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U.S. DEPARTMENT OF ENERGY
Division of Coal Conversion

QUARTERLY REPORT

April - June 1977

DOE / ET - 0026 / 2

Publication Date: January 1978

COAL

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COAL LIQUEFACTION

QUARTERLY REPORT

APRIL - JUNE 1977

MASTER

U.S. DEPARTMENT OF ENERGY

Division of Coal Conversion

Washington, D.C. 20545

Publication Date: January 1978

FOREWORD

This document was prepared by the Energy Research and Development Administration (ERDA) prior to the activation of the Department of Energy (DOE) by P.L. 95-91 on October 1, 1977. Therefore, wherever ERDA is mentioned, it should be noted that its functions have been transferred to DOE.

The technical reviews contained in this document were prepared by Cameron Engineers, Inc., Denver, Colorado, based on information supplied by DOE and its various contractors. The reviews, as they appear herein, have been authorized for publication by representatives of DOE. Cameron Engineers, Inc. assumes responsibility for the accuracy of the reviews but not for the accuracy of the information provided by either DOE or its contractors.

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EXECUTIVE SUMMARY

The United States has more energy available in coal than in petroleum, natural gas, oil shale, and tar sands combined. Nationwide energy shortages, together with the availability of abundant coal reserves, make commercial production of synthetic fuels from coal vital to the Nation's total supply of clean energy. In response to this need, the Office of Fossil Energy of the Energy Research and Development Administration (ERDA) is conducting a research and development program to provide technology that will permit rapid commercialization of processes for converting coal to synthetic liquid and gaseous fuels and for improved direct combustion of coal. These fuels must be storable and suitable for power generation, transportation, and residential and industrial uses.

ERDA's program for the conversion of coal to liquid fuels was begun by two of ERDA's predecessor agencies: Office of Coal Research (OCR) in 1962, and Bureau of Mines, U.S. Department of the Interior, in the 1930's. Current work is aimed at improved process configurations for both catalytic and non-catalytic processes to provide more attractive processing economics and lower capital investment. The advantage of coal liquefaction is that the entire range of liquid products, including heavy boiler fuel, distillate fuel oil, gasoline, jet fuel, and diesel oil, can be produced from coal by varying the type of process and operating conditions used in the process. Furthermore, coal-derived liquids also have the potential for use as chemical feedstocks.

To provide efficient and practical means of utilizing coal resources, ERDA is sponsoring the development of several conversion processes that are currently in the pilot plant stage. Responsibility for the design, construction, and operation of the *Solvent-Refined Coal (SRC) Process* pilot plant at Ft. Lewis, Washington is assigned to Pittsburgh and Midway Coal Mining Company. SRC II modifications were completed at the plant during the quarter and two SRC II material balance runs were completed. The laboratory continued development of the SRC process through a series of exploratory runs with Wyoming subbituminous coal and a Pittsburgh seam coal.

Ashland Synthetic Fuels, Inc. (ASFI) is responsible for management of the construction of the *H-Coal Project* pilot plant. Construction continued on schedule and was 12.66 percent complete at the end of the quarter. Hydro-

carbon Research, Inc. (HRI) continued modifications and revisions of the process development unit (PDU) and design work on the pilot plant.

ERDA together with the Electric Power Research Institute (EPRI), has contracted with Southern Services Company, Inc. for the *Operation of a Solvent-Refined Coal Pilot Plant* at Wilsonville, Alabama. Emery coal from Utah was processed most of the quarter in order to provide data for producing SRC from this coal. In June, processing of Indiana V coal began.

Using engineering and design technology similar to that practiced in the petroleum industry, the *Donor Solvent Liquefaction Process* is being developed by Exxon Research and Engineering Company. As part of Phase III A, lignite operations were concluded in the 50-pound-per-day Recycle Coal Liquefaction Unit during the quarter. Operations with Illinois No. 6 coal were completed on the one-ton-per-day Coal Liquefaction Pilot Plant and operability testing of Wyoming coal began.

Cities Service Research and Development Company is conducting sub-scale studies as the first phase of the *Commercial Scale Ebullated-Bed Hydroprocessing of Solvent Refined Coal Extract*. The hydrogenation of solvent in the LC-Fining unit was completed during the quarter.

Fluor Engineers and Constructors, Inc. is responsible for reactivating an ERDA-owned equipment test and evaluation facility in Cresap, West Virginia. Further renovation work and some startup operations continued at the *Cresap Test Facility* throughout the quarter.

Construction of the 10 ton-per-day process development unit (PDU) to test the *Synthoil Process* is being managed by Foster Wheeler Energy Corporation. Overall total construction effort of the PDU was 65 percent complete at the end of the quarter. Associated laboratory research was conducted by the Pittsburgh Energy Research Center (PERC), Sandia Laboratories, Argonne National Laboratory, and Battelle Memorial Institute (Columbus Laboratories).

The *Clean Coke Process* is under development by USS Engineers and Consultants, Inc., a subsidiary of United States Steel Corporation. The results of laboratory tests and data obtained from the process development unit (PDU) will be used for possible scale-up. Carbonization PDU tests were conducted during the quarter with West Kentucky coal. The binder-preparation PDU continued to process liquid blowdown from the carbonization PDU. The last tests using raw coal were completed in the hydrogenation PDU, and studies of two-stage vapor stripping of residues from various liquefaction process began.

The Rocketdyne Division of Rockwell International is conducting a program to develop a technology which will permit the *Liquefaction of Coal by Direct Hydrogenation* in an entrained-flow reactor. The initial series of one-ton-per-hour reactor tests was completed during the quarter. Approval to continue testing was received and three additional tests were completed.

Studies are being conducted by the Occidental Research Corporation to evaluate the commercial potential of the company's *Flash Pyrolysis Coal Liquefaction Process*. The planned work with subbituminous coal was completed during the quarter, and process development unit (PDU) tests with bituminous coal began. Hydrotreating of subbituminous coal and catalyst screening tests continued.

Research to improve solid-liquid separation is being conducted by Johns-Manville Sales Corporation in their *Filtration Process and Equipment Studies*. During the quarter, a total of 57 filtration runs were made. Fifty-two runs were made on unfiltered solvent refined coal (SRC) from the Fort Lewis Pilot Plant, and five runs were made on filter feed from the Wilsonville facility.

Dow Chemical Company has conducted studies dealing with the production of *Chemicals From Coal* using the liquids from four coal liquefaction processes (COED, Synthoil, H-Coal, and SRC) in standard petrochemical processing experiments. Experimental studies were completed at the beginning of the year. Efforts during the quarter were directed at compiling the interim reports. Work on the conceptual design for a plant to produce chemical from coal liquefaction products continued.

Oak Ridge National Laboratory is providing *R&D Engineering Services* dealing with *Hydrocarbonization of Coal*. Hydrocarbonization is a type of coal liquefaction process which combines high-temperature, high pressure, and fluidized-bed operation using hydrogen-rich gas for fluidization to enhance product yields. Hydrocarbonization experiments used an agglomerating coal, Illinois No. 6, for the first time during the quarter.

Conoco Coal Development Company, with the participation of Shell Development Company, is developing *Zinc Halide Hydrocracking Process*, a technique designed to produce clean liquid and gaseous fuels from coal or coal extract. All major equipment for the 1-ton-per-day process development unit had been received or was in transit at the end of the quarter.

Exxon Research and Engineering is studying the potential of *Fluid Coking of Coal Liquefaction Residues* to cover distillate liquids. During the quarter, analytical tests were performed on a sample of stripped SRC solvent

deashing underflow provided by Conoco Coal Development Company as well as a one-gallon sample of stripped SRC solvent deashing underflow from C. E.-Lummus.

To facilitate the possibly desirable upgrading of primary coal liquids using technology in the petroleum industry, UOP, Inc. is conducting a study of *Upgrading of Coal Liquids*. First-stage hydrocracking of rerun H-Coal process atmospheric still bottoms continued throughout the quarter. All required analyses for the upgraded H-Coal naphtha were completed in April.

Texaco, Inc. is conducting a study of the *Gasification of Residual Coal Liquefaction Products*. Material from several coal liquefaction pilot plants will be evaluated to determine their suitability as feedstock for the proprietary Texaco partial oxidation process.

The *Production of Clean Industrial and Transportation Fuels From Coal* is being investigated by the Lummus Company using a multistage ebullated-bed catalytic reactor developed through the firm's activities with a bench-scale pilot plant. Integrated shakedown operations continued during the quarter on the modified 30-pound-per-hour bench-scale expanded-bed test unit.

I. SOLVENT-REFINED COAL (SRC) PROCESS

THE PITTSBURGH & MIDWAY COAL MINING
COMPANY

MERRIAM, KANSAS

Plant Site: Tacoma, Washington

Contract No.: E(49-18)-496

Contract Funding: \$88,759,707
(100% ERDA)

INTRODUCTION

The SRC process is being developed by Pittsburgh & Midway Coal Mining Company (P&M), a subsidiary of Gulf Oil Corporation, under the sponsorship of ERDA. The specific objectives of the contract are to (1) continue obtaining technical and economic data from the pilot plant to validate scale-up to commercial production and (2) provide large samples of solvent-refined coal and by-products from the process for market development studies. Figure I-1 provides a schedule for the development of the process.

PROCESS DESCRIPTION

The SRC process converts high-sulfur, high-ash coals to low-sulfur, low-ash liquid fuel. Figure I-2 shows a schematic of the process. The coal is first pulverized and mixed with a coal-derived solvent in a slurry mix tank. The slurry is combined with hydrogen and is then pumped through a fired preheater and passed into a dissolver. In this unit, the coal is hydrogenated and thereby depolymerized, leading to an overall decrease in product molecular weight and dissolution of the coal. The solvent is also hydrocracked in the dissolver unit, yielding lower molecular weight hydrocarbons ranging from light oil to methane. Yet another reaction occurring

in the dissolver is the hydrogenation of the organic sulfur in coal, thus producing hydrogen sulfide.

From the dissolver, the mixture passes to a separator where the gases are separated from the slurry of undissolved solids and coal solution. The raw gas is sent to a hydrogen recovery and gas desulfurization unit. The recovered hydrogen is then recycled and combined with the slurry coming from the slurry mix tank. Hydrocarbon gases are released and recovered and the hydrogen sulfide is converted to elemental sulfur.

The slurry of undissolved solids and the coal solution is then separated in a filtration unit. In a commercial-scale process, the solids would be sent to a gasifier-converter where they would react with supplemental coal, steam, and oxygen to produce hydrogen for use in the process. The coal solution passes to the solvent recovery unit, and the final liquid product, solvent-refined coal, is produced. This material has a solidification point of 350°F to 400°F and a heating value of approximately 16,000 Btu per pound.

HISTORY OF THE PROJECT

The SRC project was begun in 1962 when Spencer Chemical Company was awarded a research contract by the Office of Coal Research (OCR, now part of ERDA) to study the technical feasibility of a coal de-ashing

LEGEND: △ BEGIN MILESTONE
▽ COMPLETE MILESTONE

Figure I-1. SOLVENT-REFINED COAL PROGRAM SCHEDULE

Figure I-2. SRC PROCESS SCHEMATIC

process (now called the SRC process). In 1965, the process was successfully demonstrated in a 50-pound-per-hour continuous-flow unit, and the work on the contract was therefore completed. During the term of the contract, Gulf Oil Corporation acquired Spencer Chemical Company. After reorganization, the contract was assigned to the research department of P&M.

To develop the SRC process further, a contract was awarded to P&M to design, construct, and operate a pilot plant that would be capable of processing 50 tons of coal per day. In 1969, Stearns-Roger Corporation completed the design for the pilot plant, but funds to begin construction were not available until late 1971. In June 1972, OCR extended its contract with P&M to construct and operate the pilot plant. Rust Engineering Company began constructing the pilot plant in July 1972 at Ft. Lewis, Washington, near Tacoma. The pilot plant became fully operational in October 1974.

Tests run during 1975 studied the effects of different coal feed rates and dissolver temperatures. A 3,000-ton run was begun late in 1975 for production of specification SRC for use in combustion testing by Southern Services Company's Georgia Power Company (see Section III) and was completed in December of 1976.

After modification of filtration instrumentation following the production run, filtration tests were conducted. The plant was then shut down for a major maintenance turnaround and SRC II (slurry recycle) piping tie-ins. Plans had been completed during the year for modifications of the pilot plant for SRC II testing.

The laboratory unit has been run in both the distillate solvent mode and slurry recycle mode. The variables investigated included filtration rate, solvent recycle rate, slurry residence time, concentration of coal in the slurry, and temperature. Tests were also run to simulate operating conditions at the pilot plant. A series of experimental runs were designed to develop the SRC II process. The effect of process variables in SRC II operation was investigated.

In addition to laboratory experiments conducted at the P&M Merriam, Kansas laboratory, other research supporting the development of the SRC process included:

- Formulation of an interagency agreement between EPA and ERDA to conduct environmental studies concerning the removal of sulfur and nitrogen compounds from the coal.
- Experiments by Oklahoma State University to test the efficiency of commercial catalysts in removing sulfur and nitrogen from coal liquids, and to deter-

mine the rate of decay of hydrogenation catalyst activity.

- Tests at Washington State University to determine the content of coal mineral residues and trace elements in the SRC process.

PROGRESS DURING APRIL-JUNE 1977

Summary

SRC II modifications were completed during the quarter and two SRC II material balance runs were completed.

The pilot plant was operated for eight days at the beginning of the quarter in the SRC I mode before the final changeover to SRC II. During that period of operation, three filter test runs were completed and filter feed samples were collected for laboratory deashing tests being conducted by the Lummus Company.

The Merriam laboratory continued development of the SRC process through a series of exploratory runs with Wyoming sub-bituminous coal and a Pittsburgh seam coal.

Pilot Plant Operation

After a two and one-half months shutdown for SRC II modifications, the plant was returned to service at the end of April and operated in the SRC I mode. Purpose of the run was to supply fresh samples of filter feed slurry for deashing studies being conducted by Lummus in the plant laboratory, and to conduct additional filtration studies on filter "B". A total of 204 tons of Kentucky coal was processed in eight days. The plant was operated with a half dissolver (50 ft³) at 3500 pounds per hour coal feed rate, solvent to-coal ratio of 1.67, 1500 psig, and 850° F dissolver outlet temperature.

After the run was completed, the plant was shut down for final SRC II modifications. These included the repiping of dissolver "A" for full dissolver operation, and the installation of the modified dissolver head, thermocouples, and quench system.

Except during startup and periods of upset conditions, all operation in May was in the SRC II mode. A total of

366 tons of Kentucky 9 and 14 coal were processed during 18 days of operation. Problems were experienced with ball valves around the high pressure let-down valve, with the new vacuum flash system, and with the seal flush from the stripper side draw accumulator. Intermittent plugging occurred in the new coal feed and mixing system.

In June, two SRC II material balance runs were completed. Total liquid yields of 35-38 percent were obtained with SRC yields of about 20 percent. These runs were made at the base conditions of 24-26 percent coal feed in a total slurry of about 6600-6700 pounds-per-hour with a full dissolver at 860°F and 1900 psig. During 25 days of operation, 449 tons of Kentucky 9 and 14 coal were processed. The plant was shut down at the end of June and was down at the end of the quarter. Because of continuing problems with the new coal feed and vacuum flash systems, the original systems were returned to service pending further modifications.

Laboratory — Merriam, Kansas

Three laboratory runs were made in April with Amax coal, varying temperature, pressure, and residence time. One run was made to determine if the plugging tendency could be reduced by inclusion of unfiltered coal solution in the slurry feed to increase viscosity and inhibit settling

of mineral matter in the dissolvers. The duration of the run was not sufficient to demonstrate the absence of any long-term plugging problems. During the runs, it appeared that improved reactivity could be correlated with accumulation of iron in the reactor.

An initial SRC I run was made in April with the Pittsburgh Seam coal. Six SRC II runs were made with the Pittsburgh Seam coal in May. Two of the runs were made to illustrate the effect of iron addition and allow comparisons of reactivity of the Loveridge Mine coal with the Kentucky coal from the Colonial Mine. The lower iron content of the Loveridge Mine coal was believed to be a major factor in the lower reactivity of that coal in comparison to Kentucky coal. Iron spiking to a level similar to that in the Kentucky coal substantially increased reactivity but it was still less than that of the Kentucky coal.

In June, four SRC II runs were completed. Three runs feeding Kentucky 9 and 14 coal were made to explore the effect of hydrogen circulation rate at lower rates than earlier work. Decreased hydrogen feed rate resulted in decreased hydrocarbon gas yields, decreased distillate oil yield, increased vacuum residual yield (SRC), and decreased MAF conversion. One run was an additional attempt to reach steady state operation using a slurry feed containing only recycle slurry and Loveridge Mine coal. Slurry pump problems again precluded steady state operation.

II. H-COAL PROJECT

Plant Site: Catlettsburg, Kentucky

Total Funding: \$179,000,000

ERDA: \$143,000,000

Industry: \$ 36,000,000

HYDROCARBON RESEARCH, INC.

TRENTON, NEW JERSEY

Contract Nos.: E(49-18)-1544 and
EF-77-C-01-2547

ASHLAND SYNTHETIC FUELS, INC.

ASHLAND, KENTUCKY

Contract No.: EX-76-C-01-2260

INTRODUCTION

The H-Coal process is being developed by Hydrocarbon Research, Inc. (HRI) under the joint sponsorship of (1) ERDA, (2) a private industry consortium composed of Electric Power Research Institute, Ashland Synthetic Fuels, Inc., Conoco Coal Development Company, Mobil Oil Corporation, and Standard Oil Company (Indiana), and (3) the Commonwealth of Kentucky. The overall objectives of this project are to further develop the H-Coal process and to demonstrate its technical and economic feasibility on larger scales. The specific objectives are to:

- Perform laboratory research on all phases of the H-Coal process, using the existing bench-scale unit and process development unit to establish design criteria.
- Design a pilot plant capable of converting 600 tons of coal per day to 2,000 barrels per day of low-

sulfur boiler fuel or 200 tons of coal per day to 2,000 barrels per day of synthetic crude oil.

Ultimately, the pilot plant will be constructed and operated to provide data for further evaluation of the process and design of a full-size commercial plant. The overall program plan is shown in Figure II-1.

The H-Coal Project is being conducted in three phases:

- Phase I - Design
- Phase II - Construction
- Phase III - Operation

Hydrocarbon Research, Inc. (HRI) developer of the H-Coal process, designed the H-Coal Pilot Plant under Phase I of the H-Coal Project.

Ashland Synthetic Fuels, Inc. (ASFI) is responsible for construction and operation of the H-Coal Pilot Plant under Phases II and III of the H-Coal Project.

LEGEND : BEGIN MILESTONE
 COMPLETE MILESTONE
 DECISION MILESTONE
● EVENT
1 RELEASE RFP
2 PROPOSALS RECEIVED
3 CONTRACTOR SELECTED
4 CONTRACT AWARDED
5 ORDER LONG-TERM ITEMS

Figure II-1. H-COAL PROGRAM SCHEDULE

PROCESS DESCRIPTION

The H-Coal process is a catalytic hydroliquefaction process that converts high-sulfur coal to boiler fuels and synthetic crude. A schematic of the process is provided in Figure II-2. Coal is crushed to minus 60 mesh, dried, slurried with recycled oil, and then pumped to a pressure of about 200 atm. Compressed hydrogen is added to the slurry and the mixture is preheated and charged continuously to the bottom of the ebullated-bed catalytic reactor. The upward passage of internally recycled reaction mixture maintains the catalyst in a fluidized state. Catalyst activity is maintained by the periodic addition of fresh catalyst and the withdrawal of spent catalyst. The temperature of the ebullated-bed catalytic reactor is controlled by adjusting the preheater outlet temperature. Typically, the temperature of the mixture entering the reactor is 650°F to 700°F.

Vapor product leaving the top of the reactor is cooled to separate the heavier components as a liquid. Light hydrocarbons, ammonia, and hydrogen sulfide are absorbed from the gas stream and sent to a separator and a sulfur recovery unit. The remaining hydrogen-rich gas is recompressed and combined with the input slurry. The liquid from the condenser is fed to an atmospheric distillation unit. The liquid-solid product from the

reactor, containing unconverted coal, ash, and oil, is fed into a flash separator. The material that is flashed off is passed to the atmospheric distillation unit which yields light and heavy distillate products. The bottoms product from the flash separator (solids and heavy oil) is further separated with a hydroclone, a liquid-solid separator, and by vacuum distillation.

The gas and liquid products, composed of hydrocarbon gas, hydrogen sulfide, ammonia, light distillate, heavy distillate, and residual fuel, may be further refined as necessary. A portion of the heavy distillate is recycled as the slurry medium. The stream containing the unreacted carbon and some liquid will eventually be processed in a commercial installation to produce additional hydrogen needed for the process.

The specific operating conditions of the H-Coal process affect the type of fuel produced. For example, to produce synthetic crude, more hydrogen is required and there is a lower yield of residual fuel oil. In this mode, the separation of the solids (unconverted carbon and ash) from the liquid can be accomplished by vacuum distillation, and a special liquid-solid separation unit is not required. To produce gas and low-sulfur residual oil as major products clean fuel, the temperature and pressure in the ebullated-bed reactor are lowered, and less hydrogen is required.

Figure II-2. H-COAL PROCESS SCHEMATIC

Before the H-Coal process can become commercially and economically competitive, an adequate supply of hydrogen must be generated from the process itself. The H-Coal process requires between 14,000 and 20,000 standard cubic feet of hydrogen for each ton of coal processed, depending on the type of oil produced. In the pilot plant, some of the hydrogen required has been obtained from the gas produced by the ebullated-bed reactor. Additional hydrogen has been purchased to meet processing demands. In a commercial operation, hydrogen demand will be met by manufacture on site. Because the H-Coal process converts about 90 percent of the carbon contained in the coal to a liquid, the feed to the hydrogen plant could be liquid rather than solid. This suggests that commercial hydrogen manufacturing processes could be adapted. The solid cake char could also be used to produce hydrogen.

In the H-Coal process, hydroclones are used to remove about two-thirds of the solids from recycled liquid used

for slurring the coal. Anti-solvent precipitation is the method used to remove solids from H-Coal fuel oil. Solids will be removed from the syncrude as vacuum tower bottoms.

HISTORY OF THE PROJECT

The H-Coal process was developed by HRI as a further application of the ebullated-bed processing technology originally used to convert heavy oil residues from petroleum into lighter fractions (H-Oil process). Early development of the H-Coal process, beginning in 1964, involved research with a bench-scale unit and a process development unit and preparation of a conceptual process design. An independent evaluation in 1968 confirmed the technical and economic feasibility of the H-Coal process.

The H-Coal process proved to be highly flexible in bench-scale and process development unit tests. The bench-scale unit can process 25 pounds of coal per day, the process development unit can process three tons of coal per day. Many types of coal have been tested satisfactorily on the bench-scale level, including eastern, midwestern, and western bituminous coals, western subbituminous coals, lignite from Texas and North Dakota, and Australian brown coal. The bench-scale unit has also provided information on the effect of process variables (temperature, pressure, coal feed rate, slurry oil composition, and catalyst age) on coal conversion and product yield. The process development unit tests demonstrated sustained H-Coal process operation with satisfactory control of the catalyst bed expansion.

Based on the data obtained from the bench-scale and process development units, design and engineering of the 700-600 ton per day pilot plant began in December 1973. During 1974, design of the coal preparation section of the pilot plant was completed to the extent that engineering and plant layout could begin. HRI concentrated, however, on the design and engineering for the ebullated-bed reactor, since the equipment needed for this section requires long lead times. Engineering for other sections of the pilot plant was started in late 1974. Also during 1974, three possible sites for construction of a pilot plant were evaluated. Based on detailed environmental assessments, it was decided during the second quarter of 1975 to locate the plant adjacent to the Ashland Oil, Inc. refinery in Catlettsburg, Kentucky.

Except for the steam supply, this arrangement would allow the utility system of the pilot plant to be shared with that of the refinery. All process descriptions, equipment specifications, and planning drawings were completed for the coal preparation section. Work in the hydrogenation area involved the modification and rearrangement of major equipment to conform to the physical characteristics of the new plant site. Preliminary

process designs were also completed for the solid-liquid separation techniques of filtration, centrifugation, and solvent precipitation.

Ashland Synthetic Fuels, Inc. (ASFI) is responsible for the construction of the plant under contract No. E(49-18)-2260. Field work began in December 1976 by Badger Plants Inc., subcontractor to ASFI. The Lummus Company will design the antisolvent deashing unit for the pilot plant. HRI will provide engineering, design services, and field support as a subcontractor to ASFI during the construction and operation phases.

PROGRESS DURING APRIL-JUNE 1977

At the end of the quarter, construction of the H-Coal Pilot Plant was 12.66 percent complete. Eighty percent of the equipment and commodities for the plant were either on order or committed as of the end of June. The remaining 20 percent was in various intermediate processing stages prior to placing an order.

Under support activities, HRI continued modifications and revisions of the process development unit (PDU). PDU Run 5, scheduled for June, was delayed due to mechanical problems.

Viscosity data on vacuum still bottoms product were obtained in April to re-establish the design basis for the Pilot Plant system.

An American Cyanamid nickel molybdate and a Harshaw catalyst for bench-scale denitrogenation catalyst testing were identified as commercially available in Pilot Plant quantities.

Kerosine-nitrogen entrainment studies continued throughout the quarter in an effort to complete the Pilot Plant Recycle Cup design recommendations.

III. OPERATION OF SOLVENT-REFINED COAL PILOT PLANT

SOUTHERN COMPANY SERVICES, INC.
BIRMINGHAM, ALABAMA

Plant Site: Wilsonville, Alabama
Contract No.: E(49-18)-2270
Contract Funding: \$8,729,892
ERDA: \$5,741,892
Industry: \$2,988,000

INTRODUCTION

The operation of the solvent-refined coal (SRC) pilot plant at Wilsonville is jointly sponsored by ERDA and the Electric Power Research Institute (EPRI). The schedule for the project is shown in Figure III-1. The objectives of the project are to:

- Correlate the results of operation at the Wilsonville SRC pilot plant with those from the Ft. Lewis, Washington SRC pilot plant.
- Test several coals of various ranks and locations to determine the effects of process variables on product yield and quality.
- Evaluate SRC process improvements.
- Evaluate improvements in equipment and operations.

PROCESS DESCRIPTION

The SRC process converts high-sulfur, high-ash coals to a clean distillate fuel and a clean high-Btu gas. A schematic of the process is shown in Figure III-2. The pulverized coal is mixed with two to three parts process-derived solvent. Hydrogen is then added to the coal-solvent slurry. The mixture is pumped to a preheater and then fed into a single-stage reactor or dissolver which

Figure III-1. SRC PILOT PLANT OPERATION SCHEDULE

operates at 800°-875° F, and a total pressure of approximately 1700 psig. Under these conditions, approximately 93 percent of the carbonaceous material in the coal feed is dissolved during the residence time of approximately 45 minutes. The product gases from the dissolver are separated and sent to a clean-up system to remove the hydrogen sulfide (about 60 percent of the organic sulfur in the coal is converted to hydrogen sulfide) and convert it to elemental sulfur. The liquid slurry is then subjected to a mineral separation step wherein the undissolved solids are removed. Process solvent is recovered from the coal solution by distillation and recycled to slurry the coal feed. The residue which remains is the solvent refined coal product, which solidifies at about 350° F.

Figure III-2. SRC PROCESS FLOW DIAGRAM

HISTORY OF THE PROJECT

In March 1972, the Edison Electric Institute and the Southern Company Services, Inc. began a joint project to study the key steps in the SRC process. Consequently, Catalytic, Inc. designed, built, and is operating the six-ton-per-day pilot plant. The facility was completed in August 1973. The Electric Power Research Institute (EPRI) assumed the responsibilities of the Edison Electric Institute for utility industry sponsorship in April 1973.

The pilot plant began operation in January 1974, and by the end of 1976, had been in operation for more than 13,500 hours, including periods of 45 and 75 days of sustained operation. One subbituminous and four bituminous coals have been tested. SRC product meeting plant specifications of 0.16 percent maximum ash and 0.96 percent maximum sulfur has been produced from each coal. A fuel meeting these specifications and having

a heating value of 16,000 Btu per pound could be used to achieve compliance with most emission limits and EPA's "new source performance standards."

PROGRESS DURING APRIL-JUNE 1977

Summary

Emery coal from Utah was processed most of the quarter in order to provide data for producing SRC from this coal. In June, processing of Indiana V coal began.

Pilot Plant Operations

During April, the pilot plant operated 458 hours and was on stream 64 percent of the time. The distillation

section operated an additional 27 hours to condition process solvent.

A reconstituted coal run (Run 119) using Emery coal from Consolidation Coal Company's Emery, Utah mine was completed at the beginning of April. After the run, process work continued with Emery coal producing SRC product.

In Run 119, the feed rate was increased to 600 pounds per hour, coal concentration was increased from 25 to 27 percent, and the dissolver pressure was lowered from 2400 to 2000 psig. Wash solvent was added to the process solvent to give 20 to 25 percent minus 450°F boiling range fraction in the process solvent because of previous indications that lighter solvent promoted higher conversions. The coal conversion remained between 70 and 74 percent, however.

The purpose of Runs 120 and 121 was to compare plus 350°F boiling range process solvent with plus 450°F boiling range solvent using Emery coal. The conditions for Run 120 were: 27 percent coal concentration, 380 pounds-per-hour feed rate, 2450 psig, 825°F dissolver outlet temperature, and 9000 scfh of feed gas at 85 percent hydrogen purity. Conditions of Run 121 were also the same as 120 except that plus 450°F boiling range solvent was used instead of plus 350°F boiling range solvent as in Run 120. Comparison of the data for the runs follow:

	Run 118	Run 119	Run 120
Conversion, % MAF Coal	74	71	76
Hydrogen Consumption, % MAF Coal	1.5	1.1	2.6
SRC Yield, % MAF Coal	51	54	50
Gas Make, % MAF Coal	8	10	11
Sulfur in SRC, wt. %	0.4	—	0.4

Conditions for Run 122 were the same as for Run 121 except the dissolver temperature was lowered from 825 to

815°F, the feed rate was increased to 500 pounds-per-hour from 375 pounds-per-hour, and the coal slurry concentrations was increased from 27 to 33 percent. The coal conversions of Emery mine coal reached a maximum of 83-84 percent for both Runs 121 and 122. This was the upper limit of coal conversion for all the Emery mine coal processed at Wilsonville. Comparison of the data for the runs follow:

	Run 121	Run 122
Conversion, % MAF Coal	83	80
Hydrogen Consumption, % MAF Coal	2.6	2.4
SRC Yield, % MAF Coal	64	52
GAs Make, % MAF Coal	10	10
Sulfur in SRC, wt. %	0.4	0.4

SRC product was produced with 0.16 percent or less ash content using filter screens of 80 x 80 mesh and Celite precoat. This was the first time that specification product was produced with the Funda Filter when using a precoat material that did not contain asbestos.

After the last run with Emery coal was completed in June, the plant was shutdown for inspection, cleaning, and maintenance. The dissolver volume was changed from 100 to 50 percent, in preparation for processing Indiana V bituminous coal from the Old Ben No. 1 mine.

Run 124 with Indiana V coal began in June. Conditions selected were: 1700 psig, 50 percent dissolver volume, and coal concentration of 38.5 percent. Coal conversions with Indiana V coal were between 92 and 94 percent.

The pilot plant operated 469 hours and was on-stream 65 percent of the time during June. The distillation sections operated an additional sixty hours to condition process solvent. Tests with the Sharples centrifuge were made in both May and June.

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IV. DONOR SOLVENT LIQUEFACTION PROCESS

EXXON RESEARCH AND ENGINEERING
COMPANY

BAYTOWN, TEXAS

Contract No.: No. E(49-18)-2353

Contract Funding: \$12,740,360

INTRODUCTION

Under the terms of this contract, details of prior development work on the Exxon Donor Solvent (EDS) process conducted between 1966 and January 1976 are confidential. The program schedule for this project is shown in Figure IV-1. The objectives of this contract are to:

- Optimize the processing sequence and operating conditions for the EDS Process.
- Further develop process correlations relating yields and product qualities to operating conditions and feed coals.
- Identify and implement leads to improve process economics.
- Provide process engineering and cost evaluations.
- Develop engineering technology.

PROCESS DESCRIPTION

The chief features of the EDS Process are illustrated in the process schematic, Figure IV-2. Crushed coal is liquified in a non-catalytic tubular reactor in the presence of molecular hydrogen and the hydrogen-donor solvent. The liquifaction reactor operates at 800-880°F and 1500-2000 pounds per square inch.

The hydrogen-donor solvent is a 400°-850°F boiling range material. The solvent is a catalytically hydrogen-

ated recycle stream fractionated from the middle boiling range of the liquid product. After hydrogenation, the solvent is mixed with fresh coal feed and pumped through a preheat furnace into the liquefaction reactor. Slurry leaving the liquefaction reactor is separated by distillation into gas, naphtha, distillates, and a vacuum bottoms slurry. The vacuum bottom slurry is coked to produce additional liquids.

The plant is "balanced" in that it is self-sufficient in both process fuel and hydrogen requirements. Process fuel and hydrogen are produced by gasifying the coke and by reforming C₁-C₂ gases from the liquefaction process.

The process is simple, and critical processing steps are adaptations of Exxon's base in petroleum refinery technology. Distinguishing features are the decoupled configuration of the liquefaction and catalytic hydrogenation sections and the use of vacuum distillation for solids/liquid separation. The catalyst does not contact coal minerals or high-boiling liquids, thereby leading to longer catalyst life at high activity. Use of hydrogenated rather than unhydrogenated recycle solvent produces a very significant improvement in process operability, particularly in downstream processing vessels. Also, hydrogenated solvent produces higher distillate product yields than unhydrogenated solvent. The use of mechanical separation devices for solids/liquids separation is avoided.

The process gives high yields of low-sulfur liquids from bituminous or subbituminous coals or lignites. For Illinois bituminous coal, the liquid yield is 2.6 barrels of C₄+ liquid per ton of dry coal. Ammonia and elemental

PROJECT	FY 76	TR. Q.R.	FY 77	FY 78	FY 79	FY 80	FY 81	FY 82
PDU & PILOT PLANT PDU	△							▽
			○					
			△		▽			
				△		▽		
						△		▽

LEGEND : △ BEGIN MILESTONE
▽ COMPLETE MILESTONE

Figure IV-1. DONOR SOLVENT PROJECT SCHEDULE

Figure IV-2. DONOR SOLVENT LIQUEFACTION PROCESS

sulfur are the only by-products of significance. By varying liquefaction conditions or adjusting solvent properties, product distribution may be varied over a wide range.

studies were conducted at the Baytown, Texas, Research and Development Division at Linden, New Jersey, and the Exxon Research and Development Laboratories in Baton Rouge, Louisiana. The Engineering Research and Development studies were performed at the Synthetic Fuels Engineering Department and Exxon Engineering Technology Department at Florham Park, New Jersey.

HISTORY OF THE PROJECT

Phase I of EDS Process Development (1966-1973)

Phase I of Exxon's research, the Predevelopment Phase, spanned the period 1966 through 1973, and concentrated on identifying the basic process flow scheme for coal liquefaction. This research was very broad in scope and was conducted in experimental equipment ranging in complexity from 100 cubic centimeter batch units to a continuous 1/2-ton-per-day integrated pilot plant.

The following reactor configurations were evaluated: stirred tank, tubular with external and internal recirculation, ebullating bed, and tubular plug flow. Both hydrogenated and unhydrogenated recycle solvents were evaluated for both bituminous and subbituminous coals. Several solids/liquids separation techniques were studied, such as distillation, centrifugation, filtration and hydroclones. Wide ranges of temperatures, pressures and residence times were evaluated in both the liquefaction and solvent hydrotreating reactors. Based on these results, a hydrogenated recycle solvent process was selected for further development, and vacuum distillation was identified as the most promising product-separation technique. A noncatalytic, tubular plug-flow liquefaction reactor was selected. These choices circumvented the need for ash-tolerant catalysts and mechanical separation devices.

Engineering studies closely paralleled the laboratory effort during Phase I. These engineering studies concentrated on the following areas.

- Evaluation of laboratory data.
- Development of conceptual commercial plant designs.
- Studies of processing alternatives to define incentives and provide direction to the laboratory effort.
- Development of cost parameters.

Phase II of EDS Process Development (1974-1975)

Phase II of Exxon's research, the Planning and Design Phase, was conducted during 1974-1975. The major efforts during this phase were concentrated in the following three areas.

- The basic design of the 250-ton-per-day Exxon Coal Liquefaction Pilot Plant (ECLP).
- The construction and initial operation of the one ton-per-day Coal Liquefaction Pilot Plant (CLPP).

- The supporting laboratory and engineering R&D program.

The laboratory and engineering R&D program more closely defined operating conditions for the process flow scheme selected in Phase I. This program concentrated on process variable studies of both bituminous and subbituminous coal. Wide ranges of temperature, pressure, residence time and solvent/coal ratio were studied. In addition, detailed investigations were conducted of solvent/hydrogenation in fixed-bed catalytic reactors.

PROGRESS DURING APRIL-JUNE 1977

Recycle Coal Liquefaction Unit Operations

The process variable study with Wyoming subbituminous coal was completed in the 50 pound-per-day Recycle Coal Liquefaction Unit (RCLU-I) in mid-April. The study was concluded with a run using low quality solvent and two runs at typical RCLU-I conditions.

In May, Phase III A lignite operations were concluded. Earlier yield data was obtained with North Dakota lignite at standard RCLU-I conditions. Process variables investigated in May were temperature and residence time.

During June, operations began with Wyodak coal. The purpose of the study was to determine the effect of heavier solvent on operability and yields. The effect of recycling coker liquids derived from Illinois No. 6 coal to liquefaction was also investigated. The objective of the study was to determine if heavy coker liquids could be upgraded to <1000° F boiling point liquids in liquefaction.

Coal Liquefaction Pilot Plant Operations

During the first part of April, the operational phase of the engineering studies with Illinois No. 6 coal was completed on the one ton-per-day Coal Liquefaction Pilot Plant (CLPP). In conjunction with the engineering studies, five yield periods were made at both standard residence times and short residence times. Preliminary yields compared favorably with previous CLPP and RCLU results.

After completion of the engineering studies with Illinois No. 6 coal, operations began with untreated Wyoming coal. The purpose of the run was to begin assessing the operability of Wyoming coal in larger scale

equipment than the RCLU. In the initial run, the unit was operated for 192 hours before the run was terminated due to a plug in a liquefaction separator.

A 16 day, continuous, integrated run was completed on the CLPP with the Wyoming coal during May. A liquefaction reactor solids withdrawal system was operated during the entire run. In order to evaluate the effect of the system, the unit was brought down under controlled conditions at the end of 16 days. Inspection of the reactor revealed that no significant particle growth occurred during the run.

In June, the operability of the CLPP with Wyoming coal was at lower temperatures and longer residence time was investigated.

Supporting Studies

Validation of the two-train solvent hydrotreating unit was completed and the unit used for process variable studies. Operability studies continued on Wyoming coal. As the Wyoming coal shows a greater tendency to form carbonaceous deposits than does Illinois coal, potential solutions to this operability problem were screened.

Modifications to the Large Stirred Coking Unit (LSCU) continued during the quarter resulting in improved and more reliable operation.

The preparation of the existing gasifier for future EDS bottoms integrated coking and gasification studies was completed in the 2 barrel-per-day Coking/Gasification Pilot Plant (IKG). The unit was pressure tested in June.

V. COMMERCIAL SCALE EBULLATED-BED HYDROPROCESSING OF SOLVENT REFINED COAL EXTRACT

CITIES SERVICE RESEARCH AND
DEVELOPMENT COMPANY
TULSA, OKLAHOMA

Contract No.: E(49-18)-2038

Contract Funding: \$591,122

INTRODUCTION

Cities Service, under ERDA sponsorship, is determining the operating conditions and modifications necessary to utilize an existing LC-Fining unit (ebullated-bed hydroprocessing unit) to upgrade a production quantity of Solvent Refined Coal (SRC) extract.

Present operations are being conducted at the New Brunswick process development unit site of C. E. Lummus, subcontractor for operations for Cities Service. After completion of the present tasks, experimentation will begin at Cities Service Lake Charles, Louisiana plant. The specific objectives of this contract are to:

- Determine the ebullated-bed conditions required to hydrotreat SRC extract at severity levels ranging from desulfurization to extensive hydrocracking.
- Upgrade a production quantity of SRC extract from the Ft. Lewis pilot plant using commercial-scale ebullated-bed hydroprocessing equipment.
- Demonstrate that the SRC process and ebullated-bed hydroprocessing can be combined to provide a valuable liquid fuel or refinery feedstock from coal.

PROCESS DESCRIPTION

Solvent Refined Coal (SRC) extract is a hydrocarbon mixture containing significant quantities of oxygen, nitrogen, sulfur, and ash. SRC extract composition and

properties are similar to heavy sour petroleum crude and process-derived residua. These are problem stocks which must be upgraded by treatment with hydrogen before they can be further refined to liquid finished products. Ebullated-bed hydrotreating can upgrade these stocks and maintain continuous operation for extended periods of time as well. A flow diagram of the process is shown in Figure V-1.

The SRC extract, together with hydrogen, is fed upward through the reactor as a liquid/gas mixture at a velocity such that the catalyst is in continuous motion. Part of the reactor effluent is recycled back through the reactors to maintain the necessary liquid velocity through the reactor system (the ebullated-bed). Because of the movement of catalyst particles in the liquid/gas medium, deposition of tar and coke is minimized and fine solids

Figure V-1. EBULLATED-BED HYDROPROCESSING PROCESS SCHEMATIC

entrained in the feed will not lead to reactor plugging. Reactor effluent is cooled by exchange, and a vapor-liquid separation is made. After scrubbing, the hydrogen is recycled and liquid product is either stored or further fractionated.

Bench-scale pilot plant studies will be performed for the purpose of determining operational conditions for the commercial unit. Engineering studies will define the degree of effort and resources required to adapt a commercially available ebullated-bed unit to process a large quantity of SRC extract.

HISTORY OF THE PROJECT

Active work on the commercial scale ebullated-bed hydroprocessing of Solvent Refined Coal (SRC) extract began on June 1, 1976 with bench-scale pilot plant operations. Experimental runs designed to determine the operating conditions necessary to process Solvent Refined Coal (SRC) in the commercial LC-Fining unit were completed during the year. The first 30-day catalyst aging run LCF-24, processing a blend of 67.0 weight percent SRC and 33.0 weight percent Koppers heavy residue creosote oil, was also completed. Conversion of the 850°F-plus material averaged 45.9 volume percent.

PROGRESS DURING APRIL-JUNE 1977

Detailed summaries of the liquid products from the three thirty-day catalyst aging runs (LCF-24, LCF-25, LCF-26) were completed. Results showed that the operating conditions for Run LCF-26 provided a liquid product with the highest conversion of 850°F+ feed (approximately 62 volume percent 850°F+ to 850°F-) and enhanced nitrogen and sulfur removal (57 and 91 percent of feed respectively). The ash disappearance of approximately 52 weight percent based on ash in the feed for Run LCF-26 indicated that all the ash was not passing through the LC-Finer, but was probably retained in an undetermined manner by the catalyst.

The hydrogenation of solvent in the LC-Fining unit was completed during the quarter. The hydrogenated solvent was intended for use in the catalyst screening study. The analytical results indicate that the heavy oil hydrogen content of the Koppers Heavy Residue Creosote Oil (KC-Oil) was 7.9 weight percent and the nitrogen and sulfur removal was approximately 90 weight percent. This compares with the KC-Oil change of 5.81 weight percent hydrogen.

Due to operational difficulties, insufficient solvent was produced in the LC-Fining unit to complete the catalyst screening study. Therefore, hydrogenated solvent from the Lummus HDS pilot unit was substituted for the hydrogenated solvent from the LC-Fining unit.

VI. CRESAP TEST FACILITY

FLUOR ENGINEERS AND CONSTRUCTORS,
INC.

LOS ANGELES, CALIFORNIA

Project Site: Cresap, West Virginia

Contract No.: E(49-18)-1517

Contract Funding: \$45,699,780

ERDA: \$43,042,140

Industry: \$ 2,657,640

INTRODUCTION

Under contract to ERDA, Fluor Engineers and Constructors, Inc., is renovating and will operate an ERDA-owned facility in Cresap, West Virginia. Industrial financing is being provided by American Electric Power Corporation (\$1 million), Allegheny Power Corporation (\$1 million), and Fluor (\$657,640). This facility, inactive since 1970, contains much of the equipment required for coal liquefaction. Fluor is to (1) renovate the Cresap facility to test the various critical mechanical components required for coal liquefaction and (2) operate the renovated facility to demonstrate an integrated process for the production of low-sulfur liquid fuel. The project schedule is provided in Figure VI-1.

PROCESS DESCRIPTION

In this process, shown schematically in Figure VI-2, coal is converted to low-sulfur liquid fuel. The coal is first crushed to 100-mesh and then combined with aromatic solvent in a mixer. The coal-solvent slurry is then pressurized and preheated. The slurry passes to an

extractor where coal dissolution occurs. The product stream is then treated in a liquid-solid separation unit where the coal extract and solvent are removed, leaving a thickened slurry. This stream is sent to a carbonizer where the material is converted to char, oil, and gas. The oil is recycled to the liquid-solid separation unit and the char and gas are recovered.

One of the more persistent problems encountered in coal liquefaction processes is the separation of solids from the liquid product streams. As the Cresap Test Facility, several methods of separation will be tested. One of the most reliable methods for concentrating the solids is a system of hydroclones which was successfully used during prior pilot plant operations. Other processes to be evaluated will be filtration and chemical precipitation.

The coal extract and solvent leaving the liquid-solid separation unit are heated and fed into a flash still where the heavy components of the coal extracted are separated from the light liquids. The light liquids are then fractionated into light and middle distillates. The middle distillate may either be recycled as a component of coal solvent or recovered as product fuel oil. The heavier components of the coal extract are heated in a fired preheater, hydrocracked, and fractionated into three distillate fractions. The middle distillate is used as coal solvent and the heavy distillate is recovered as fuel oil product.

LEGEND:
 BEGIN MILESTONE
 COMPLETE MILESTONE
 DECISION MILESTONE
 EVENT
 5 ORDER LONG-LEAD ITEMS

Figure VI-1. CRESAP TEST FACILITY PROGRAM SCHEDULE

Figure VI-2. LIQUID FUEL FROM COAL PROCESS SCHEMATIC

HISTORY OF THE PROJECT

Reactivation of the Cresap facility to develop an integrated process test center for producing low-sulfur liquid fuel from coal was initiated in May 1974. Since then, the facility has been upgraded to meet environmental requirements and current design standards of piping, electrical, and mechanical equipment codes. A task force has been established to organize, coordinate, and direct the testing program.

Renovation of the pilot plant involved the refurbishing of existing equipment and the installation of new equipment to replace outmoded or worn out equipment. Inquiry packages were developed for most of the new equipment, and flow diagrams and specifications were prepared for the refurbishing of some of the existing equipment. The Liquefied Coal Development Corporation, a wholly-owned subsidiary of Fluor, was formed during 1975 to operate the pilot plant.

PROGRESS DURING APRIL-JUNE 1977

Startup operations continued during the quarter at the Cresap Test Facility. Included were operating portions of the plant on water or fuel oil in order to simulate normal plant operations for mechanical checkout. This has proved beneficial in extraction, vacuum distillation, atmospheric distillation, and carbonization sections in uncovering problems prior to full startup. The remaining process units are undergoing prestartup electrical and mechanical activities.

In order to finish a number of remaining construction activities all operations efforts were halted in mid-May and the plant was completely shutdown. The construction work included completion of final construction work, critical items required in preparation for integrated front-end startup, and a review of the status of the None Destructive Evaluation program.

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VII. SYNTHOIL PROCESS

FOSTER WHEELER ENERGY CORPORATION
LIVINGSTON, NEW JERSEY

Contract Funding: \$24,425,776

SANDIA LABORATORIES
ALBUQUERQUE, NEW MEXICO

Contract Funding: \$1,636,000

ARGONNE NATIONAL LABORATORY
ARGONNE, ILLINOIS

Contract Funding: \$1,574,000

BATTELLE MEMORIAL INSTITUTE,
COLUMBUS LABORATORIES
COLUMBUS, OHIO

Contract Funding: \$156,000

Plant Site: Bruceton, Pennsylvania

INTRODUCTION

Development of the Synthoil process, initiated by the U.S. Bureau of Mines, is currently being managed by ERDA through the Pittsburgh Energy Research Center (PERC) in Pennsylvania. The objective of this project is to determine the technical and economic feasibility of the process for scale-up to commercial use. Foster Wheeler Energy Corporation is responsible for designing and managing the construction of a ten-ton-per-day process development unit to test the Synthoil process. PERC is conducting support research for the design of the unit. In addition, PERC is monitoring laboratory research on various aspects of the Synthoil process being conducted by Sandia Laboratories, and Argonne National Laboratory. Research being conducted at Exxon Research and Engineering and Battelle Memorial Institute is being monitored by the Morgantown Energy Research Center

(MERC) in West Virginia. The schedule for the Synthoil project is shown in Figure VII-1.

PROCESS DESCRIPTION

The Synthoil process is a hydrodesulfurization process that converts high-sulfur coal to a low-sulfur, low-ash synthetic fuel oil. A schematic of the process is shown in Figure VII-2. The coal is first crushed to 70 percent through 200-mesh and dried to approximately two weight percent moisture. It is then mixed with process product oil to form a slurry of 35-40 percent coal. The resulting slurry is then combined with hydrogen and fed into a fired preheater. From the preheater, the slurry enters a fixed-bed catalytic reactor packed with catalyst pellets of cobalt molybdate on a silica-alumina base. The

Figure VII-1. SYNTHOIL PROGRAM SCHEDULE

Figure VII-2. SYNTHOIL PROCESS SCHEMATIC

product mixture is then cooled and the liquid and unreacted solids are separated from the gases.

Hydrogen propels the slurry so violently through the fixed-bed catalytic reactor that plugging of the bed by the mineral matter in the coal is prevented as the coal becomes liquified.

The liquids and unreacted solids leave the bottom of the separator and pass into a centrifuge. The solids are separated and may be fed into a pyrolyzer. Part of the liquid leaving the centrifuge is recycled to the mixer and the remainder is drawn off as fuel oil product. The solids are further pyrolyzed to yield additional quantities of product oil and an ash residue. This residue, which

contains some carbonaceous material, will be sent to a gasifier and shift converter to produce additional hydrogen in any commercial operation.

The separator off gases are sent through a gas purification system where they are separated into five product streams: hydrogen sulfide, water, ammonia, hydrocarbon gases, and hydrogen. The hydrogen sulfide is sent to a sulfur recovery system yielding elemental sulfur as the product. The hydrocarbon gases are fed to the gasifier and shift converter. The hydrogen stream which is mixed with the hydrogen produced in the gasifier is fed into the slurry stream entering the preheater.

HISTORY OF THE PROJECT

Work on this process began in 1969 at PERC. The initial work on the Synthoil process used a reactor with an internal diameter of 5/16-inch in a bench-scale plant that processed five pounds of slurry per hour. Experimental work was conducted on various coals, such as Pittsburgh seam, Indiana No. 5, Middle Kittenning Ohio No. 6, and Kentucky strip coal. All of these types of coal were satisfactorily converted to low-sulfur fuel oil with no significant attrition of the catalyst. Experiments were conducted to study the effects of hydrogen flow rate, the percentage of coal in the feed slurry, the recycle rate of the product oil, and the effect of hydrogen sulfide in the recycle gas.

To demonstrate the broad applicability of the process, a 1/2-ton-per-day slurry unit was constructed that used a reactor of 1.1-inch ID made of two interconnected stainless steel pipes that were each 14.5 feet long. The operations were carried out on several types of coal at reactor pressures of 140 atm and 280 atm and 840°F. High yields of low-sulfur and low-ash fuel oil were obtained. At the lower reactor pressure the calorific value, product oil yield, and hydrogen consumption were also lower. However, the sulfur and ash content of the product oil were higher than that of the oil produced at 280 atm. Economic analyses will decide the appropriate pressure for this process, considering the capital and

operating costs for high-pressure and low-pressure operations, and the environmentally acceptable sulfur and ash contents of the product oil.

The overall design package for the process development unit was completed during 1975. In addition, long lead equipment was placed on order. Actual field construction of the facility began during the fourth quarter of 1975.

PROGRESS DURING APRIL-JUNE 1977

The overall total construction effort for the 10 ton-per-day process development unit was 65 percent complete at the end of the quarter.

In the laboratory studies aimed at optimizing preheater design, one noncatalytic run (FB-60) was conducted during the quarter with a catalytic run in progress at the end of June.

Mass balances were calculated for each of 15 runs on the continuous centrifuge. The average balance was 99.4 percent for the 15 runs. Additional density and chemical analyses permitted an estimate of the density of the organic benzene insolubles (OBI), the ash, and the solids-free Synthoil. The results, in conjunction with new viscosity measurements, yielded curves of OBI and ash particle size distribution. Small changes in operating conditions affecting the viscosity of the gross liquid product could produce significant improvements in ash separation, but major changes would be required for equivalent improvement in OBI removal.

Reactor plugging continued to be the main obstacle to successful operation of the miniature Synthoil catalyst life-testing unit. Plugs occurred in three of the four tests attempted during the quarter.

Construction of the Synthoil II unit continued. The hot pressure stall supporting structures were being prepared for the installation of the preheater and reactor furnaces and the high-pressure vessels.

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VIII. CLEAN COKE PROCESS

USS ENGINEERS AND CONSULTANTS, INC.
UNITED STATES STEEL CORPORATION
PITTSBURGH, PENNSYLVANIA

Plant Site: Pittsburgh, Pennsylvania
Contract No.: E(49-18)-1220
Contract Funding: \$11,792,329
ERDA: \$8,337,176
Industry: \$3,455,153

INTRODUCTION

The Clean Coke process is being developed by USS Engineers and Consultants, Inc., a subsidiary of United States Steel Corporation, under the sponsorship of ERDA. The contract was initiated in 1972 under the auspices of the Office of Coal Research (OCR, now a part of ERDA). The objective of the project is to develop a process design for a pilot plant that is capable of converting low-grade, high-sulfur coal to low-sulfur, low-ash metallurgical coke, chemical feedstocks, and liquid and gaseous fuels. The pilot plant design will be based on the results of laboratory tests and data obtained from process development units. The schedule for developing the process is shown in Figure VIII-1.

PROCESS DESCRIPTION

The Clean Coke process is shown schematically in Figure VIII-2. This process combines coal carbonization and hydrogenation in an environmentally acceptable manner to produce solid, liquid, and gaseous fuel products. No external hydrogen source is required and only minimal amounts of external energy will be consumed.

Figure VIII-1. CLEAN COKE PROCESS SCHEDULE

The feed coal is first pulverized and classified into two portions. One portion is sent to the carbonizer. The coal is pyrolyzed in a fluidized bed yielding char, liquid, and a hydrogen-rich gas which is subsequently cleaned and recycled to fluidize the bed. The hydrogen-rich atmosphere of the bed provides a hydrodesulfurization environment that reduces the high-sulfur coal to low-sulfur char. This char is then pelletized using a process-derived heavy oil binder and the resulting pellets are cured and calcined in a kiln. The products are low-sulfur metallurgical coke and a hydrogen-rich gas.

The other portion of the coal is fed to the hydrogenation unit where it is slurried with a medium-weight oil recycled from the liquid processing section. This slurry is

completed by the end of 1976 was 3,667 hours. The hydrogenation PDU was operated during the last quarter of 1976, with an extended run planned for the beginning of 1977.

PROGRESS DURING APRIL-JUNE 1977

Summary

Carbonization process development unit (PDU) tests were conducted during the quarter with West Kentucky coal. The binder-preparation PDU continued to process liquid blowdown from the carbonization PDU. The latest tests using raw coal were completed in the Hydrogenation PDU, and studies of two-stage vapor stripping from various liquefaction processes began. Preparation and testing of binders from SRC product continued.

Carbonization Process Development Unit

Two carbonization process development unit (PDU) tests were completed in April. The first was a shakedown run after an extended maintenance period. The second run was made to confirm the material balance results in a previous 1400°F run.

In May, work in the carbonization area included successful processing of West Kentucky coal through both stages of carbonization, 820 and 1400°F. In the first stage, 1294 pounds of coal containing 2.70 percent sulfur were processed to 1085 pounds of semichar containing 2.05 percent sulfur. In the 1400°F run, 1071 pounds of semichar were processed to 831 pounds of char containing 0.99 percent sulfur.

Runs in June continued with West Kentucky coal under more severe carbonization conditions. In the first run, the coal was processed at 840°F and 165 psia, with a residence time of 80 minutes. The second run was at 1450°F and 165 psia and a residence time of 150 minutes.

Binder-Preparation Process Development Unit

The flash vaporizer of the binder-preparation process development unit (PDU) was used to process filtered vapor-stripper bottoms product from the hydrogenation PDU. The tar residue was prepared as binder for coke-making in April. In addition, three runs were made to process liquid blowdown from the carbonization PDU.

The batch distillation column was operated to develop an optimum procedure for decreasing the naphthalene content of the flash-vaporizer overhead product. Up to 85 percent of the naphthalene was removed.

After maintenance work on the flash vaporizer in May, the unit was again operated to process liquid blowdown from the carbonization PDU. The clear wash oil recovered amounted to 1870 pounds. This wash oil was then processed in the batch distillation column where 90 percent of the naphthalene was removed. This procedure continued in June.

Hydrogenation Process Development Unit

Two coal-conversion runs were made in the hydrogenation PDU in April to complete the testing with raw coal and to determine whether slurry oil recovered from conversion of raw coal can be recycled without further hydrogenation. Coal conversion efficiency was 83.7 percent of moisture - and ash-free coal. Slurry oil recovered from vapor-stripper overhead showed an increase in hydrogen-content of about 0.2 percent. It was apparent that when raw coal is used as feed, continued recycle of the slurry oil derived from the process is possible without the expensive, time consuming rehydrogenation step required when processing beneficiated coal. The catalytic action of the ash minerals is significant in improving the coal-liquefaction process.

As a result of a meeting with ERDA officials in April, work on the hydrogenation PDU was re-directed at studies of two-stage vapor stripping for materials from other liquefaction processes such as SRC, Synthoil, H-Coal, and Exxon. Cokemaking studies will be conducted by using binders prepared from these products. Evaluation of the binders will be completed in cokemaking tests with Clean Coke process char.

Coke Preparation

Testing of binders prepared from SRC product continued throughout the quarter. Two pelletization runs were made in April with high-temperature carbonization char and a binder consisting of 57.5 percent SRC product fluxed with 42.5 percent SRC process-derived solvent oil. The pellets produced from this test and a test in March were calcined in May. The coke pellets exhibited satisfactory strength and hardness.

In June, an attempt to produce coke with greater stability was made by using char pulverized to minus 100 mesh and a 55/45 SRC binder.

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IX. LIQUEFACTION OF COAL BY DIRECT HYDROGENATION

ROCKETDYNE DIVISION
ROCKWELL INTERNATIONAL CORPORATION
CANOGA, PARK, CALIFORNIA

Plant Site: Canoga Park, California
Contract No.: E(49-18)-2044
Contract Funding: \$994,375
(100% ERDA)

INTRODUCTION

Under contract to ERDA, the Rocketdyne Division of Rockwell International is conducting a research program to develop a technology to liquefy coal via direct hydrogenation. This technique would involve rapid mixing, reaction, and quenching of a gaseous hydrogen and pulverized coal mixture to form hydrocarbon liquids. These products would be suitable for conversion to fuel oil or chemical feedstocks.

The program is divided into two phases. Phase I involves the design and fabrication of cold-flow equipment and the operation of the equipment in a series of tests to evaluate methods of transport and injection of pulverized coal into the reactor.

Upon completion of Phase I, the results of the cold-flow tests were submitted to ERDA for a decision on proceeding with Phase II. Upon Phase II authorization, small-scale reactor testing with initiated to evaluate the effects of reactor configuration and operating conditions. Based on these results, a larger reactor, with diverse injection methods and a coal flow rate of approximately one ton per hour, will be built and tested. Test results and analyses will be evaluated to determine the feasibility of this process. The schedule for the development of this process is shown in Figure IX-1.

Figure IX-1. PROGRAM SCHEDULE FOR LIQUEFACTION OF COAL BY DIRECT HYDROGENATION

In addition to the ERDA funded effort, Rockwell International is sponsoring a corollary effort to provide supporting facilities.

PROCESS DESCRIPTION

The basic concept of this process for the liquefaction of coal is that high liquid yields are favored by rapid mixing, reaction, and quenching of a mixture of pulverized coal and hot gaseous hydrogen. In this process, as shown in Figure IX-2, coal is fed into the coal feeder from a batch feeder, with the transport gas. The coal passes through the feeder, is partially fluidized with hydrogen at the conical exit, and then passes to the entrained-flow reactor where it is mixed rapidly with 1500°F hydrogen. The pulverized coal and hydrogen react for about 10 to 100 milliseconds. The reactor is expected to be operated over a pressure range of 35 to 100 atmospheres and a temperature of approximately 1800°F.

The reactor effluent is passed into a quench section which has a set of water spray nozzles. From there the effluent passes into a char receiver.

HISTORY OF THE PROJECT

Development of a process for the liquefaction of coal by direct hydrogenation was started in August 1975. Phase I testing was completed in 1976, and Phase II testing began. The first 1/4-ton-per-hour test was made at the end of May, and final testing on the unit was completed by the end of the year. Results of the tests allowed a confident transaction to the one-ton-per-hour system and indicated directions for testing at the larger scale.

Construction of the one-ton-per-hour test facility was completed in December 1976, and preparation for testing began.

Figure IX-2. SCHEMATIC OF LIQUEFACTION OF COAL BY DIRECT HYDROGENATION

PROGRESS DURING APRIL-JUNE 1977

Summary

The initial series of one-ton-per-hour reactor tests was completed in April. Material balances were calculated for carbon, hydrogen, and ash. Sulfur and nitrogen contents and heating values of the product char were correlated. The compositions and heating values of the liquid product recovered from the condenser were summarized.

Approval to resume testing in the one-ton-per-hour unit was received early in June. Three additional tests were completed by the end of the quarter.

Discussion of Activities

A final test of the first test series with the one-ton-per-hour reactor was completed at the beginning of the

quarter. The test was made with the 2.37-inch diameter reactor tube, at a pressure of 1000 psig, a reactor temperature of 1690°F, and a residence time of 69 milliseconds. During the test, 1013 pounds of coal were processed in 37 minutes. The results showed a consistent trend with reactor temperature and indicated a moderately weak dependence of liquid yield on reactor temperature. Somewhat greater overall conversions were obtained with longer residence times but with an attendant loss in liquid yield. In a concurrent effort, a dense-phase flow, char removal system was installed under company funding.

Approval to resume testing was received early in June. A packed bed adsorber was installed to recover entrained and vapor-phase liquid product from the gas stream. Three tests were performed late in June with the 2.37-inch diameter, 60-inch long reactor.

Carbon and ash balance calculations were completed for the first of these tests. Results from ash analysis indicated 100.1 to 103.0 percent ash recovery.

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X. FLASH PYROLYSIS COAL LIQUEFACTION PROCESS

OCCIDENTAL RESEARCH CORPORATION
LA VERNE, CALIFORNIA

Plant Site. La Verne, California
Contract No.: EX-76-C-01-2244
Contract Funding: \$3,780,000
(100% ERDA)

INTRODUCTION

The Occidental Research Corporation (ORC), a wholly-owned subsidiary of Occidental Petroleum Corporation, will evaluate the commercial potential of its flash pyrolysis coal liquefaction process under the sponsorship of ERDA. Figure X-1 provides the schedule for the project.

The main objectives of the program are to:

- Demonstrate that caking coals can be processed continuously in a specially designed single-stage pyrolysis reactor without oxidative pretreatment, and that this method will result in a significantly higher yield of liquids than other proposed pyrolysis processes.
- Conduct extended runs in the three-ton-per-day process development unit (PDU) in order to obtain steady state heat and material balances.
- Produce and recover large quantities of the primary tar, and to evaluate methods for upgrading this material to a clean fuel or synthetic crude oil.
- Continue development of specific areas of the pyrolysis and liquids collection systems to ensure a technologically sound basis for future scale-up.
- Obtain sufficient process and environmental data for detailed design of a larger plant and conduct an assessment of the potential commercial viability of the process.

Figure X-1. FLASH PYROLYSIS PROJECT SCHEDULE

PROCESS DESCRIPTION

The ORC process consists of rapidly pyrolyzing coal particles at a temperature of less than 1400°F in an entrained stream of hot coal char and gas, substantially free of oxidizing constituents. The process features short residence time of coal particles thereby increasing the throughput per unit of cross sectional reactor area, and producing a relatively high yield of liquid products. Figure X-2 provides a schematic flow diagram of the process.

After the coal is extensively milled and screened, it is transported to the pyrolysis reactor by heated nitrogen at rates of up to 300 pounds per hour. In the reactor, the

Catalysts for screening and for use in hydrotreating operations were prepared. Hydrotreating of subbituminous coal and catalyst screening tests continued.

Process Development Unit

The third baseline run was successfully completed in April. The objectives of this run were to maintain steady state operation for an extended period, and to obtain material balance data operating on subbituminous non-caking coal. After a smooth startup, 25,000 pounds of coal were processed in 125 hours of uninterrupted steady state operation at an average rate of 200 pounds per hour. Four pounds of full range tar were collected by slip stream sampling and separated into four fractions by distillation.

The tar yield results of all three baseline runs are interpreted to be roughly equivalent, but uniformly lower than the bench-scale results. Factors which may have contributed to low yields included oxidation of the feed coal, leakage of combustion air into the pyrolysis zone, inadequate sampling procedures, and thermal cracking. A series of tests were structured for the PDU and bench scale reactor to systematically evaluate these factors.

In May, six PDU tests were made pyrolyzing bituminous coal. Subbituminous coal-derived char was used as the heat carrier. Approximately 1100 pounds of bituminous coal were pyrolyzed under varying conditions at rates up to 195 pounds per hour.

Bench-Scale Activities

The objective of hydrotreating work was to prepare bulk quantities of coked catalysts for screening and for use in hydrotreating operations. Five catalysts of about 120 grams were prepared by depositing coke at 750°F, and 60 minutes reaction time under a pressure of 2000 psi. Catalyst screening tests continued throughout the quarter.

A forty hour hydrotreater run was made in May processing subbituminous tar produced in the PDU. The run was conducted at 2500 psi and 750°F. Product inspections showed a 27° API increase in specific gravity. Sulfur, asphaltenes, and pre-asphaltenes removal were over 95 percent. Nitrogen and oxygen removal were approximately 75 percent. Hydrogen consumption was about 300 SCF/bbl.

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XI. FILTRATION PROCESS AND EQUIPMENT STUDIES

JOHNS-MANVILLE SALES CORPORATION
DENVER, COLORADO

Project Site: Denver, Colorado
Contract No.: E(49-18)-2007
Contract Funding: \$190,000
(100% ERDA)

INTRODUCTION

Under contract to ERDA, Johns-Manville Sales Corporation is conducting research to improve solid-liquid separation, a critical step in all coal liquefaction processes. Rotary drum pressurized precoat filtration is being studied with the objective of the program to achieve major improvements in the filtration process and in the filtration equipment used in the coal liquefaction process. The goals are to double the filtration rate, to reduce filter aid usage, clarify filtrate, and to demonstrate the results on the pilot-plant scale.

PROGRAM DESCRIPTION

The filtration research program is divided into two phases. The objectives of Phase I are to (1) develop basic knowledge of the coal oil filtration process and its dependence on liquefaction process parameters and (2) apply the knowledge gained to improve the performance of filtration operations in existing liquefaction plants. Phase II involves the design and construction of a rotary drum pressurized precoat filter capable of processing the product flow from an existing liquefaction pilot plant. Phase I is being funded entirely by ERDA, and Phase II is being supported by the Electric Power Research Institute (EPRI) in cooperation with the ERDA effort. One of the criteria established for a viable system is the capability of

filtering in excess of 20 gallons of solid-liquid mixture per square foot per hour. Data obtained from operation of the pilot scale filtration unit will be used to design filters for commercial plants.

Phase I of the project is being implemented through laboratory bench-scale research. The initial step involves the design and construction of a laboratory test facility that will simulate the operation of a rotary drum pressurized precoat filter capable of operating at 750°F and 27 atmospheres. A series of tests will then be conducted to study pressure precoat filtration operating parameters at the temperatures and pressures expected in normal pilot plant operations. In these tests, two liquefaction filter feed process slurries are to be used: solvent-refined coal (SRC) from both the Wilsonville and Tacoma pilot plants and Synthoil boiler fuel supplied by the Pittsburgh Energy Research Center.

Upon completion of these tests, experiments will be performed on hot, fresh samples of Tacoma Filter Feed in order to establish the relationship between test results obtained on fresh feed and those on product that have been cooled. Due to the chemical and physical changes which may take place during aging and irreversible alternations which occur due to various thermal histories, it is vital that product behavior during pilot testing be accurately correlated to the anticipated behavior during commercial operations.

In cooperation with P&M, Johns-Manville will apply its filtration knowledge to improve the filtration rate and

reduce the filter aid usage on existing Goslin-Birmingham filters. Similar filter process optimization studies will then be conducted at other ERDA-sponsored coal liquefaction plants. Johns-Manville is also responsible for assisting in the selection of a suitable precoat filter for the Cresap Test Facility in Cresap, West Virginia. This investigation will include (1) the feasibility of modifying the existing filtration equipment to accommodate the experimental work and (2) assistance in the start-up and establishment of operating conditions for the filter system selected.

HISTORY OF THE PROJECT

The first phase of this program was initiated in May 1975. During that year, emphasis was placed on designing and constructing a laboratory test facility. The experimental program for the filtration studies was also planned. The statistical method of fractional factorial design was selected as the experimental design with the principal independent variables of temperature, differential pressure, filtration time per cut, depth of cut, degree of submergence, immersion time and consumption rate of filter aid selected for investigation. The design involves treatment of each of the five variables at two levels in eight experiments with coal product and type of precoat kept constant.

The first filtration tests were run late in 1975 using water and organic media to establish precoating and cake cutting procedures. Work on the design and construction of the laboratory test facility continued during the first quarter of 1976. Technique and equipment refinements were developed from the experimental work. During 1976, filtration tests were conducted using samples of Tacoma Filter Feed (TFF), Synthoil product, Wilsonville Filter Feedstock, and hot, fresh TFF.

PROGRESS DURING APRIL-JUNE 1977

During the quarter, a total of 57 filtration runs were made. In addition to 52 runs with unfiltered solvent refined-coal (SRC) from Fort Lewis, five samples of filter feed from Wilsonville were also tested for the second time since December 1975.

Twenty-one filtration runs were performed on the rotary pressure precoat filter test leaf during April on

four different samples of unfiltered solvent refined coal (SRC) from Fort Lewis.

Filtration rates in excess of 700 pounds per hour per square foot were obtained at 100 psid. It appeared that a marginal increase in filtration rate of about 5 to 10 percent was obtained by increasing the differential pressure from 70 psid to 100 psid.

The relationship between filtration rate and drum speed at different knife blade advance rates was also investigated. Filtration rates at one mil per cycle blade advance were about 20 percent lower than at two mil per cycle rates. Filter aid usage at one mil per cycle was 60 percent of that obtained at two mil per cycle.

A series of tests investigated the use of a non-asbestos pre-precoat. A 1/4-inch thick pre-precoat of CELITE 550 was applied to the test leaf prior to precoating with CELITE AF5. Filtration rates with the CELITE 550 pre-precoat were about 12 percent higher than for the asbestos filled pre-precoat. The filtrate ash analysis averaged 0.07 percent ash with the CELITE 550 pre-precoat compared to the 0.05 percent when the FIBRA-FLO 76 was used.

Experimental work in May also included the evaluation of the effect upon filtration rate of blowdown time.

In June, 19 filtration runs were performed on unfiltered SRC filter feed from Fort Lewis, and five filtration runs on unfiltered SRC filter feed from Wilsonville. The filtration rate characteristics at 1/2 mil per cycle knife blade advance of TFF-216 filter feed from Fort Lewis at both 70 and 100 psid were explored. Results indicated that further studies at very small blade advance with inherently low filter aid usage rates would be worthwhile.

A series of runs were performed to evaluate CELITE AF 13, a relatively coarse filter aid, as a non-asbestos pre-precoat material. Results indicated essentially no difference in filtration rate or filtrate ash content when compared with previous tests under similar conditions using the asbestos filled FIBRA-FLO 76 as the pre-precoat material.

The first tests on unfiltered filter feed from Wilsonville were first conducted in the latter part of 1976, using a fractional factorial design. Initial results indicated filtration rates comparable with those obtained upon filtration of filter feed from Fort Lewis during tests also conducted at that time. More recent tests indicated that the filtration rate of Wilsonville filter feedstock is independent of blade advance from 1/2 to 4 mils. The Wilsonville material was also found to be easier to filter than the Fort Lewis SRC filter feedstock.

XII. CHEMICALS FROM COAL

THE DOW CHEMICAL COMPANY
MIDLAND, MICHIGAN

Project Site: Midland, Michigan
Contract No.: E(49-18)-1534
Contract Funding: \$342,896
(100% ERDA)

INTRODUCTION

Historically, coal was used as a chemical feedstock, but petroleum and natural gas were substituted as cheaper feedstocks when they became available. However, because of the recent rising costs and decreasing availability of petroleum and natural gas, The Dow Chemical Company under sponsorship of ERDA, is studying the possibility of using coal-derived liquids as chemical feedstock. The current contract was initiated in 1974. Dow is testing liquids from four coal liquefaction processes (COED, Synthoil, H-Coal, and SRC) in standard petrochemical processing experiments and is evaluating these results to select one process for further study. The selection will be based on process design, quality of chemicals produced, and overall plant economics. For the process selected, further development will include material balances and utility requirements for each major process step, a preliminary process flowsheet, and estimates of capital and operating costs for a commercial plant.

PROGRAM DESCRIPTION

The liquids produced by the four processes are tested in standard petrochemical processing operations to determine the advantages and disadvantages of using coal-derived liquids for petrochemical feedstocks. The experimental work is being conducted in small units capable of

operating unattended at pressures up to 204 atm., temperatures up to 1300°F, and feed rates from 25 to 400 milliliters per hour.

The processing sequence used for all of the samples is:

- (1) Distillation into nominally three cuts- straight-run naphtha (350°F or less), middle distillate (350°F to 650°F), and heavy gas oil (650°F to 950°F).
- (2) Hydrocracking of the middle-distillate and heavy gas oil to reduce molecular weight.
- (3) Hydrotreating of the straight-run naphtha and hydrocracked naphtha to remove heteroatoms.
- (4) Reforming of the hydrotreated naphthas to maximize aromatics.

Steam coil cracking of some of the fractions is also performed to evaluate potential ethylene yields.

Characterization studies on all of the samples in the hydroprocessing experiments are performed using the following procedures:

- Component analysis of the hydrocarbons.
- Atmospheric, vacuum, and American Society for Testing Materials (ASTM) distillations.
- Elemental analysis (carbon, hydrogen, oxygen, nitrogen, sulfur).
- Trace metal analysis.
- Liquid chromatography for class separations (aliphatics, monoaromatics, diaromatics).

HISTORY OF THE PROJECT

The laboratory investigation of the COED Syncrude and H-Coal oil began in early 1974. During that year, characterization studies and straight run distillations were completed on the COED material. Two stage hydrocracking of the mid-distillate, and steam coil cracking of naphthas, mid-distillate, and first stage hydrocrackate products followed in early 1975. Hydrocracking of the straight run heavy gas oil was completed in September 1975 and two stage hydrotreating of the straight run naphtha followed.

The H-Coal processing followed the pattern of the COED, but required an additional step. The H-Coal material was received as atmospheric still bottoms and overheads, and vacuum still bottoms slurry. While the former was processed in a manner similar to the COED Syncrude, a disposable still was constructed to separate the heavy gas oil from the solidified slurry for further processing. The resulting vacuum gas oil was hydrocracked in October 1975. The two stage hydrotreated H-Coal naphtha was reformed in December 1975.

Samples of Synthoil and SRC materials were also obtained during 1975. Based on characterizations of the samples, it was decided to limit the study of the Synthoil to the mid-distillate. The Synthoil straight run distillation was completed in December 1975.

Hydrocrackate SRC product was received from the U.S. Bureau of Mines. Simulated distillation data indicated a low conversion level for the mid-distillate oil. Therefore, it was decided that only the light oil produced in the process would be studied.

In the steam coil cracking experiments performed on COED and H-Coal oil naphthas and mid-distillates, the

olefins yield was relatively low, but the aromatic yield made the naphtha feedstocks attractive. The results showed that:

- H-Coal straight-run naphtha was a slightly better ethylene feedstock than COED straight run naphtha.
- Cracking H-Coal mid-distillate produced twice as much fuel, 38 percent fewer olefins, and 22 percent less benzene than cracking H-Coal naphtha.
- Cracking H-Coal mid-distillate produced approximately the same amount of olefins and fuel, but nearly twice the benzene than cracking COED mid-distillate.

In order for the COED straight-run mid-distillate to be used as feedstock for petrochemical production, hydroprocessing would probably be necessary. A combination of hydroprocessing and steam coil cracking might convert the heavier COED oils to desirable petrochemicals more economically.

The petrochemical processing of COED syncrude was completed in the beginning of 1976. Reforming of H-Coal hydrocrackate naphthas was also completed after changing the presulfiding procedure for the hydrotreat-int catalyst. First and second pass hydrocracking of Synthoil and SRC II (slurry recycle) mid-distillates was completed as was the hydrotreating of the product naphthas.

PROGRESS DURING APRIL-JUNE 1977

Efforts during the quarter were directed at compiling the interim reports. Work on the conceptual design for a plant to produce chemicals from coal liquefaction products continued.

XIII. R&D ENGINEERING SERVICES — HYDROCARBONIZATION

OAK RIDGE NATIONAL LABORATORY

OAK RIDGE, TENNESSEE

Contract No.: E(49-18)-1740

Contract Funding: \$2,135,000
(100% ERDA)

INTRODUCTION

The Oak Ridge National Laboratory (ORNL) is conducting several research and development projects in support of the increased utilization of coal as a source of clean energy. Under this agreement, begun in 1974, ORNL estimated the optimum operating conditions of a fluidized-bed hydrocarbonization reactor and designed, constructed, and operated a fluidized-bed reactor capable of continuously processing ten pounds of coal per hour. Operating conditions for this reactor are at least 20 atm and up to 1,200°F.

PROCESS DESCRIPTION

Hydrocarbonization is a type of coal liquefaction process which combines high-temperature, high-pressure, and fluidized-bed operation using hydrogen-rich gas for fluidization. The hydrocarbonization process can produce controlled yields of desulfurized char, liquid fuels, and substitute natural gas. The product char may be suitable for use in boilers, without stack gas treatment units. The liquid products may be composed of naphtha, light gas oil, and heavy gas oil. A flow diagram of the bench-scale hydrocarbonization unit is shown in Figure XIII-1.

The work under this contract is divided into three tasks:

- Review and evaluation, involving the analysis of prior work to estimate operating conditions and guide reactor design.
- Experimental development, which includes the investigation of hydrocarbonizer components at atmospheric pressure and temperatures up to 1,200°F.
- Bench-scale studies involving the design, construction, and operation of continuous hydrocarbonization system capable of processing ten pounds of coal per hour at temperatures up to 1,200°F and pressures as great as 20 atm.

HISTORY OF THE PROJECT

An intensive literature survey conducted during 1975 led to the preliminary conclusions that very little reaction takes place below 750°F and the yield of both oil and gas increases directly with temperature. A maximum oil yield was noted at 1,020°F to 1,110°F. Gas production increased proportionally with temperature and therefore did not show an optimum over the temperature range studied.

Two Lucite mock-ups were constructed to study the mechanics of hydrocarbonization processes. An atmospheric pressure high-temperature ten-pound-per-hour hydrocarbonization bench-scale unit was designed based on tests on the Lucite mock-ups. The system was fabricated and installed during 1975 and shakedown tests were initiated late in the year. Modifications of the bench-scale

Figure XIII-1. FLOW DIAGRAM OF BENCH-SCALE HYDROCARBONIZATION SYSTEM

system to permit recirculating fluidized-bed operation was completed in early August 1976.

PROGRESS DURING APRIL-JUNE 1977

Summary

The first hydrocarbonization experiments using an agglomerating coal, Illinois No. 6, were performed during the quarter. Efforts were directed at attempts to develop a procedure for handling of caking coals. Cold model testing in support of the bench-scale hydrocarbonization experiments also continued.

Discussion of Activities

At the beginning of the quarter, several changes were made in the hydrocarbonization equipment. In an effort to more accurately measure the gas flowrates to the equipment, all the orifice flow meters were recalibrated, and one new orifice plate was installed directly upstream of the gas preheater.

The first attempt at hydrocarbonizing an agglomerating coal was performed in April with Run HC-15. Coal feed for the run was prepared by mixing 20 pounds of Illinois No. 6 coal, sized to -50 +150 mesh, with 100

pounds of char from Wyodak coal which had been devolatilized at 1200°F in the atmospheric pressure carbonizer. Three attempts at feeding coal were terminated by the coal transport tube plugging at the inlet to the reactor. Modifications were subsequently made to the equipment in an attempt to develop a precedence for handling of caking coals.

During May, two operability tests, Runs HC-16 and HC-17, were successfully completed using Illinois No. 6 coal diluted with char. Run HC-16 was the first successful attempt to sustain feeding of caking coal to the reactor. A mixture of one part Illinois No. 6 coal to five parts char was fed to the reactor at a rate of 11.4 pounds-per-hour for a period of 1.25 hours. The reactor bed was at 825°F at 300 psi hydrogen pressure. To aid in handling agglomerating coal, the hydrogen feed rate was increased from 10.7 standard cubic feet per minute used in Run HC-15 to 19.0 in this run. The higher hydrogen supply rate assured recirculation of the bed of char in the reactor.

In Run HC-17, a mixture of one part coal to six parts char was fed at a rate of 9.6 pounds-per-hour for seven hours without interruption. The reactor bed was at 825°F and 300 psig hydrogen pressure. A partial blockage in the reactor char overflow line terminated the run. The hydrogen flow rates were the same as for Run HC-16 and a char starter bed was also used.

During June, three operability tests were completed on the Illinois No. 6 coal. In Run HC-18, coal diluted six to

one with char which had previously been devolatilized at 1200°F was fed at 8.6 pounds-per-hour to the 950°F, 300 psig reactor bed. Run HC-19 was the first attempt to operate with a lower feed char to Illinois No. 6 coal dilution ratio of three to one. Hydrogen flow rates into the reactor were about the same as used in Run HC-18. The reactor pressure was 300 psig. The coal dilution ratio was the same in Run HC-20 as in Run HC-19. Uninterrupted feed periods of nine hours for Run HC-18, four hours for Run HC-19, and 4.5 hours for Run HC-20 were achieved with 8.5 pounds-per-hour nominal feed rate. A recurrent problem in each run was blockage of the reactor downcomer region by agglomerated solids adhering to the hot reactor wall.

Experiments in support of the hydrocarbonization tests were conducted in the ambient mock-up during the quarter. After modification of the mock-up, experiments were resumed in April with distributor plate evaluation and minimum fluidization velocity tests. In June, cold model testing included solids feeding and withdrawal experiments.

Gas flowrates for bed circulation during bench-scale hydrocarbonization experiments are scaled from the mock-up studies by maintaining constant the ratio of gas velocities to calculated minimum fluidization velocity. Critical evaluation of this scaling procedure began at the end of the quarter.

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XIV. ZINC HALIDE HYDROCRACKING PROCESS

CONOCO COAL DEVELOPMENT COMPANY
LIBRARY, PENNSYLVANIA

Project Site: Library, Pennsylvania
Contract No.: EX-76-C-01-1743
Contract Funding: \$11,410,400
ERDA: \$10,269,360
Industry: \$ 1,141,040

INTRODUCTION

Continental Oil Company, with the participation of Shell Development Company, a division of Shell Oil Company, is under contract to ERDA to develop the zinc halide hydrocracking process. The overall objective of the project is the production of clean liquid and gaseous fuels, of which approximately fifty percent will be distillate, equivalent to four barrels per ton of coal on a moisture and ash-free (MAF) basis.

Specifically, experimental studies will be performed involving both continuous bench-scale and process development unit work dealing with the investigation of zinc halide as a catalyst for the hydrogenation and hydrocracking of coal extract and subbituminous coal. Studies will also involve the development of an economical regeneration process for the efficient recovery of zinc halide from the spent melt. The schedule for the program is shown in Figure XIV-1.

PROCESS DESCRIPTION

The zinc halide hydrocracking process is designed to maximize the conversion of coal into light distillates by severe catalytic cracking. A schematic of the process is provided in Figure XIV-2. The coal is dried and pulverized and then introduced into a feed tank where it is

slurried with a process-derived recycle oil. The slurry feed is transferred to a hydrocracking reactor where it is mixed with hydrogen and the zinc chloride catalyst. In the reactor, the coal is cracked to distillates in the gasoline range, producing on distillation a clear 90 RON gasoline without further processing, and a small quantity of very low sulfur, low-nitrogen fuel oil. The gas is subsequently separated from the liquid in a product receiver. Spent catalyst is then fed to a regenerator for catalyst recovery.

PROGRESS DURING APRIL-JUNE 1977

Summary

All major equipment for the 100-pound-per-hour process development unit (PDU) had been received or was in transit at the end of the quarter. The optimization study on liquefaction of subbituminous coal with molten zinc chloride continued on the bench-scale hydrocracker. Supporting studies investigated the kinetics of secondary zinc recovery from cyclone underflow solids.

Process Development Unit

Construction of the PDU was proceeding well at the end of the quarter. All major equipment had arrived or

PROJECT	FY 75	FY 76	TR. QR.	FY 77	FY 78	FY 79	FY 80
LABORATORY SUPPORT	△ 2-5 lb./hr. BENCH SCALE UNIT			▽			
PROCESS DEVELOPMENT UNIT	PROCUREMENT-DESIGN	▽		CONSTRUCTION	▽ SHUTDOWN	OPERATION	▽

LEGEND : △ BEGIN MILESTONE
▽ COMPLETE MILESTONE

Figure XIV-1. ZINC HALIDE HYDROPROCESSING PROJECT SCHEDULE

Figure XIV-2. ZINC HALIDE HYDROCRACKING PROCESS FLOWSHEET

was in transit. The only area of concern was the high pressure valves. Efforts were being made to correct the faulty design of the valves.

Bench-Scale Hydrocracker Studies

During the quarter, material balance calculations were completed for four hydrotreating runs feeding 100 x 200°C coal derived distillate. Under typically severe hydrocracking conditions, only a total of about one percent of the 100 x 200°C distillate feed was converted to light gases or lost to spent catalyst. This indicates that coal-derived naphtha is quite resistant to zinc chloride hydrocracking and, if necessary, can be used to supplement heavier recycle oils to be used as slurry vehicle for the feed coal.

Two runs at 800°F reactor temperature and 3500 psig total pressure gave low gas yields and relatively low +475°C organic residue yields, thus showing that an upper limit on operating temperature at 3500 psig likely had not yet been reached in the continuous unit. Higher temperatures are favorable in that they reduce the amount of hydrocarbon contained in the spent melt going to regeneration.

Seven continuous hydrocracking runs were made in the bench-scale unit during May. In addition to coal runs,

these included a run with 200 x 475°C recycle distillate as the vehicle, a run using regenerated catalyst, a blank run with vehicle only and a run with PAMCO SRC. Slurry feeding was improved, with all runs giving complete material balances.

During June, five material balance runs were completed on the continuous zinc chloride liquefaction unit. Two of these extended the variable study with subbituminous coal to higher temperatures. Two runs defined the results when using toluene instead of benzene as the coal feed vehicle. The fifth run was made with Kentucky bituminous coal.

Supporting Laboratory Studies

Six continuous runs were made in April investigating the kinetics of secondary zinc recovery from cyclone underflow solids. The solids were a composite from a number of primary regeneration runs with natural, low-carbon spent melt. The experimental work to define conditions for secondary recovery of zinc from effluent coal ash continued in May with synthetic cyclone solids.

The continuous kinetics study was completed with a run in June. An economic evaluation of secondary recovery of zinc began during the quarter.

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XV. FLUID COKING OF COAL LIQUEFACTION RESIDUES

EXXON RESEARCH AND ENGINEERING
COMPANY

Contract No.: EX-76-C-012422

Contract Funding: \$1,312,470

INTRODUCTION

Exxon Research and Engineering Company, under the sponsorship of ERDA, will study the potential of fluid coking to recover distillate liquids. The primary objective of this contract is to evaluate the applicability of current commercial fluid coking technology to processing of residues from one selected coal liquefaction process. The program schedule for this project is shown in Figure XV-1.

PROGRAM DESCRIPTION

The treatment of residual, solids-containing liquids produced by various coal liquefaction processes is important in determining the overall economic viability of the process. Solids are removed from primary coal liquids by methods such as hydrocyclones, vacuum distillation, and solvent deashing. The solids-containing residues from these operations may contain additional valuable liquid products which may be recovered by fluid coking.

Three samples of coal liquid residue produced in ERDA-sponsored coal liquefaction projects will be analyzed and processed using bench-scale equipment. A Continuous Stirred Coking Unit (CSCU) will be used to obtain quantitative yield data for fluid coking of the coal liquefaction residues. This type of pilot unit was utilized successfully in scale-up of yield and quality data for petroleum. CSCU simulates the steam fluidization of a

commercial fluid coker by means of a mechanical agitator. A schematic is shown in Figure XV-2. One of the selected coal liquid residues will be processed in a nominal two-barrel-per-day fluidized-bed pilot unit.

HISTORY OF THE PROJECT

Work under this contract began in the last quarter of 1976. H-Coal solvent precipitation underflow was the first of three candidate coal liquefaction residues to be evaluated, with analytical tests completed by the end of the year. Bench-scale studies in the one-gram unit on coking yields for the H-Coal sample were completed and tests began in November on the Continuous Stirred Coking Unit (CSCU).

Figure XVI-1. FLUID COKING PROGRAM SCHEDULE

Figure XVI-2. FLUID COKING PROCESS SCHEMATIC

PROGRESS DURING APRIL-JUNE 1977

The second coal liquefaction residue sample, a stripped SRC coal liquefaction settler underflow sample (ERDA Residue 2) provided by Conoco Coal Development Company was received for testing in April. A one-gallon sample of stripped SRC solvent deashing underflow (ERDA Residue 3) was received from C-E Lummus Combustion Engineering Inc. in May. All Task 1 analytical tests for both samples were completed during the quarter. Analyses included elemental analysis, Conradson Carbon, pyridine insolubles, toluene insolubles, viscosity, specific gravity, and glass sprial/microlube distillations.

The elemental analysis for Residue 2 showed higher ash and sulfur contents, but a lower oxygen content, than

previously observed in coal liquefaction residues. The higher ash content was reflected in higher than previously encountered Conradson Carbon, pyridine and toluene insolubles, and apparent viscosity. The atomic hydrogen to carbon ratio of 0.76 was in the range of value observed for other coal liquefaction residues. The distillation results indicated the settler underflow sample contained approximately 13 percent 1000° F liquids. The specific gravity of 1.65 at 77° F was slightly high, but consistent with the high ash content.

The elemental analysis for Residue 3 also showed a low oxygen content and high ash and sulfur contents. The high ash content was reflected in high Conradson Carbon and pyridine and toluene insolubles. The 20.8 percent 1000° F liquid content was high and responsible for the low viscosity. The Residue 3 viscosity varied from 7 poise at 500° F to 3 poise at 600° F. The specific gravity, 1.47 at

77°F, and the hydrogen to carbon ratio of 0.81 were similar to values observed in other coal liquefaction residues.

Bench-scale screening tests were completed in the bench coking unit to define approximate coking yields for Residue 3. No studies were scheduled for Residue 2.

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XVI. UPGRADING OF COAL LIQUIDS

UOP, INC.

DES PLAINES, ILLINOIS

Contract No.: EF-77-C-01-2566

Contract Funding: \$997,782

INTRODUCTION

UOP, Inc. is under contract to ERDA to determine the applicability of commercial UOP conversion processes for all distillate coal liquids. Coal liquefaction processes may be separated into two groups: those that produce resid containing coal liquids, and those that produce a distillate coal liquid. UOP completed studies on upgrading resid containing coal liquids in 1976.

Distillate coal liquids being studied under the present contract include H-Coal Syncrude Mode and Exxon Donor Solvent.

The objectives of this contract are to:

- Process selected ERDA sponsored coal liquid distillates through a comprehensive hydrocracking scheme.
- Determine the response of the fluid catalytic cracking unit to hydrotreated coal liquid distillates.
- Maximize the production of either motor gasoline or No. 2 fuel oil by the hydrocracking scheme as stated in the first objective.
- Provide yield-octane curves by catalytic reforming naphthas produced in primary coal liquefaction programs and/or by the hydrocracking scheme as stated in the first objective.
- Correlate feedstock properties and process conditions data with product yields and qualities.

PROJECT DESCRIPTION

Four tasks are identified under this program. The first task involves two stage continuous catalyst bed hydrocracking of two coal liquid distillates. The second task entails processing of two coal liquid distillates through continuous fixed catalyst bed hydrotreating and fluid catalytic cracking units. The third task covers processing of two naphthas through continuous refining-reforming bench scale units. The fourth task involves the correlation of feedstock properties and process conditions with product yields and qualities which can be composed into a simple process model.

HISTORY OF THE PROJECT

The present contract is an extension of the program begun by UOP under contract E(49-18)-2010. Evaluations of the primary products of coal liquefaction generated by the Synthoil, Solvent Refined Coal (SRC), and H-Coal processes were completed in 1976, and the final report of the project was written. The present program began in January of 1977 under contract EF-77-C-01-2566.

PROGRESS DURING APRIL-JUNE 1977

First-stage hydrocracking of rerun H-Coal process atmospheric still bottoms over a commercial hydrocrack-

ing catalyst continued on the bench-scale continuous unit during the quarter. Primary objective of these studies was to investigate the effects of process variables and to arrive at a set of optimum conditions for a preparative run to produce a suitable feedstock for second-stage hydrocracking studies.

The first series was conducted in April at 500 psig above base pressure, one temperature, and three space velocities. The second series was carried out at 1000 psig above base pressure and otherwise similar conditions. In all cases a substantial amount of nitrogen was eliminated.

Four additional series were completed in May. Tests were run at 500 and 1000 psig above base pressure for two temperatures and three space velocities. In all cases, substantial amounts of nitrogen were removed.

In June, a new run was started to make a series of studies at base pressure and to recheck a few of the data points obtained previously.

At the end of the quarter, the first stage hydrocracking preparative run had been on stream a total of 660 hours during which 69 gallons of H-Coal gas oil was charged. The catalyst life at the end of the run was estimated to be 1.4 barrels per pound of catalyst.

All required analyses for the upgraded H-Coal naphtha were completed in April. This stock was reformed over a commercial Platforming catalyst. Experiments were carried out over a series of conditions for the purpose of obtaining data for a yield octane curve.

XVII. GASIFICATION OF RESIDUAL COAL LIQUEFACTION PRODUCTS

TEXACO, INC.

Contract No.: E(49-18)-2247

Contract Funding: \$939,840
(100% ERDA)

INTRODUCTION

Under this agreement, Texaco, Inc. will perform the research concerning the gasification of residual material from coal liquefaction samples supplied by ERDA. The objective of the contract is to evaluate materials from several coal liquefaction pilot plants to determine their suitability as feedstocks for a coal gasification process. After initial laboratory testing and evaluation, promising materials selected by ERDA will be tested in large scale pilot plant gasification runs.

PROCESS DESCRIPTION

The thermal efficiency of a coal liquefaction plant using a process that requires hydrogen or synthesis gas is dependent upon the efficient recovery of that gas from the non-liquefied fraction of the coal. This material, together with inorganic ash and some fraction of the converted coal, can be recovered after liquefaction as a residue from a vacuum distillation tower. The chemical and physical properties of the high-ash coal-derived residues determine both the method of introducing these materials into the gasifier and the efficiency of the gasifier. As Texaco has demonstrated that high-ash coal-derived residues can be gasified directly using the Texaco coal gasification process, the necessary design parameters will be determined for the materials resulting from ERDA-sponsored liquefaction projects.

HISTORY OF THE PROJECT

The contract was awarded to Texaco in June of 1976. Work began with receipt of two H-Coal residue samples during the last quarter of the year. Results of the tests performed on the feedstocks indicated that the "Syn-crude Vacuum Bottoms" was a suitable feedstock for the Texaco coal gasification process, while the "Settler Stripped Underflow" seemed a poor candidate because of its physical properties.

PROGRESS DURING APRIL-JUNE 1977

A type II preliminary pilot plant evaluation of 20 barrels of Syncrude Vacuum Tower Bottoms from the H-Coal liquefaction of Illinois No. 6 coal was successfully completed. The data indicated that a high conversion of carbon to syngas was achieved with predicted oxygen consumption.

During the quarter, 8,750 pounds of residue were gasified in 8.75 hours of continuous operation at 350 psig. The molten residue was fed to the Texaco pilot plant gasifier at a rate of about 1,000 pounds-per-hour, producing 33,000 SCFH of dry syngas. At the oxygen rate employed, 97 percent of the carbon in the residue was gasified. About 80 percent of the ash in the residue was recovered, 18 percent as coarse slag, 32 percent as fire slag, and 30 percent as soot.

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XVIII. PRODUCTION OF CLEAN INDUSTRIAL AND TRANSPORTATION FUELS FROM COAL

THE LUMMUS COMPANY
BLOOMFIELD, NEW JERSEY

Plant Site: Bloomfield, New Jersey
Contract No.: E(49-18)-2514
Contract Funding: \$4,700,000

INTRODUCTION

The Lummus Clean Fuel From Coal (CFFC) Process is being studied under this contract for the purpose of establishing optimum operating conditions and proving the reliability of the process. The primary objectives of this development program are to:

- Establish recycle oil self-sufficiency, catalyst life, and process operability for coal liquefaction and demonstrate comparative hydroliquefaction performance of a proprietary Lummus catalyst versus a standard cobalt-molybdate catalyst.
- Convert the all-distillate coal liquid product to gasoline by continuous fixed-bed hydrotreating/hydrocracking operations.
- Provide a preliminary engineering design of a Lummus CFFC pilot plant facility.

The effort is divided into three tasks. Task I is Coal Liquefaction and involves the modification and operation of an existing 30-pound-per-hour expanded bed test unit. The modifications will permit continuous integrated operation of the process, including the hydroliquefaction reactors, solvent de-ashing module, the distillation unit, and the vacuum flashing units. Illinois No. 6 coal, containing at least three percent sulfur and one percent nitrogen, will be used in the bench-scale tests to evaluate the process. Operations will be conducted to produce an all-distillate product.

Task II, Hydrotreating/Hydrocracking, will involve the operation of a multistage fixed-bed hydrotreating/hydrocracking unit to determine the temperature, pressure, space velocity, and recycle rates required to convert the coal liquid to gasoline. A proprietary Lummus denitrogenation catalyst will be used in the first stage, and tungsten nickel sulfide will be used in the second stage.

In Task III, Engineering and Estimating, the results of studies conducted in the previous tasks will be used to formulate a preliminary engineering design of a pilot plant facility. The overall program plan is shown in Figure XVIII-1.

PROCESS DESCRIPTION

The Lummus Clean Fuels From Coal (CFFC) Process is based on direct catalytic hydroliquefaction in a multi-stage, plug-flow ebullated-bed reactor. A schematic of the process is presented in Figure XVIII-2. The process employs an integrated solvent de-ashing technique requiring no mechanical separation operations. The ebullated bed is produced by the upward flow of coal feed, slurry oil, and hydrogen-rich gas entering at the bottom of the reactor. The Lummus process takes advantage of both the high reaction rates of a plug-flow reactor and the non-fouling features of the ebullated bed reactor.

Figure XVIII-1. CLEAN FUELS FROM COAL PROGRAM DEVELOPMENT SCHEDULE

Figure XVIII-2. CLEAN FUELS FROM COAL PROCESS SCHEMATIC

The use of multiple staging permits superior control of reactor conditions resulting in minimal hydrogen consumption. Since most of the process heat release is due to hydrogen consumption, the reduced hydrogen uptake in the process also minimizes heat dissipation problems. While net hydrogen consumption is kept at a minimum, the Lummus process also exhibits a high degree of efficiency in hydrogen utilization.

The Lummus process uses a proprietary solvent de-ashing system which, unlike filters and centrifuges, does not contain costly and hard-to-develop mechanical equipment, thus simplifying scaleup. In addition, solvent de-ashing does not require increased process severity to produce a lighter product capable of recovery from a vacuum distillation.

HISTORY OF THE PROJECT

The original Lummus bench-scale coal liquefaction pilot plant became operational in 1972 and a one-year process development program was conducted using Illinois No. 6 coal. A technically feasible process capable of producing a liquid product with a sulfur content of 0.3 percent and an ash content of 0.1 percent was demonstrated. The Lummus de-ashing system was found to be operational. Since the initial work, two subbituminous coals have been processed successfully in the bench-scale pilot plant.

Lummus also performed exploratory work on denitrogenation catalysts. A highly active catalyst was formulated in late 1974 and a patent application on the catalyst was filed.

The proposed program was initially submitted to ERDA in 1975 as an unsolicited proposal. Following

contract negotiations, a revised experimental program was authorized and a contract granted by ERDA on September 28, 1976. Modifications were started to convert the expanded-bed liquefaction test unit and its ancillary anti-solvent de-ashing module, a distillation unit, and two vacuum flashing units to permit integrated continuous operation.

PROGRESS DURING APRIL-JUNE 1977

The mechanical and electrical subcontract work was completed by the third week of April. Final pressure testing and insulation was completed on the new equipment. Integrated shakedown operations on the downstream units continued during the quarter in preparation for the introduction of coal feed to the hydroliquefaction unit.

Testing of the denitrification catalyst continued during the quarter. A small-scale catalyst test unit was erected for the purpose of making comparative tests of the proprietary catalyst versus commercially prepared high activity catalyst. Tests in April were performed on coal paste made with the use of prehydrogenated paste oil. Coal pasted with raw (non-hydrogenated) paste oil is found to be more difficult to denitrify than coal paste prepared with prehydrogenated solvent. Recycling of the coal paste to the hydroliquefaction unit improves the kinetics of nitrogen removal.

The heavy coal tar distillate to be used as the source material for startup paste oil was received from Koppers and analyzed in April.

Mechanical problems hampered operations of the hydroliquefaction unit during the quarter, delaying perhydrogenation of the startup inventory of paste oil.

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GLOSSARY

absorption—an imprecise term suggesting the taking up of one substance by another by either a physical process or a chemical combination.

acceptor—calcined carbonate that absorbs carbon dioxide evolved during gasification, liberating heat.

acid gas removal—the process of selectively removing hydrogen sulfide and carbon dioxide from a gas stream.

activated carbon—carbon obtained by carbonization in the absence of air, preferably in a vacuum; has the property of absorbing large quantities of gases, solvent vapors; used also for clarifying liquids.

adiabatic—any process where heat is neither given off nor absorbed.

adsorption—the process by which the surface of a solid or liquid attracts and holds any atom, molecule, or ion from a solution or gas with which it is in contact.

agglomerate—assemblage of ash particles rigidly joined together, as by partial fusion (sintering).

anthracite coal—hard coal containing 86 to 98 percent fixed carbon and small percentages of volatile material and ash.

API—American Petroleum Institute.

API gravity—a scale adopted by the API for measuring the density of oils; ° API = $\frac{141.5}{\text{Specific gravity, } 60^{\circ}\text{F}/60^{\circ}\text{F}} - 131.5$

aromatic hydrocarbon—a cyclic hydrocarbon containing one or more six-carbon (benzene) rings.

ash—solid residue remaining after the combustion of coal.

ASTM—American Society for Testing Materials.

autoclave—a vessel, constructed of thick-walled steel, for carrying out chemical reactions under high pressures and temperatures.

bench-scale unit—a small-scale laboratory unit for testing process concepts and operating parameters as a first step in the evaluation of a process.

binder—carbon products, tars, etc., used to impart cohesion to the body to be formed; a coal-extract binder may be used to prepare formed-coke pellets from non-coking coals.

bituminous coal—a broad class of coals containing 46 to 86 percent fixed carbon and 20 to 40 percent volatile matter.

blow down—periodic or continuous removal of water from a boiler to prevent accumulation of solids.

bottoming cycle—the lower temperature thermodynamic power cycle of a combined-cycle system.

Btu—British thermal unit, the quantity of energy required to raise the temperature of one pound of water one degree Fahrenheit.

BTX—benzene, toluene, xylene; aromatic hydrocarbons.

caking—the softening and agglomeration of coal as a result of the application of heat.

calcination—the process of heating a solid to a high temperature to cause the decomposition of hydrates and carbonates.

caloric value—the quantity of heat obtained by the complete combustion of a unit mass of a fuel under prescribed conditions.

carbon fiber—fine filaments of carbon about eight microns in diameter which are used in composite materials, being bound with resins.

carbonization—destructive heating of carbonaceous substances with the production of a solid, porous residue, or coke, and the evolution of a number of volatile products. For coal, there are two principal classes of carbonization, high-temperature coking (about 900°C) and low-temperature carbonization (about 700°C).

catalyst—a substance that accelerates the rate of a chemical reaction without itself undergoing a permanent chemical change.

centrifuge—an apparatus rotating at high speed which utilizes the centrifugal force generated to separate materials of different densities, e.g., undissolved residue from coal solution in the SRC process.

char—the solid residue remaining after the removal of moisture and volatile matter from coal.

Claus process—industrial method of obtaining elemental sulfur through the partial oxidation of gaseous hydrogen sulfide in air followed by catalytic conversion to molten sulfur.

coal—a readily combustible rock containing more than 50 weight percent and more than 70 volume percent of carbonaceous material including inherent moisture, formed from compaction and induration of variously altered plant remains similar to those in peat.

coalification—metamorphosis of vegetable debris into coal.

coke—strong porous residue consisting of carbon and mineral ash formed when bituminous coal is heated in a limited air supply or in the absence of air. Coke may also be formed by thermal decomposition of petroleum residues.

coke breeze—the fine screenings from crushed coke usually passing a 1/2 inch or 3/4 inch screen opening.

combined cycle—two sequential thermodynamic power conversion systems operating at different temperatures.

combustion gas—gas formed by the combustion of coal, e.g., burning.

combustor—a vessel in which combustion takes place.

coupon—a polished metal strip used to measure the rate of corrosion of the metal in a specific gaseous or liquid environment.

cracking—the partial decomposition of high-molecular-weight organic compounds into lower-molecular-weight compounds, generally as a result of high temperatures.

crude gas—impure gas produced in a gasifier.

culm—the waste or slack from anthracite mines or preparation plants consisting of fine coal, coal dust, and dirt.

cyclone separator—essentially a settling chamber to separate solid particles from a gas, in which gravitational acceleration is replaced by centrifugal acceleration.

degasification—a process for removing naturally occurring methane from coal seams.

delayed coking—a process wherein coal is subjected to a long period of carbonization at moderate temperatures to form coke.

demineralization—removal of mineral matter (ash) from coal by solvent extraction, usually under hydrogen atmosphere.

depolymerization—the change of a large molecule into simpler molecules usually accompanied by the substitution of hydrogen for oxygen in the molecular structure.

destructive distillation—the distillation of coal accompanied by its thermal decomposition.

desulfurization—the removal of sulfur from hydrocarbonaceous substances by chemical reactions.

devolatilization—the removal of a portion of the volatile matter from medium- and high-volatile coals.

diatomaceous earth—a yellow, white, or light-gray, siliceous porous deposit made up of opaline shells of diatoms; used as a filter aid, paint filler, adsorbent, abrasive, and thermal insulator. Also known as kieselguhr.

diatomite—See Diatomaceous Earth.

dissolution—the taking up of a substance by a liquid with the formation of a homogeneous solution.

distillation—a process of vaporizing a liquid and condensing the vapor by cooling; used for separating liquids into various fractions according to their boiling points or boiling ranges.

dolomite—a carbonate of calcium and magnesium having the chemical formula $\text{CaMg}(\text{CO}_3)_2$.

Dowtherm—trademark for a series of eutectic mixtures of diphenyl oxide and diphenyl used as high-temperature heat-transfer fluids.

ebullated bed—gas containing a relatively small proportion of suspended solids, bubbles through a higher density fluidized phase with the result that the system takes on the appearance of a boiling liquid.

economizer—heat exchanging mechanism for recovering heat from flue gases.

effluent gas—gas given off from a process vessel.

elutriation—the preferential removal of the small constituents of a mixture of solid particles by a stream of high-velocity gas.

endothermic reaction—a process in which heat is absorbed.

enthalpy change—the increase or decrease in heat content of a substance or system which accompanies its change from one state to another under constant pressure.

entrained bed (flow)—a bed in which solid particles are suspended in a moving fluid and are continuously carried over in the effluent stream.

eutectic—that combination of two or more components which produces the lowest melting temperature.

exothermic reaction—a process in which heat is liberated.

extraction—a method of separation in which a solid or solution is contacted with a liquid solvent (the two being essentially mutually insoluble) to transfer components into the solvent.

extractive coking—similar to delayed coking process, with the emphasis on high tar yields to produce liquids.

filter aid—finely divided solids used to increase efficiency of filtering.

filter cake—the moist residue remaining from the filtration of a slurry to produce a clean filtrate.

filtrate—a liquid free of solid matter after having passed through a filter.

filtration—the separation of solids from liquids by passing the mixture through a suitable medium, e.g., cloth, paper, diatomaceous earth.

Fischer assay—method for determining the tar and light oil yields from coal or oil shale; conducted in a retort under an inert atmosphere with a prescribed increase in temperature to 500°C.

Fischer-Tropsch catalyst—catalysts developed for the catalytic synthesis of liquid fuels from coal-derived synthesis gas; catalysts contain principally iron, cobalt, nickel, or ruthenium.

Fischer-Tropsch process—method of hydrogenating mixtures of

carbon monoxide and hydrogen produced from coal, lignite, or natural gas by means of steam, at 1-10 atmospheres and 360-410°F to yield liquid and gaseous fuels, and a wide spectrum of industrial chemicals.

fixed-bed—stationary solid particles in intimate contact with fluid passing through them.

fixed carbon—the solid residue, other than ash, obtained by destructive distillation; determined by definite prescribed methods.

flash carbonization—a carbonization process characterized by short residence times of coal in the reactor to optimize tar yields.

flue gas—gaseous combustion products.

fluidization (dense phase)—the turbulent motion of solid particles in a fluid stream; the particles are close enough as to interact and give the appearance of a boiling liquid.

fluidization (entrained)—gas-solid contacting process in which a bed of finely divided solid particles is lifted and agitated by a rising stream of gas.

fluidized-bed—assemblage of small solid particles maintained in balanced suspension against gravity by the upward motion of a gas.

fly ash—a fine ash from the pulverized coal burned in power station boilers, or entrained ash carried over from a gasifier.

fractionation—distillation process for the separation of the various components of liquid mixtures.

freeboard—the space in a fluidized-bed reactor between the top of the bed and the top of the reactor.

free swelling index—a standard test that indicates the caking characteristics of coal when burned as a fuel.

Friedel-Crafts reaction—a substitution reaction, catalyzed by aluminum chloride in which an alkyl (R-) or acyl (RCO-) group replaces a hydrogen atom of an aromatic nucleus to produce a hydrocarbon or a ketone.

fuel cell—a galvanic cell in which the chemical energy of a conventional fuel is utilized to produce electricity.

fuel gas—low heating value (150-350 BTU/scf) product generally utilized on site for power generation or industrial use.

gasification of coal—the conversion of solid coal into a gaseous form by various chemical reactions with steam.

gasifier—a vessel in which gasification occurs, usually utilizing fluidized-bed, fixed-bed, or entrained-bed units.

heat capacity—quantity of heat required to raise the temperature of one pound of a substance one degree Fahrenheit.

high-Btu gas—a gas having a heating value of 900 to 1,000 Btu per standard cubic foot, which approaches the value for natural gas.

higher heating value (HHV)—the heat liberated during a combustion process in which the product water vapor is condensed to a liquid and the heat of condensation is recovered.

hydroclone—a small cyclone extractor for removal of suspended solids from a flowing liquid by means of the centrifugal forces set up when the liquid is made to flow through a tight conical vortex.

hydrocoking—coking of tars, SRC, etc., under hydrogenating conditions to form liquid products.

hydrocracking—the combination of cracking and hydrogenation of organic compounds.

hydrogasification—gasification that involves the direct reaction of fuels with hydrogen to optimize formation of methane.

hydrogenation—chemical reactions involving the addition of gaseous hydrogen to a substance in the presence of a catalyst under high temperatures and pressures.

hydrogen donor solvent—solvent, such as anthracene oil, tetralin (tetrahydronaphthalene), decalin, etc., which transfers hydro-

gen to coal constituents causing depolymerization and consequent conversion to liquid products of lower boiling range which are then dissolved by the solvent.

hydrotreating—a process to catalytically stabilize petroleum or other liquid hydrocarbon products and/or remove objectionable elements from products or feedstocks by reacting them with hydrogen.

Ideal gas—any gas whose equation of state is expressed by the ideal gas law, namely $PV = nRT$ where P is the pressure, V is the volume, R is the gas constant, T is the absolute temperature, and n = number of moles.

Ignition temperature—the minimum temperature necessary to initiate self-sustained combustion of a substance.

Industrial gas—see fuel gas.

Inerts—constituents of a coal which decrease its efficiency in use, e.g., mineral matter (ash) and moisture in fuel for combustion.

In situ—in its original place, e.g., underground gasification of a coal seam.

Intermediate-Btu gas—synthesis gas product with a higher heating value between 350 and 500 Btu per standard cubic foot.

lignite—brownish-black coal containing 65-72 percent carbon on a mineral-matter-free basis, with a rank between peat and subbituminous coal.

limestone—sedimentary rock containing 50 percent carbonate (CO_3) of lime or magnesia. Chemical formula (for calcite limestone) is CaCO_3 .

liquefaction—conversion of a solid to a liquid; with coal, this appears to involve the thermal fracture of carbon-carbon and carbon-oxygen bonds, forming free radicals. These radicals abstract hydrogen atoms yielding low molecular weight gaseous and condensed aromatic liquids.

liquefied petroleum gas (LPG)—those hydrocarbons that have a vapor pressure (at 70°F) slightly above atmospheric (such as propane and butane); kept in liquid form under a pressure higher than 1 atm.

lock hopper—a mechanical device that permits the introduction of a solid into an environment of different pressure.

low-Btu gas—a gas having a heating value up to 350 Btu per standard cubic foot.

lower heating value—the heat liberated by a combustion process assuming that none of the water vapor resulting from the process is condensed, so that its latent heat is not available.

MAF—moisture and ash-free; a term that relates to the organic fraction in coal.

mesh—measure of fineness of a screen, e.g., a 400-mesh sieve has 400 openings per linear inch.

methanation—the production of methane (CH_4) from carbon monoxide or dioxide and hydrogen.

methane— CH_4 , a colorless, odorless, and tasteless gas, lighter than air; the chief component of natural gas.

methanol—methyl alcohol, CH_3OH .

micron—a unit of length equal to one millionth of a meter; 10^{-6} meter.

moving bed—particled solids in a process vessel that are circulated (moved) either mechanically or by gravity flow.

natural gas—naturally occurring gas extracted from sedimentary structures consisting mainly of methane and having a higher heating value of approximately 1,050 Btu per standard cubic foot.

noncoking coal—a coal that does not form coke under normal coking conditions.

olefinic hydrocarbon—a class of unsaturated hydrocarbons containing one or more double bonds and having the general chemical formula C_nH_{2n} .

open cycle—a thermodynamic power cycle in which the working fluid passes through the system only once and is then exhausted to the atmosphere.

peat—an unconsolidated, hydrophilic, yellowish-brown to brownish-black, carbonaceous sediment, formed by accumulation of partially fragmented and decomposed plant remains in swamps and marshes which retains more than 75 percent inherent moisture and less than 12 percent mineral matter in saturated natural deposits.

petrochemicals—those derived from crude oil or natural gas, or their coal-derived substitutes; they include light hydrocarbons such as butylene, ethylene and propylene, the raw materials for the production of plastics by polymerization.

phenols—a group of aromatic compounds having the hydroxyl (OH) group directly attached to the benzene ring.

pilot plant—a chemical process plant containing all the processes of a commercial unit, but on a smaller scale, for the purpose of studying the technical and economic feasibility of the process.

pipeline gas—a methane-rich gas that conforms to certain standards and has a higher heating value between 950 and 1,050 Btu per standard cubic foot.

plenum chamber—an enclosed space through which air is forced for slow distribution through ducts.

precoat—layer of suitable filtering medium, e.g., diatomaceous earth, laid down on a rotary filter cloth prior to operation.

prilling tower—a tower that produces small solid agglomerates by spraying a liquid solution in the top and blowing air up from the bottom.

process development unit—a system used to study the effects of process variables on performance; sized between a bench-scale unit and a pilot plant.

proximate analysis—analysis of coal based on the percentages of moisture, volatile matter, fixed carbon (by difference), and ash, using prescribed methods. Reported on different bases, such as as-received (or as-fired), dry, mineral-matter-free (mmf), and dry mineral-matter-free (dmmf).

purification—removal of a wide range of impurities present in gases from coal gasification.

pyrolysis—thermal decomposition of organic compounds in the absence of oxygen.

quenching—cooling by immersion in oil, water bath, or water spray.

Raney nickel catalyst—specially prepared nickel catalyst used in the hydrogenation of organic materials and the methanation of synthesis gas to methane.

raw gas—see crude gas.

reactivity—susceptibility to chemical change; for example, in coal liquefaction, the reactivity of the coal for conversion to liquid products is a function of the coal rank, among other things.

reactor—vessel in which coal-conversion reactions take place.

Rectisol process—a process for the purification of coal-gasification gas based on the capability of cold methanol to absorb all gas impurities in a single step; gas naphtha, unsaturated hydrocarbons, sulfur compounds, hydrogen cyanide, and carbon dioxide are removed from the gas stream by the methanol at temperatures below 0°C.

reducing gas—a gas which, at high temperatures, lowers the state of oxidation of other chemicals.

reforming processes—a group of proprietary processes in which low-grade or low molecular weight hydrocarbons are catalytically converted to higher grade or higher molecular weight materials; also applies to the endothermic reforming of methane, for the production of hydrogen, by the reaction of methane and steam in the presence of nickel catalysts.

refractory—a material capable of withstanding extremely high temperatures and having a relatively low thermal conductivity.

residence time—time spent by a typical particle in a particular zone.

saturated hydrocarbon—a carbon-hydrogen compound with all carbon bonds filled; that is, there are no double or triple bonds as in olefins and acetylenes.

scrubber—apparatus in which a gas stream is freed of tar, ammonia, and hydrogen sulfide.

seam coal—coal which is intermediate in rank between bituminous coal and anthracite; contains 8 to 22 percent volatile matter and from 91 to 93 percent carbon.

semi-water gas—a mixture of carbon monoxide, carbon dioxide, hydrogen, and nitrogen, obtained by passing an air-stream mixture through a hot bed of coke, having a higher heating value of about 120 Btu per standard cubic foot.

sensible heat—that heat which results in only the elevation of the temperature of a substance with no phase changes.

shift conversion—process for the production of gas with a desired carbon monoxide content from crude gases derived from coal gasification; carbon monoxide-rich gas is saturated with steam and passed through a catalytic reactor where the carbon monoxide reacts with steam to produce hydrogen and carbon dioxide, the latter being subsequently removed in a scrubber employing a suitable sorbent.

sintering—the agglomeration of solids at temperatures below their melting point, usually as a consequence of heat and pressure.

slag—molten coal ash composed primarily of silica, alumina, iron oxides, and calcium and magnesium oxides.

slurry—a suspension of pulverized solid in a liquid.

solvation—the association or combination of molecules of solvent with solute ions or molecules.

solvent—that component of a solution which is present in excess; liquid used to dissolve a substance.

solvent extraction—selective solution of coal constituents from finely divided coal particles into a suitable solvent after intimate mixing, usually at high temperatures and pressures in the presence of hydrogen, with or without a catalyst, followed by phase separation.

solvent refined coal (SRC)—a coal extract derived by solvent extraction; a brittle, vitreous solid (m.p. 300°F to 400°F) containing about 0.1 percent ash and about 10 percent of the sulfur in the original coal feedstock; calorific value is about 16,000 Btu per pound; may be used as a clean fuel for power generation by combustion; utilized for the production of high-grade metallurgical coke, anode carbon, and activated carbon by coking, or hydrogenated to produce synthetic crude oil.

space velocity—volume of a gas (measured at standard temperature and pressure) or liquid passing through a given volume of catalyst in a unit time.

specific gravity—ratio of the weight of any volume of a substance to the weight of an equal volume of water at 4°C.

specific heat—heat capacity of a substance as compared with the heat capacity of an equal weight of water.

standard cubic foot (SCF)—the volume of a gas at standard conditions of temperature and pressure. The American Gas Association

uses moisture-free gas at 60°F and 30 inches of mercury (1.0037 atm) as its standard conditions. The pressure standard is not universal in the gas industry: 14.7 psia (1.000 atm) and 14.4 psia (0.980 atm) are also used. The scientific community uses 32°F and 1 atm as standard conditions.

stoichiometry—the definite proportions in which molecules react chemically to form new molecules.

stripping—the removal of the more volatile components from a liquid mixture of compounds.

subbituminous coal—the rank of coal between bituminous and lignite, classified by ASTM as having a range of heating values between 8,300 and 11,000 Btu per pound on a moist mineral-matter-free basis.

substitute natural gas (SNG)—a gas produced from coal, oil sands, or oil shale conforming to natural gas standards.

superficial velocity—the linear velocity of a fluid flowing through a bed of solid particles calculated as though the particles were not present.

superheater—a heat exchanger which adds heat to the saturated steam leaving a boiler.

syncrude—synthetic crude oil; oil produced by the hydrogenation of coal, coal extracts, oil sands, or oil shale, which is similar to petroleum crude.

synthesis gas—a mixture of hydrogen and carbon monoxide which can be reacted to yield a hydrocarbon.

tail gas—a gas issuing from a gas-treatment unit which may be recycled to the process or exhausted.

tar (coal)—a dark brown or black, viscous, combustible liquid formed by the destructive distillation of coal.

therm—a unit of heat used as a basis for the sale of natural gas; equal to 100,000 Btu.

topping cycle—the higher temperature thermodynamic power cycle of a combined-cycle system.

turndown ratio—the minimum ratio of actual flowrate to design flowrate at which a process unit can be operated.

ultimate analysis—the determination by prescribed method of carbon and hydrogen in the material as found in the gaseous products of its complete combustion, the determination of sulfur, nitrogen, and ash in the material as a whole and the estimation of oxygen by difference; may be reported on different bases, such as as-received (or as-fired), dry, mineral-matter-free (mmf), and dry mineral-matter-free (dmmf).

Venturi scrubber—a gas cleaning device which involves the injection of water into a stream of dust-laden gas flowing at a high velocity through a contracted portion of a duct, thus transferring the dust particles to the water droplets which are subsequently removed.

volatile matter—those constituents of coal, exclusive of moisture, that are liberated from a sample when heated to 1750°F for seven minutes in the absence of oxygen.

water gas—gas produced by the reaction of carbon (in coal or coke) and steam to yield mixtures of carbon monoxide and hydrogen; similar to synthesis gas.

water gas shift—the reaction between water vapor and carbon monoxide to produce hydrogen and carbon dioxide or the reverse: $\text{CO} + \text{H}_2\text{O} \rightleftharpoons \text{H}_2 + \text{CO}_2$

working fluid—a gas stream which directly does work, e.g., powering a gas turbine.