THE DEVELOPMENT OF AN INTEGRATED MULTISTAGE FLUID BED RETORTING PROCESS

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by

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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 April 1, 1993 through June 30, 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 activities for this quarter included: system leak proofing, cold flow testing, shake down of the data acquisition system, instrumentation verification, and preparation for hot operation. Once the tasks necessary for heat up are completed, shake down and operation of the Process Demonstration Unit will begin.

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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. Task 1.3 was essentially completed and Task 2.1 was initiated 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, all tasks necessary for cold flow testing were completed. Priority was given to these tasks so that cold flow testing could be completed as soon as possible. Preparation for hot operation proceeded simultaneously with the leak proofing and cold flow testing of the system. During the next quarter, items necessary for heat up will be completed and shakedown of the retort will begin.

Items completed during this quarter include:

Pressure Transducer Installation. Pressure transducers were connected to all relevant pressure taps throughout the system via stainless steel tubing. The transducers were supplied with power, and signal outputs were connected to the data acquisition system.

Screw Feeder Calibration. The screw feeder was calibrated for two different feedstock sizes. Calibration curves were generated for 8 X 60 mesh and 20 X 60 mesh shale. During calibration, the triboelectric flow sensor used to monitor flow from the feed hopper to the screw feeder was tested and found to operate satisfactorily. At the completion of the screw feeder calibration, the feed hopper was filled with 20 X 60 mesh shale for use during cold flow testing.

Exhaust Gas Plumbing. The piping network that will carry exhaust gas from the pyrolyzer and combustor to the ventilation system was installed. The pyrolysis gas exhaust line was fabricated using 2" PVC pipe. A 2" gate valve, used for controlling the pressure between the gasifier and combustor, was installed in this line. The combustor exhaust line consists of 2" stainless steel pipe. An orifice plate was designed and installed in this line as a means of allowing pressure control between the gasifier and combustor.

Data Acquisition System. The computerized data acquisition system was programmed and all instrumentation signals on the process demonstration unit were verified prior to cold flow testing.

Spent Shale Bin. The spent shale bin was mounted and the rotary valve that will control the flow of solids out of the cooling zone was mounted, powered, and tested prior to cold flow testing.

Final Leak Testing. The entire system, including upstream and downstream equipment and piping was leak tested. Some minor leaks were found in the start up burner, the electrostatic precipitator housing door, and the heat exchanger. These deficiencies were corrected.

Heat Tracing. The pyrolyzer bed, cooling zone bed, and the gasifier vessel were wrapped with high temperature heat tape. The tape will provide heat to the process during start up. It may also be used to supply additional heat to the retort during operation.

Insulation. The combustor, cooling zone, inlet steam line, combustor exhaust line and cyclone, and the pyrolyzer exhaust line and cyclone were wrapped with an alumina silicate insulation material. The remaining sections will be insulated during the next quarterly period.

Start-up Burner. All electrical connections necessary for operation of the start-up burner were completed. Also, all auxiliary equipment associated with the burner was mounted.

During the next quarterly period, the following tasks must be completed before hot operation can begin:

- o Testing of the start-up burner.
- o Complete insulation of the system.
- o Finish electrical connection of electrostatic precipitator.
- o Mount oil collection system to electrostatic precipitator.
- o Finish electrical connections for heat tape.

Once these tasks have been completed, shake down of the system will begin.

COLD-FLOW TESTING OF THE 50-LB/HR PDU

During this quarter, cold flow testing of the 50 lb/hr Process Demonstration Unit was completed. The tests were conducted using room temperature air which was supplied by a 220 CFM air blower. The feedstock was 20 X 60 mesh raw oil shale. The major objectives of the cold flow testing were to test the capacity of the recirculation loops, determine the stability of the PDU, determine the amount of fines being trapped in the combustor and pyrolyzer cyclones, and to refine the method to be used for balancing the pressure between the gasifier and combustor.

Recirculation Loops. Cold flow testing of the PDU demonstrated that the recirculation loops from the gasifier to the pyrolyzer and combustor worked well. No modifications to the J-valves or lift pipe designs were necessary. The target recirculation rates of 200 lb/hr and 500 lb/hr to the pyrolyzer and combustor respectively, were achieved.

System Stability. During 9.5 hours of cold flow testing, no incidents of unexpected system instability were experienced. For the most part, the PDU behaved very much like the full scale cold flow model even though the cooling zone of the PDU is 2" smaller in diameter, a requirement for hot operations. The resulting higher velocities in the cooling zone did not cause elutriation of the bed or bypassing of the fluidizing gas into the gasifier via the downcomer.

Fines Collection. On two separate occasions, after a period of cold flow testing, the collection bins mounted under the pyrolyzer and combustor cyclones were emptied and their contents weighed. These measurements showed that the pyrolysis cyclone was removing approximately 11% of the feed as fines. This percentage is within design limits for the system. Also, equipment down stream of the pyrolyzer cyclone was examined to determine the level of fines build up to be expected in these areas during operation. Small quantities of fines were found in the pyrolyzer heat exchanger and in the electrostatic precipitator.

Orifice Plate Design. Initial cold flow testing of the PDU utilized two 2" gate valves mounted in the exhaust lines for both the pyrolyzer and combustor as the means for controlling the pressure between the gasifier and combustor. This arrangement would not be satisfactory at operational conditions. The temperatures at the gate valve in the combustor exhaust line would require the valve to be made of exotic alloys and it was determined that an orifice plate would be substituted in its place. Once the combustor exhaust valve was adjusted properly, and the pressure difference between the gasifier and combustor could be adjusted using the pyrolyzer exhaust valve, the combustor exhaust valve was removed. An orifice plate, fabricated using 1/4" 304 stainless steel was mounted in place of the valve. By trial and error, the required orifice diameter was determined to

be 3/4". A final 4 hour cold flow test demonstrated the stability of the system with the orifice plate replacing the gate valve in the combustor exhaust line.

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