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Coal Technology Program Progress Report for April 1976

OAK RIDGE NATIONAL LABORATORY

OPERATED BY UNION CARBIDE CORPORATION FOR THE ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION

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COAL TECHNOLOGY PROGRAM
PROGRESS REPORT FOR APRIL 1976

JUNE 1976

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

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ORNL/TM-5045, Progress Report for September 1974
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COAL TECHNOLOGY PROGRAM PROGRESS REPORT FOR APRIL 1976

ABSTRACT

This report — the twenty-first 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, engineering evaluations of nuclear process heat for coal conversion, coal-fueled MIUS, materials, and engineering evaluations of the Synthoil and Hydrocarbonization processes.

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1. SUMMARY

J. P. Nichols

Highlights of our progress in April are summarized below:

° In the Hydrocarbonization Research program, two successful experiments were completed in the bench-scale hydrocarbonizer. The first, HC-1, involved feeding 1.3 lb/hr of Wyodak coal to the reactor at 1075°F and 300 psig H_2 pressure for a period of 9.6 hr. The second, HC-2, involved feeding 8.3 lb/hr of Wyodak coal to the reactor at 1030°F and 300 psig H_2 pressure for a period of 13.2 hr. The system will be operated in a routine manner to complete a campaign of experiments with Wyodak coal.

° The Supporting Research and Development in Separations Technology project initiated a settling test at a lower temperature (390°F) using 20% toluene in Solvent Refined Coal (SRC) Unfiltered Oil (UFO) which produced a 30% clarified product in 2 hr.

Characterization tests include distillation curves for Wilsonville's SRC-UFO and a particle size distribution of Pittsburg and Midway Coal Mining Company's (PAMCO) SRC-UFO.

° In Experimental Engineering Support of an In-Situ Gasification Process studies of intermediate-temperature pyrolysis of large blocks have been maintained this month with char samples continuing to demonstrate pyrophoricity, even after heating to 700°C. Simulated distillation analysis of tars produced by the last eight experiments are being compared with those performed at Laramie upon tars produced by the Hanna No. 2 experiment.

° In Coal-Fueled MIUS, stainless steel tubing to be used in one of the furnace tube bundles was ordered and the bid package for the furnace was completed. Tests continued on the coal feed system and with the cold flow fluidized bed model.

° In the Materials program, further work with x-ray fluorescence, using a gamma source and cryogenic detector, indicates adequate sensitivity to measure 0.001-in. thickness changes in ZrO_2 coatings.

° The following progress was made in Engineering Evaluations of the Synthoil and Hydrocarbonization Processes:

In Synthoil, flow diagrams, material balances, and utilities requirements were completed for the entire facility. A computer program was written to calculate the product price required to yield a specified rate of return or investment when the capital investment and operating costs are known. Work continued on the preliminary cost estimate. The overall cost estimating program using the factor method was tested on several units and checked against other estimates; the equipment cost factors are being refined to make results check more closely.

In Hydrocarbonization, flowsheets were reviewed for compatibility, equipment lists were brought up to date, and utilities requirements were compiled from the individual flowsheets. The char recovery and storage subsystem flowsheet was completed. The design status of the fluid-bed combustor was reviewed with representatives of the Foster Wheeler Corporation. Completed flowsheets and preliminary detail design drawings were presented, and sizes of major components and the selection of critical design temperatures were described and discussed.

2. HYDROCARBONIZATION RESEARCH

H. D. Cochran, Jr.

Summary

Two successful experiments were completed in the bench-scale hydrocarbonizer. The first, HC-1, involved feeding 1.3 lb/hr of Wyodak coal to the reactor at 1075°F and 300 psig H_2 pressure for a period of 9.6 hr. The second, HC-2, involved feeding 8.3 lb/hr of Wyodak coal to the reactor at 1030°F and 300 psig H_2 pressure for a period of 13.2 hr. The system will be operated in a routine manner to complete a campaign of experiments with Wyodak coal.

2.1 Bench-Scale System

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

Work Accomplished

Design and Review. Design of modifications for 80 atm operation of the bench-scale hydrocarbonizer has continued, but no significant new results are available.

Fabrication and Installation. An available refrigeration unit was installed to cool cold trap heat transfer fluid. A modified rotary feeder was used in the two successful experiments consisting of a rotating Teflon plug within a steel body. A small number of holes were machined on the circumference of the cylindrical plug, so that coal is fed in a controlled fashion as the plug rotates.

Operation. Routine experimentation began in April with two hydrocarbonization experiments as follows:

HC-1 - Uniformly fluidized bed reactor at 1075°F and 300 psig H_2 pressure, Wyodak subbituminous coal fed at 1.30 lb/hr for 9.6 hr.

HC-2 - Uniformly fluidized bed reactor at 1030°F and 300 psig H_2 pressure, Wyodak subbituminous coal fed at 8.33 lb/hr for 13.2 hr.

Analysis of coal and products was initiated.

In each experiment, the rates of coal feed was observed by the coal flow monitor and by evolution of carbon-containing gases as indicated by the on-line gas chromatograph. Char and liquids were collected, sampled, and distributed for analysis so that carbon balances could be determined. Solid size and density were also measured (Table 2-1).

Table 2-1. Coal and char analyses from Run HC-1

	Coal	Char
Moisture:	9.95%	0.0%
Proximate analysis, moisture-free		
Ash:	7.00%	14.78%
Volatile matter:	43.6%	8.24%
Fixed carbon:	49.4%	76.0%
Ultimate analysis, moisture-and-ash-free		
Carbon:	71.3%	93.34%
Hydrogen:	5.59%	3.51%
Nitrogen:	1.02%	0.84%
Sulfur:	0.45%	0.60%
Oxygen:	21.7%	1.70%
Calorific content, Btu/lb maf	11,960	15,363
Bulk density (g/cm^3)	0.61	0.58
Particle density (g/cm^3, using isopropyl alcohol)	1.1	1.19
Particle size: mesh, mean diameter, weight percent		
-20+30 (707 μ)	0.01%	0.03%
-30+50 (500 μ)	0.06%	0.27%
-40+50 (353 μ)	0.19%	0.18%
-50+60 (273 μ)	10.61%	0.22%
-60+80 (210 μ)	30.84%	6.87%
-80+100(162.4 μ)	33.69%	54.52%
-100+200 (105.0 μ)	10.15%	12.99%
-200+325 (57.1 μ)	13.95%	23.68%
-325 (~22 μ)	0.50%	1.26%
Mean particle size, weight basis	132 μ	104 μ

Work Forecast

Design and Review. Effort will continue on modifications for 80 atm operation of the bench-scale system.

Fabrication and Installation. No effort is planned in this area.

Operation. Routine operation of the bench-scale reactor will continue. A series of experiments at 300 psig H_2 pressure is planned to determine the effect of bed temperature on hydrocarbonization yields. Later experiments will assess the effect of hydrogen pressure and solids residence time.

2.2 Residue Carbonization

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

Work Accomplished

A third residue carbonization run, RC-3, was performed in early April. Residue was fed to the system at a rate of 2.5 lb/hr and internal solids recirculation rates were greater than in Run RC-2. The reactor temperature was 1200°F during the run, and an inert ceramic material was used as a bed starter for the recirculating bed. The run was aborted after 0.75 lb of residue was fed because the feed line plugged.

Inspection of the residue carbonizer after Run RC-3 revealed that the entire cross section of the reactor bottom was plugged. The lower 6 in. of the draft tube, including the region where the feed line entered the draft tube, was plugged and had to be discarded.

As a result of these findings, the reactor internals were redesigned. In the new design, the bend in the feed line within the reactor was eliminated. The feed will now enter the reactor from directly below the draft tube and the feed tube will not enter the draft tube. (The region where the feed tube entered the draft tube appeared to be a site of initial agglomeration.) There are no pressure taps on the draft tube and it can be easily replaced.

A small scale batch reactor, 1 in. OD, has been constructed and will be used to study the agglomeration tendencies of residues at various temperatures.

Work Forecast

An experimental run is planned for early May with reaction conditions the same as those for RC-3 in order to test the new geometry of the residue carbonizer. Parameters for subsequent runs this month will be determined by the results of this run and results of the agglomeration tests.

The small scale reactor will be used to outline temperatures at which agglomeration occurs with the H-Coal vacuum tower bottoms.

3. SUPPORTING RESEARCH AND DEVELOPMENT IN SEPARATIONS TECHNOLOGY

B. R. Rodgers

Summary

An initial settling test at a lower temperature (390°F) using 20% toluene in Solvent Refined Coal (SRC) Unfiltered Oil (UFO) produced a 30% clarified product in 2 hr. Following this run, the settler was dismantled for alterations intended to give a more uniform heat distribution. This should eliminate the previous problems with convection currents.

Characterization tests include distillation curves for Wilsonville's SRC-UFO and a particle size distribution of Pittsburg and Midway Coal Mining Company's (PAMCO) SRC-UFO.

3.1 Bench-Scale Tests

B. R. Rodgers and D. A. McWhirter

A settling test was made of a Wilsonville SRC-UFO mixture containing 20% toluene, a concentration which gave good settling in the laboratory-scale apparatus. The bench-scale settler produced 30% clarified product in 2 hr at 390°F, while the laboratory-scale settler produced 30% clarified product in 1 hr under these conditions. These values are consistent when the ratio of bench-scale to laboratory-scale settling height (2/1) is considered.

Following the above tests, the settler was disassembled for alterations intended to correct uneven heating which might have retarded settling due to convection currents during previous runs. Some carbonaceous material was firmly attached to the wall (~1/16 in. to 1/8 in. layer), and was removed with a wire brush. Three additional external and three internal thermocouples were installed prior to spraying the external surface with a 0.20-in. conductive layer of copper. The calrods will be completely embedded in a heat transfer paste (Thermon) prior to reinstallation. The unit is expected to be in operable condition within 3 weeks. During this period a few settling runs will be made in the autoclave (with no stirring) followed by some initial precoat filtration runs.

3.2 Characterization Tests

B. R. Rodgers and D. A. McWhirter

Distillation of SRC-UFO and Filtered Oil (FO) from Wilsonville revealed a high degree stability during storage; four distillations for FO after 6 months under different storage conditions[1] are indistinguishable. PAMCO SRC-UFO and -FO is a less volatile material than the Wilsonville SRC.

Electron microscope particle size distributions of PAMCO SRC-UFO showed that these particles are only slightly larger than those found in Wilsonville SRC-UFO from Illinois No. 6 coal, and slightly smaller than those found in Wilsonville SRC-UFO from Pittsburgh No. 8 coal. Fifty percent of the particles had diameters less than 1 μ.

3.3 References for Section 3

1. Coal Technology Program Quarterly Progress Report for Period Ending March 31, 1976, ORNL-5159 (in publication).

4. EXPERIMENTAL ENGINEERING SUPPORT OF AN IN SITU GASIFICATION PROCESS

R. C. Forrester III

Summary

Intermediate-temperature pyrolysis studies of large blocks have been maintained this month with char samples continuing to demonstrate pyrophoricity, even after heating to 700°C. Simulated distillation analysis of tars produced by the last 8 experiments are being compared with those performed at Laramie upon tars produced by the Hanna #2 experiment.

4.1 Large-Block Pyrolysis Studies

R. C. Forrester III, T. M. Somers, and G. D. Owen

Six-inch right circular cylinders of Wyodak subbituminous coal have been pyrolyzed at 700°C using heating rates of 3°C/minute and 0.3°C/minute. At the conclusion of both experiments, exposure of the chars to air resulted in self-heating to temperatures as high as 300°C, though ignition did not occur as with some of the chars produced at lower temperatures.

The computer code which has been developed for data analysis is being altered slightly to ensure computational conformance to recent equipment changes in the block pyrolyzer apparatus.

4.2 ERDA's UCG Program

R. C. Forrester III

Dr. Albert P. Sikri, who recently joined the Underground Coal Gasification Branch of ERDA's Division of Oil, Gas, and Shale Technology visited ORNL on April 15 for a review of the Coal Technology Program. Though he was primarily concerned with in situ support studies, he took detailed interest in our hydrocarbonization (bench-scale) unit, the solids/liquid separations activities, and other aspects of the Oak Ridge program.

We were also visited by Dr. John O. H. Newman of the National Coal Board, England, who has spent the last year with PERC's Synthoil research group. Dr. Newman was not familiar with the in situ program, being involved principally in the area of liquefaction technology, but he was interested in char pyrophoricity and related details of British experience with partially-devolatilized, ground coal which had been pressed into briquettes. These briquettes were highly pyrophoric and self-ignited in transport and in storage. He also suggested that we check the German literature for some references to pyrophoric chars.

5. ENGINEERING EVALUATIONS OF NUCLEAR PROCESS HEAT FOR COAL CONVERSION

W. R. Gambill

Further minor revisions were made in the report to Fossil Energy titled "A Critical Evaluation of the Application of Gas-Cooled Reactors to Coal Conversion." Following further internal review, it will be issued as ORNL/TM-5341.

The writer participated in a Nuclear Process Heat Discussion Meeting held at the ERDA Training Center in Oak Ridge on April 23, and reviewed in detail an overview paper by A. R. Jones, I. Spiewak, and W. R. Gambill titled "Assessment of Very High Temperature Nuclear Reactors in Process Applications," to be presented at the Eleventh Intersociety Energy Conversion Engineering Conference, September 12-17, 1976.

6. 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.

Summary

Stainless steel tubing to be used in one of the furnace tube bundles was ordered and the bid package for the furnace was completed. Tests continued on the coal feed system and with the cold flow fluidized bed model.

Furnace Procurement

The order was placed for the stainless steel tubes that are to be included in one of the tube bundles. All of the tubing for the furnace is now on order.

Preparation of final drawings and specifications for procurement of the fluidized bed furnace assembly was completed. The bid package is expected to be sent to vendors in May 1976.

Turbine-Generator Units

Preparation of contractural arrangements with AiResearch continued for modification of the Model 831-200 engine for closed cycle operation.

Cold Flow Tests of a Fluidized Bed

The air flow tests in the 4-ft square cold flow model were resumed to obtain pressure drop data. Eleven test runs were completed over a range of fluidizing velocity from 0.5 to 3 ft/sec. Additional tests are planned for May in which some of these runs will be repeated to obtain tube vibration data and the velocity range will be extended up to about 4 ft/sec.

Coal Metering and Feed System

Endurance running of the flow splitter feed system continued at full design power coal feed rate. The system has been operated for a total of 85 hr and no operating problems have been encountered.

A prototype furnace coal feed nozzle was fabricated. Performance tests and short-time endurance tests of the nozzle are planned to begin next month.

Supplemental Studies

The work scope of the proposed study on solid materials handling problem has been revised and is under review.

Negotiations to arrange a subcontract with FluiDyne Engineering Corporation to conduct corrosion tests on air-cooled tubes in their fluidized bed furnace have continued. A proposed schedule of materials and specimen tube arrangement was prepared for planning and cost estimating.

A preliminary proposal for installation and operation of a heat transfer test of an air-cooled tube in the FluiDyne furnace was prepared and reviewed with FluiDyne. Detailed cost and design information are in preparation.

Work continued on the analysis of reliability, system performance with alternate cycles, and economic comparison of the closed cycle gas turbine with two alternative energy systems.

Reports

A draft report on the heat transfer test run in the small cold flow model was completed and submitted for review. Final reports of Phase I, Conceptual Preliminary Evaluation, were published as listed below:

A. P. Fraas et al., "Use of Coal and Coal-Derived Fuels in Total Energy Systems for MIUS Applications," Vol. I, Summary Report, ORNL/HUD/MIUS-27 (April 1976).

A. P. Fraas et al., "Use of Coal and Coal-Derived Fuels in Total Energy Systems for MIUS Applications," Vol. II, ORNL/HUD/MIUS-28 (April 1976).

7. 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

Preliminary beta-ray backscatter tests to measure coating thickness were unsuccessful. Further work with x-ray fluorescence, using a gamma source and cryogenic detector, indicates adequate sensitivity to measure .001-in. thickness changes in ZrO_2 coatings. Initial attempts at thermal testing did not provide confirmation of feasibility but did provide an improved estimate of the type of technique required.

A wet chemical method for the quantitative analysis of nickel carbonyl has been tested and optimized. The detection limit for a 50 liter gas sample by this method has been improved to 1 ppb. The method allows simultaneous analysis of iron and nickel carbonyl.

Our prestressed concrete pressure vessel studies are underway. The initial phases of conceptual design for HYGAS and Synthane gasifiers was continued. We visited the Synthane Plant on April 14 and obtained much information germane to our study.

7.1 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.

Visits were made to PERC to discuss both the Synthoil and Synthane coal conversion processes. A representative of Coalcon visited ORNL and discussed pressure vessel and piping material needs for their 3000 ton per day demonstration plant. Taylor-Forge, Cicero, Illinois, was visited and discussions were held relative to the availability of large elbows and fittings for commercial-sized conversion plants. Taylor-Forge does not, at this time, have the capabilities of producing large (five to seven ft diam), thick-walled (three-four in.) elbows. They estimated a two-year, approximately $2,000,000 effort would be required to advance the state-of-the-art.

Contact was made with Cameron Iron Works and Gray Tool Company in Houston, Texas. Cameron supplies large piping; their maximum size is about 48 in. diam and 3-4 in. wall thickness. They are limited to a 40,000 lb forged weight. These pipe sizes are smaller than those suggested in the design concepts proposed by the architectural engineering firms that we have visited.

Gray Tool Company produces Grayloc fittings, a joint favored by most designers. Current connectors are limited to a 40-in. diam.

7.2 Inspection Techniques for Wear- and Process-Resistant Coatings

R. W. McClung and G. W. Scott

Literature search, equipment searches, and techniques evaluation are continuing. Corona discharge, electrolytic probe, and microwave techniques are earmarked for further investigation.

Preliminary beta-ray backscatter tests to measure coating thickness were unsuccessful. Further work with x-ray fluorescence, using a gamma source and cryogenic detector, indicates adequate sensitivity to measure .001-in. thickness changes in ZrO_2 coatings. Initial attempts at thermal testing did not provide confirmation of feasibility, but did provide an improved estimate of the type of technique required.

Dielectric (capacitance) gaging tests showed the ability to track the position of a dielectric surface with a linear response. Changes in the position of the surface of a uniform layer are linear; the absolute location of an arbitrary surface also depends on the thickness of the dielectric layer behind the surface. Modifications of standard liquid penetrant techniques yielded a method which discriminates against the extraneous indications typically encountered with porous surfaces. Cracks, holes, localized areas of high porosity, and the spraying pattern of the specimen are visible.

Review and Evaluation

We have continued to examine NDT literature for data or unconventional techniques which did not emerge as "favorites" from our initial survey. Techniques which appear promising in spite of an absence of available equipment or expertise include: corona discharge methods for hole or crack detection, electrolytic probe methods, also for holes or cracks, and microwaves for thickness measurement. Searches for equipment and some design efforts to support investigations of these techniques are in progress.

Radiation Techniques

Preliminary feasibility tests of beta-ray backscattering were made on ZrO_2 coating specimens (4 thicknesses), but no correlations between backscatter and coating thickness were obtained. The beta-ray source used was Pm^{147} (225 KeV). It appears that this energy is too high for use with thin coatings, particularly when the Z-value (atomic number) for the coating is close to the Z-value for th substrate. A lower energy beta-ray source such as C^{14} (156 KeV) might improve the response to coating thickness variation.

X-ray fluorescence testing of the ZrO_2 specimens and one Ni-Cr specimen was more successful. These specimens were tested on a production x-ray fluorescence gage at the UCCND Y-12 plant. Illuminating radiation came from a one-curie Gd^{153} source (97- and 103-KeV gamma) and fluorescence was measured with a cryogenic intrinsic Ge detector. The technique showed sufficient sensitivity to detect .001-in. thickness changes in ZrO_2 coatings. The two sides of the Ni-Cr specimen were distinguishable by changes in relative intensities of Fe and Ni lines. Coating thickness measurement may be feasible for this material, but thickness and change sensitivity could not be estimated from measurement of a single specimen.

Thermal Testing

Pulse heating experiments using an induction source were performed. The specimen (ZrO_2 coating) was heated with a 1 1/2-in. diam pancake coil on the uncoated side. The coated side was observed with an AGA Thermovision Camera, whose output was video-recorded. The thermal phenomena occurred too rapidly for detailed observation with this system, so the principal information gained was an estimate of the speed that will be required for other thermal techniques.

Dielectric (Capacitance) Testing

A capacitance gaging system was borrowed from the UCCND Y-12 plant to test its response to the dielectric ZrO_2 coatings. This instrument was equipped with a probe for measuring flat surfaces; it senses the separation between the surface and the probe. The range of the instrument was .020 in., .010 in. each direction from a zero point located .020 in. from a metal surface. The instrument response was linear over a range of .020 in., within .0001 in., when displaced relative to either a metal surface or a dielectric (coated) surface. With the dielectric surfaces, however, the zero point moves in closer to the surface; the amount of zero-point change is proportional to the coating thickness.

Liquid Penetrant

Specimen ANL-C5 was examined using two liquid penetrant techniques: (1) a standard procedure recommended by the penetrant manufacturer; and (2) a newer one, developed for "difficult," e.g., porous, surfaces. The newer process, "Interim Drying Equilibrium Augmentation" (IDEA), modifies the surface tension equilibrium behavior of the penetrant and allows it to be selectively stripped out of shallow features by water washing.

We used ZL-22A, a post-emulsified fluorescent penetrant. Conventional processing yielded indications over most of the surface, which were apparently generated by penetrant entrapment in coating porosity. These indications were nearly impossible to distinguish from indications caused by true holes or cracks in the coatings.

The IDEA processing produced much more localized indications consisting of bright spots, indicating holes or cracks, and small bright areas, indicating localized high porosity. The pattern of the spraying operation used to apply the coating was also visible.

7.3 Iron and Nickel Carbonyl Formation and Prevention

J. Brynestad and J. H. DeVan

The literature survey has been completed. The report is finished and is being processed.

A wet chemical method for the quantitative analysis of nickel carbonyl has been tested and optimized. The detection limit for a 50 liter gas sample by this method has been improved to 1 ppb. The method allows simultaneous analysis of iron and nickel carbonyl.

An atomic absorption spectrophotometer has been ordered. A sample of 1/2% Mo (A-335-P1) steel pipe from PERC has been rolled into 20 mil thick sheets for preliminary carbonyl reaction rate tests.

7.4 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 examination of the pressure let-down valve trim set from the Synthoil PDU at PERC is continuing. The scanning electron microscope (SEM) was used to obtain photomicrographs of the braze and to identify the chemical composition of the components. The braze is silver rich, about 72% based on a semi-quantitative analysis. The chemical analysis of the stem obtained in the energy dispersive x-ray spectrometer agrees with the nominal composition for a type 316 stainless steel. The valve seat (the valve-seat-insert was press-fitted into this part) was identified as a chromium-rich martensitic or ferritic stainless steel. The valve seat also contained small quantities of Mo and Si.

7.5 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.

Work was continued on initial phases of conceptual designs for HYGAS and Synthane gasifiers, with emphasis on the latter. To aid in collecting background information for the studies underway, a visit was made to Pittsburgh Energy Research Center and to Synthane Pilot Plant (both at Bruceton, Pennsylvania) on April 13-14, 1976. Very brief discussions were held on the Synthoil process and associated pilot plant. Major emphasis was on discussions of possible corrosion and erosion studies to be made using coupons placed at strategic locations in the Synthoil system.

A great deal of information germane to our studies was obtained from the Synthane Pilot Plant discussions. The entire mechanical system was discussed beginning with the flow diagram and following up with discussions of segments of the plant and individual components. The gasifier and the design details associated with it received a major share of attention; the equipment that attaches directly to the gasifier also was discussed at length. The information obtained will be of direct benefit in providing needed guidance for pressure vessel conceptual design preparation.

Under Task 1, the conceptual design task, sizing of the concrete vessels for different conditions was examined both for the Synthane and the HYGAS process. Wall thicknesses were calculated based on combined thermal stress and internal pressure considerations. Penetration locations and designs were addressed, and preliminary closure studies were carried out. The latter were associated with the access opening at the top of the Synthane vessel, where it was found attractive to examine the use of a steel closure plug. One of the advantages of a steel plug is the potential for supporting the internal cyclone from the plug.

Under Task 2, Tendon Considerations, preliminary study has been given to tendon arrangements. The manways needed for access to the interiors of the vessels for inspection, maintenance, and replacement of parts require special attention in tendon design.

Effects of discontinuities under Task 3 are being examined as a part of the closure studies. Under this task, liner analysis needs are being investigated, and areas requiring detailed analysis are being defined. Examples of areas that require detailed treatment are the discontinuity regions around penetrations.

Liner studies to date under Task 4 have been based primarily on the cold liner concept. As a part of the overall study, it is necessary to make scoping studies of refractory requirements and characteristics, to consider conceptual designs of internal structures and attachments to the liner, and to estimate loadings imposed on the liner. Work of this type is being done; included in the considerations are vapor stops, cyclones and cyclone supports, supporting grids for fluidized beds, etc. Estimates of forces transmitted to the liner for the Synthane gasifier were made. Finally, work was initiated on developing a hot liner concept for the Synthane gasifier. In this case, the temperature at the inside wall of the liner would be maintained at a level just above the water vapor dewpoint for the operating pressure.

7.6 Other Related Work

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

Fluidized Bed Material Support Activities

Fireside Corrosion in a Fluidized Bed Coal Combustor. — During the previous three months, numerous discussions have been held with representatives of the Fluidyne Engineering Corporation. The three primary purposes of their discussions were: (1) to aid Fluidyne in finalizing the design of their atmospheric pressure fluidized bed test facility; (2) to establish test conditions for test coupons; and (3) to complete the design of surveillance coupons to be given a 500 hr exposure in this test bed.

Based on the current design, the 18-in. x 18-in. fluidized bed will contain 55 1/2-in. OD tubes. Thirty-nine of these tubes will be used to control the bed temperature to approximately 1600°F. The remaining sixteen tubes will be test specimen tubes. These tubes will be maintained at a wall temperature of approximately 1575°F throughout the test by a continuous flow of air through each tube. Each of these tubes will have an 18-in. length exposed to the bed environment. In order to maximize the number of materials that can be tested, the 18-in. long test section of each tube will be made of three six-in. segments. This arrangement will give a total of 48 test locations.

In recent meetings, the location of the 16 test tubes within the total field of 55 tubes in the bed was determined. Materials to be evaluated in this surveillance program are Incoloy 800, Incoloy 800 aluminized, 310 stainless steel, 310 stainless steel aluminized, 316, 304 stainless steel, and Inconel 600. At this time delivery of all tube segments to Fluidyne is proceeding on schedule and should reach Fluidyne by May 15.

8. ENGINEERING EVALUATIONS OF THE SYNTHOIL AND HYDROCARBONIZATION PROCESSES

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

Summary

Synthoil. Flow diagrams, material balances, and utilities requirements were completed for the entire facility. A computer program was written to calculate the product price required to yield a specified rate of return or investment when the capital investment and operating costs are known. Work continued on the preliminary cost estimate. The overall cost estimating program using the factor method was tested on several units and checked against other estimates; the equipment cost factors are being refined to make results check more closely.

Hydrocarbonization. Flowsheets were reviewed for compatibility, equipment lists were brought up to date, and utilities requirements were compiled from the individual flowsheets. The char recovery and storage subsystem flowsheet was completed. The design status of the fluid-bed combustor was reviewed with representatives of the Foster-Wheeler Corporation. Completed flowsheets and preliminary detail design drawings were presented, and sizes of major components and the selection of critical design temperatures were described and discussed.

8.1 Synthoil Process

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

Simplified flow diagrams, material balances, and utilities requirements were completed for the oxygen plant (Unit 25) and the ammonia plant (Unit 28). This completes all the flow diagrams, material balances, and utilities requirements for the entire facility.

A computer program was written to calculate the product price required to yield a specified rate of return on investment when the capital investment and operating costs are known. The program uses the discounted cash flow procedure. Depreciation allowances and taxes are calculated internally. Provision is made for several alternative depreciation methods for tax purposes. The program handles working capital, investment tax credits, and any desired debt-equity ratio. As an option, the program can be used to calculate the rate of return on investment when the product prices are specified. We plan to use this program to calculate the economics of the Synthoil and Hydrocarbonization processes.

Work continued on the preliminary cost estimate. Vendor estimates were obtained for the cooling towers and cooling tower pumps. A computer program was written to develop the total installed cost of the various plants in each facility using the modular approach, in which a capital cost is developed from purchased equipment costs using various factors for piping, foundations, other material, labor, etc.[1] The program was tested on an example problem, using process equipment costs for the Hydrocarbonization unit, and gave an overall plant cost that seems reasonable when checked against other estimates. In another test, the program was used to determine capital costs for three plants for which both purchased equipment costs and installed costs were available. The results obtained were within ±5% of published figures. In a test on an acid gas treating unit, the overall cost appeared to be on the low side. The equipment cost factors are being refined in an effort to make the results check more closely.

A computer program was written to estimate the costs of vessels. The program develops the weight of the vessel, given the diameter, length and type of head, estimating the vessel wall thickness if it is not provided. The weight is multiplied by a cost function, based on data from the open literature, where the cost per pound varies with wall thickness, weight, and material of fabrication. The cost for refractory or other linings is added for a complete vessel cost. When the quotes on a representative assortment of vessels are received from Chicago Bridge and Iron, the cost functions will be calibrated with the vendor data.

8.2 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 effort was placed on revising flowsheets of the various sections and making them compatible with one another. Equipment lists were brought up to date, and a list of utilities requirements was compiled from the individual flowsheets. Various units of the plant progressed as follows:

(1) Hydrocarbonization: The char recovery and storage subsystem flowsheet was completed. The char is mixed with water at 125 psia and fed through chokes for screening through 8-mesh screens at atmospheric pressure. Oversize char is dewatered centrifugally and reduced in a closed-circuit hammermill-screen system to 8 x 0 mesh size. Undersize material from both screening operations is combined and sent to holding tanks. Piston pumps, fed by centrifugal slurry pumps, inject the slurry into the pipeline at 1000 psi. An alternate subsystem using rod mills was investigated, but costs were much higher.

(2) Fluid-bed combustor: Foster-Wheeler completed their second-month design effort on the FBC unit for the hydrocarbonization plant. As a result of further studies, the FBC coal dryer was eliminated by

decreasing the surface moisture of the feed coal from 5% to 2%. The high ash coal dryer in the beneficiation unit is used to dry the coal down to the 2% level. Elimination of the FBC coal dryers decreases the overall plant costs. The flowsheet and plan layout are being modified to reflect these changes. At the design review meeting held at Foster-Wheeler on April 1, extensive discussions on plant startup resulted in the addition of oil fired package boiler to be used until an FBC module pair can be brought up to power. Air or nitrogen will be used as process gases until process gases are generated. Final design of the combustor and auxiliary equipment is nearing completion and cost estimating has been initiated.

8.3 References for Section 8

1. K. M. Guthrie, Process Plant Estimating Evaluation and Control, Craftsman Book Co., Solana Beach, California (1974).

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