**THE DEVELOPMENT OF AN INTEGRATED MULTISTAGE
FLUID BED RETORTING PROCESS**

Quarterly Technical Report for the Period

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by

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MASTER

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ABSTRACT

This report summarizes the progress made on the development of an integrated multistage fluidized bed retorting process (KENTORT II) during the period of January 1, 1993 through March 31, 1993 under Cooperative Agreement No. DE-FC21-90MC27286 with the Morgantown Energy Technology Center, U.S. Department of Energy. The KENTORT II process includes integral fluidized bed zones for pyrolysis, gasification, and combustion of the oil shale. The purpose of this program is to design and test the KENTORT II process at the 50-lb/hr scale. The major activity for this quarter was to install various components of the process and provide utility support including air, water, electrical power, and computerized instrumentation. Following the completion of construction activities which is scheduled for next quarter, cold-flow testing and heat-up procedures will be performed.

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INTRODUCTION AND BACKGROUND

It is now well accepted that fluidized bed pyrolysis of oil shale at near-zero hydrogen partial pressure will generate higher than Fischer assay oil yields. This enhancement varies according to shale type, but for most shales, it can be significant. It has been shown that fluidized bed pyrolysis of eastern and western U.S. oil shales can enhance oil yields by 50%^{1,2,3,4,5} and 10%^{6,7} above Fischer assay, respectively. In addition, fluidized bed technology offers the advantages of rapid pyrolysis kinetics (short solid residence time), total use of mined shale, and thermal efficiency. The Center for Applied Energy Research (CAER) has developed a commercial retorting concept, KENTORT II, which is based on fluidized bed technology. This process has been successfully demonstrated in a 3-inch diameter, 5-lb/hr system at the CAER using eastern U.S. oil shale.⁸ The objective of the current program is to scale the KENTORT II process to a 6-inch diameter, 50-lb/hr reactor.

Approximately 40% of the original carbon remains in the spent shale following fluidized bed pyrolysis of Eastern shale. This is more carbon than is required to provide process heat, and an intermediate gasification stage was included in the KENTORT II design to utilize this excess carbon for synthesis gas production. The gasification section is also important because it permits steam/iron sulfide reactions to go nearly to completion. These reactions remove the majority of the sulfur from the shale in the case of eastern shale because approximately 90% of its sulfur is present as iron sulfides. The removal of sulfur from the shale as H_2S prior to combustion creates a relatively concentrated stream of H_2S which is easier to scrub than a dilute stream of SO_2 from combustion. The H_2S serves as feedstock for elemental sulfur production which improves process economics because of the by-product credit. For shales which do not contain large amounts of residual carbon and/or iron sulfides following pyrolysis, the gasification step is not plausible.

Following gasification, the remaining carbon in the shale is combusted to provide process heat. The heat of combustion is transferred to the gasification and pyrolysis zones via recirculation of the processed shale because sufficient heat cannot be transferred using gases alone. Following combustion, essentially all of the carbon and sulfur in the shale have been removed. This has important environmental implications because the potential for acid drainage by the disposed spent shale is significantly reduced when the sulfur content is low.^{9,10}

Operation of the KENTORT II process was shown to be viable at the 5-lb/hr scale during the previous cooperative agreement, but because of the small size of the unit, questions relative to scale-up remain. One of the most critical issues are the effects that scale-up will have on the extent of solid-recycle induced cracking and coking losses in the pyrolysis zone. The study of secondary coking and cracking reactions of model compounds has been initiated to more fully understand these phenomena. By having basic data which is directly applicable for the materials and conditions of the KENTORT

II system, we will have a reliable guide for the selection of operating parameters for the 50-lb/hr retort which will either maximize oil yield or improve oil quality.

The scaled-up retort provides the opportunity to conduct tests on larger quantities of oil products. One of the potentially attractive options for the heavy fraction of the shale oil is for use in asphaltic applications. Another option which deserves attention is the production of adsorbent or activated carbons from the heavy shale oil fractions. It would be a tremendous economic advantage to recover a high value product from the heavy ends without costly hydrotreating. By producing a high value material, the economy of scale for an oil shale plant would be lowered, and it would be much easier to get an industry off the ground in the near term.

OBJECTIVES

The primary objective of this program is to perform the research necessary to design, construct, test, and optimize the KENTORT II process at the 50-lb/hr scale. The tasks for the program are listed below. Efforts were directed toward Task 1.3 during the quarter.

Task 1 - Design and Construction of the KENTORT II Prototype Unit

- 1.1 - Cold-Flow Modeling of the Reactor
- 1.2 - Mini-Plant Investigation of Solids Recycle (Cracking and Coking Kinetics)
- 1.3 - Final Design and Construction of the KENTORT II Prototype Unit

Task 2 - Operation of the Prototype Unit

- 2.1 - Reactor and System Shakedown
- 2.2 - Prototype Operational Runs

Task 3 - Shale Acquisition and Product/By-Product Characterization

- 3.1 - Collection and Characterization of the Oil Shale Feedstock
- 3.2 - Characterization of the KENTORT II Materials and Products for Utilization and Environmental Characteristics
 - 3.2.1- Characterization of KENTORT II Products for Asphalt Application
 - 3.2.2- Characterization of Nitrogen and Mineralogic Transformations in the KENTORT II Process
- 3.3 - Economic Evaluations

50-LB/HR KENTORT II RETORT CONSTRUCTION UPDATE

At the close of this quarter, the construction phase of the 50-lb/hr KENTORT II process demonstration unit was nearly completed. All of the reactor components have now been installed, and the installation of downstream equipment and ancillary systems is approaching completion. The reader is directed to the previous quarterly technical report for a detailed description of the system.¹¹ At this writing, almost all of the equipment shown in the flowsheet in Figure 1 is in place. Incomplete installation of process piping and utilities, however, has delayed shakedown procedures to the next quarter.

Component Installation

J-valves. The aerated J-valves which serve to control the rate of shale recirculation from the gasifier to the pyrolyzer and combustor were modified and were installed onto the reactor in conjunction with the pneumatic liftpipes.

Combustor-Gasifier Downcomer. The downcomer which enables the flow of hot solids from the combustor to the gasifier was installed. To accommodate the differences in thermal expansion between the two zones, an expansion joint was an integral part of the apparatus. This, along with the installed J-valves, signaled the completion of the construction of the reactor itself.

Cyclones. The cyclones (and fines bins) for the overhead products from the pyrolyzer and combustor were installed.

Heat Exchanger. The combined liquid-injection/convection heat exchanger which is designed to cool the gases and vapors from the pyrolyzer to approximately 150°C was fabricated and installed.

Light Oil and Water Collection System. The system to condense and collect low boiling liquids was completed. An oil/water separating vessel was installed beneath the shell-and-tube condenser to collect the condensed light oil and water mixture. Light oil is drawn off the top periodically, and level switches automatically trigger a pump to remove the water to a holding tank before it is treated for disposal or for reuse by the steam generator. Downstream of the oil/water separator a mist cyclone, two mist filters and a gas pump were installed.

Screwfeeder. The raw shale feed system consisting of a screwfeeder, motor, speed regulator, tachometer, and Triboflow solids flow detector were designed and installed. Flexible hoses and gate valves were used to connect the screwfeeder outlet to the pyrolysis and gasification zones so that shale may be fed to either zone. The tachometer and solids flow detector have been installed to monitor the operation of the screwfeeder to ensure that a steady flow of shale is delivered to the retort.

Spent Shale Bin. As a departure from the original plan, a custom spent shale bin was designed and fabricated this quarter. The design of the bin in conjunction with a rotary airlock will permit withdrawal of spent shale from the bin during a run if necessary. It was decided that the original plan to change bins during operation was too cumbersome and potentially hazardous. The ability to continuously remove solids, combined with the continuous input of shale to the feed hopper, in theory, permits indefinite periods of operation. Limitations of the oil collection system may prohibit steady-state run times much in excess of 10 hours, however.

Utility Installation

Electrical Power. The electrical control cabinet was installed which includes the power distribution panel and the electrical heater controls for the system. The delivery of power to the individual pieces of equipment was initiated.

Instrumentation. Installation of the instrumentation system began. This includes emplacement of transducers (thermocouples, pressure transmitters, flow meters, etc.) and connection to the control system and the computerized data acquisition system.

Plumbing. All necessary process fluids (air, nitrogen, steam, and water) were connected to the retort.

Construction and Shakedown Schedule

The first priority is to prepare the system for cold-flow testing. There are six items which need to be completed in the next quarter before cold-flow tests can be performed:

- o Installation of pressure transducers.
- o Testing and calibration of the screwfeeder for raw shale.
- o Completion of exhaust gas plumbing to the ventilation system.
- o Complete the programming of the data acquisition system.
- o Connect the spent shale bin to the outlet of the cooling zone.
- o Final leak testing of the assembled system.

The objective of the cold-flow testing program will be to ensure that stable movement of shale from zone to zone, including recirculation, can be achieved at ambient temperature. Cold-flow testing will include feeding shale to the reactor at 50 lb/hr and withdrawing the shale at the same rate. These procedures will also provide training for the operators under less stressful conditions compared to hot operation. It is anticipated that these tests will be completed during the next quarter.

Following cold-flow testing, the reactor will be electrically heat-traced, where necessary, and insulated. Without shale, the system will then be heated to determine the

Schematic diagram of the 50-lb/hr KENTORT II process.

temperature distribution both in the reactor and the oil collection system. Once all of the heating systems are shown to be adequate, the system will be ready to retort oil shale. The target date for which is now set for July 1993.

Summary

The construction phase of the program has nearly been concluded. Following the completion of a number of routine tasks, the system will be ready for cold-flow testing next quarter. Consequently, heat-up procedures will then be performed. In the absence of any major setbacks, the target date for retorting oil shale is July 1993.

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