescence data obtained on specimen ANL-C5 were correlated to variations in the composition of the coating. Additional coated samples were prepared at ORNL consisting of NiCr and ZrO_2. These specimens, along with the ANL sample, were tested using eddy-current techniques.

On our Iron and Nickel Carbonyl Formation and Prevention Program, a wet chemical method for quantitative analysis of iron carbonyl in synthesis gas was tested and perfected resulting in a detection limit of 3 ppb. Using atomic absorption spectroscopy, we can detect 1 ppb.

In the Failure Analysis Program, an evaluation of a pressure let-down valve has revealed extensive cracking in the braze between the valve plug and stem. An evaluation of the 316 stainless steel heater coil from Project Lignite was begun.

With regard to Fireside Corrosion in a Fluidized Bed Combustion, representatives from FE/ERDA and ORNL visited Pope, Evans, and Robbins to discuss the Rivesville, West Virginia Plant. We are arranging to evaluate six different types of superheater materials to be removed from the plant this summer.

8.1 Pressure Vessel and Piping Technology Assessment

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

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.

A visit was made to the Coalcon offices and discussions were held relative to their design needs. Coalcon is in the process of designing a 3000 ton per day demonstration plant. They intend to design based on a 90% duty cycle (330 days per year). We identified a number of problem areas where materials can affect operational efficiencies. In particular, Coalcon is interested in the reliability of large pressure containing components.

Arrangements have been made to visit additional pilot plant sites, industrial firms, and architectural engineers. We have begun writing various sections of the report and these will be forwarded to ERDA/FE for their review.

8.2 Inspection Techniques for Wear- and Process-Resistant Coatings

R. W. McClung and G. W. Scott

A survey and evaluation of nondestructive testing (NDT) techniques likely to show feasibility for manufacturing inspection was initiated; preliminary results are listed.

Additional data on specimen ANL-C5 was obtained; its composition now appears consistent with x-ray fluorescence data.

We demonstrated the ability of fluorescence radiography to detect variations in the coating which appear to be related to composition. Further examination by optical methods is anticipated.

Examination of specimen ANL-C5 with commercial ultrasonic pulse-echo thickness gage indicates that the coating-substrate interface is not sufficiently detectable to trigger the instrument for a thickness measurement. However, the coated surface produces a phase shift in the reflected pulse which may be usable to detect the presence or absence of a coating on the surface.

The ORNL Welding and Brazing Laboratory made three specimens: a sandblasted Incoloy-800 substrate and one specimen each coated with NiCr and ZrO_2. These specimens and ANL-C5 were tested with eddy-current instruments. The substrate and the metallic coatings exhibit ferromagnetic responses, which means that special designs and analytical schemes will be required for eddy-current inspections.

Review and Evaluation

We have initiated a comprehensive review of NDT techniques to select those which appear to be most appropriate for manufacturing inspection of coatings. From a compiled list of over 70 methods, we selected 30 which apply to the four basic coating inspection problems: defects in the coating, coating thickness, coating material uniformity, and unbonds between the

coating and substrate. The selected techniques were then individually rated against four criteria: (1) availability of suitable equipment (at or accessible to ORNL); (2) availability of personnel with appropriate experience or expertise in the testing methodology; (3) duration and difficulty of anticipated experiments required to demonstrate feasibility; and (4) an estimate of the likelihood of success. Of course, the factors on availability of equipment and individual experience can be overcome if the other factors look sufficiently promising. Subsequent analyses will take these into consideration.

The emphasis in this initial analysis was on feasibility alone, with the deliberate exclusion of concerns about the ease of adaptation to the industrial situation, production-line reliability, etc. These questions and others will be addressed in subsequent reviews. The top three methods for each requirement in this preliminary analysis are listed.

a. Defect detection: fluorescence radiography, liquid penetrant eddy current.

b. Coating thickness: eddy current, x-ray fluorescence, dielectric (capacitance) gaging.

c. Material uniformity: fluorescence radiography, eddy current, x-ray fluorescence.

d. Unbonds: pulse-echo ultrasonics, infrared radiometry, resonance ultrasonics.

Evaluations of some methods, e.g., backscatter radiometry, are incomplete, pending completion of equipment searches.

Penetrating Radiation Testing

ANL provided additional information that corrected original data on the coating composition of specimen C5. It contains TiC plus a commercial mix of Co, Ni, Cr, and W. These last four plus Mn from the substrate were found in the x-ray fluorescence data.

Specimen ANL-C5 was also tested with a fluorescence radiographic technique. A type-R film was compressed against the coated side; an aluminum filter was placed above the film, and a copper filter was placed above the aluminum. Exposure was made by 250-kV x-rays, from a tungsten-target tube, incident from the copper side.

The image, produced by fluorescence from the specimen, contained dark areas corresponding to higher energy or intensity fluorescence and several light "dots," indicating very low-energy fluorescence or missing material. The image data will be correlated with optical microscopic examination.

Ultrasonic Testing

Specimen ANL-C5 was tested with a Panametrics 5221 (pulse-echo) thickness gage. The interface between the coating and substrate was not sufficiently distinguishable to allow the unit to trigger and measure either the coating or substrate thickness independently. However, observation and comparison of pulses reflected from the coated surface and from the back surface of an uncoated blank indicate that the pulse suffers a phase shift when reflected from the coated surface which does not occur when it is reflected from an uncoated surface. Detection of this phase shift might allow detection of missing coating, some types of defects, and unbonds by interrogation from the substrate side; however, these conditions may provide similar response and would require additional inspection to overcome ambiguity.

Eddy-Current Testing

The ORNL Welding and Brazing Laboratory has completed three specimens:

(a) An uncoated Incoloy-800 substrate, 1/8-in. thick, sandblasted on both sides;

(b) OR-JC12, 0.007-in. NiCr on Incoloy 800; and

(c) OR-JC61, 0.0025-in. ZrO_2 on Incoloy 800.

These specimens and ANL-C5 were tested with a Nortec NDT-6 instrument and a locally fabricated Brooks-coil instrument. The Nortec unit displays on a cathode-ray tube the relative impedance of the test coil as affected by the specimen. When appropriately calibrated, the instrument response can indicate the electrical conductivity and changes due to variations in ferromagnetic properties and coil-to-conductor spacing (lift-off). Results of the tests with this unit were as follows:

(a) Incoloy-800 substrate - weakly ferromagnetic, with a resistivity about 100 μΩ-cm;

(b) ANL-C5 - significant ferromagnetic response; resistivity could not be estimated from the measurements made;

(c) OR-JC12 - weakly ferromagnetic; resistivity about 125 μΩ-cm; and

(d) OR-JC61 - combined response of the coating and substrate appears as a net lift-off effect; resistivity about 115 μΩ-cm.

The resistivities measured for the coated specimens are "effective" or composite resistivities which combine the effect of resistivities of the coatings and the substrate material.

The presence of the ferromagnetic response indicates potential requirements for special probe designs and test/analysis schemes to compensate for changes in permeability and conductivity.

8.3 Iron and Nickel Carbonyl Formation and Prevention

J. Brynestad and J. H. DeVan

The literature survey has been completed. A report is being prepared.

A wet chemical method for the quantitative analysis of iron carbonyl in synthesis gas has been tested and perfected. The detection limit for a 50 liter gas sample by this method is about 3 ppb. A similar method for nickel carbonyl determination is being developed.

We have demonstrated that atomic absorption spectroscopy is very well-suited for the determination of iron (and nickel) carbonyl in combustible gases, with a detection limit of less than 1 ppb of iron carbonyl. The detection limit for nickel is not as yet established.

An autoclave flow-through system has been designed. It is being constructed for kinetic studies of carbonyl formation with iron-based materials.

J. H. DeVan and J. Brynestad travelled to PERC to discuss details of the experimental approach for the project. We obtained data on flow rates, surface areas, and geometries of the Synthane methanator, which will be used to set our test conditions.

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.

We are continuing the examination of the pressure let-down valve trim set from the Synthoil PDU at PERC. Metallographic examination has shown extensive cracking in the braze between the valve plug and stem. This could have allowed misalignment of the plug with respect to the valve seat and caused unusual turbulent flows of the product oil through the valve. This turbulence could account for the observed preferential erosion of the seat and plug material (Kennametal grade 701). Also, we have received specifications and drawings of the let-down valve (a Masoneilan Wee Willie Control Valve) and are in the process of fabricating a plexiglass model of it. This model will help us study flow patterns and give more insight into the cause of the observed erosion. There is no evidence that the valve seat insert was brazed to the valve seat. It appears to have been a mechanical joint; perhaps a pressed-fit.

We have begun a metallurgical analysis of a failed section from the heater coil from Project Lignite. There is evidence that cyclic loading may have been a contributing factor in the Cl^- stress corrosion crack(s) that appear to be responsible for the failure.

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 studied as vehicles for assessment and guidance.

Beginning in February, the collection of information on pressure vessel sizes and requirements was initiated, and pursuit of this activity is being continued. Gasifier pressure vessels for commercial-size HYGAS and Synthane coal conversion of plants were jointly selected with Fossil Energy - ERDA for study.

Visits were made to four major architectural engineering (AE) firms for discussions on coal conversion systems, with special emphasis on pressure vessel and piping considerations. The firms visited were Fluor Engineers and Constructors, Inc. (February 26), Bechtel Corporation (February 27), Ralph M. Parsons Company (March 1), and C. F. Braun Company (March 2, 1976). The range of topics addressed embraced process information, including flow diagrams, and experience with coal conversion or related systems; vessel configurations and dimensions; design and operating conditions; corrosion, erosion and embrittlement; piping design, with emphasis on refractory lined piping; codes and standards; analysis methods; procurement considerations, including fabrication; and initial and in-service inspection. A trip report was written and copies forwarded to members of the Materials and Power Generation Division of Fossil Energy - ERDA.

At C. F. Braun Company, conceptual design information and preliminary sketches for gasifier vessels were obtained for commercial-size HYGAS and Synthane processes. The information and sketches were combined with information from other cources to establish bases from which to proceed with the development of conceptual designs of concrete pressure vessels. Concurrence was obtained from Fossil Energy - ERDA to consider two-train gasification plants, having outputs of 500 billion btu per day for the HYGAS process and 250 billion btu per day for the Synthane process. The vessels in the first case will have a maximum inside diameter of about 32 ft and a height of about 240 ft; for the second, the inside diameter will again be about 32 ft and the height will be about 110 ft.

The vessel for the Synthane process is being addressed first. Initial penetration size and location and liner configuration studies are being carried out. Liner concept and closure details also are receiving early attention. Internal structures are being examined and refractory-liner interactions studied to determine loadings on the liner and vessel.

8.6 Other Related Work

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

Fluidized Bed Material Support Activities

Fireside Corrosion. — On March 16 and 17, J. H. DeVan and R. H. Cooper visited Pope, Evans, and Robbins (PER) in New York City to discuss their design of the demonstration size fluidized bed coal combustion boiler currently being built in Rivesville, West Virginia. These discussions indicated that the design of the Rivesville facility is based on experience obtained from a pilot plant located in Alexandria, Virginia. With regard to this pilot plant, PER indicated that the American Nickel Company has installed a rack of surveillance coupons in the facility. These coupons will be removed for evaluation by the American Nickel Company at the end of June. In addition, PER currently has six different types of superheater tube materials in the pilot plant. PER indicated that we were welcome to evaluate this material when it was removed from the pilot plant in June.

On March 11, a meeting was held at ORNL with representatives of the Fluidyne Corporation. The purpose of this meeting was to review the conceptional design of their fluidized bed and to define the geometry and test conditions for fireside corrosion samples to be placed in this combustor. Based on the results of this meeting, Fluidyne is currently finalizing the design of fluidized bed combustor.

9. ENGINEERING EVALUATIONS OF NUCLEAR PROCESS HEAT FOR COAL CONVERSION

W. R. Gambill

Minor revisions were made and sections retyped in the report to Fossil Energy titled "A Critical Evaluation of the Application of Gas-Cooled Reactors to Coal Conversion," which should be issued in April as ORNL-TM-5341. Final versions of two reports prepared by United Engineers and Constructors, one of which is a part of the preceding report, were received during March.

The engineering feasibility review of General Atomic's proposal to ERDA for a bench-scale test program on thermochemical water splitting for hydrogen production was completed.

An expanded trip report describing our inspection visit to the Black Mesa coal slurry pipeline was completed and issued, and a general discussion of coal-water slurry pipelines was held with T. S. Jane, a visiting economist from the Energy Policy Committee of Taiwan. Two telephone discussions were held with L. J. Keller of Dallas regarding his "Methacoal" concept (pipelining a suspension of fine coal in methyl fuel); and B. Blaustein's draft comparative analysis of coal-water slurry vs "Methacoal," received in late March, was reviewed.

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