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The Extraction of Bitumen from Western Oil Sands

**Quarterly Report
April - June 1993**

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Work Performed Under Contract No.: DE-FC21-89MC26268

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July 1993

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QUARTERLY REPORT
PERIOD ENDING 30 JUNE 1993

UNIVERSITY OF UTAH
(DE-FC21-89MC26268)

Tasks are referenced to Part II - Statement of Cooperative Agreement Objectives of contract noted above.

This report cites Task number followed by brief restatement of each Task as stipulated in the contract, followed by Action this Quarter.

Task 1 Environmental Impact Statement

Action this Quarter: Prepared NEPA section of the 1993-1994 new cooperative agreement as required by US DOE.

Task 2 Coupled Fluidized Bed Bitumen Recovery and Coked Sand Combustion

Task 2.1 Conduct extended tests on at least three different Utah tar sands.

Action this Quarter: Leakage problems in the equipment occurred, but were repaired. Difficulties were experienced in feeding tar sands, but were alleviated with a new procedure.

Task 2.2 Process the liquid products from the coupled fluidized bed system by hydrotreating and determine the attractiveness of the treated products as synthetic crude oil feedstocks.

Action this Quarter: The hydrotreating equipment was moved to a new location.

Task 2.3 Develop a superior control algorithm for the reactor system to enhance controllability.

Action this Quarter: The new control strategy is under review by outside experts on controls.

Task 2.4 Produce design and economic studies for commercial scale bitumen recovery and upgrading to synthetic crude oil.

Action this Quarter: The pyrolysis model was used to study the effect of residence time and temperature. Comparison with experimental data was encouraging.

Task 3 - Water-based Recovery of Bitumen

Task 3.1 Perform specified studies of the surface chemistry as related to tar sand digestion, and bitumen separation by gravity and flotation.

Task 3.1.1 Study the effect of mineral composition on the bitumen concentrate quality.

Action this Quarter: Study of the impact of bitumen-water interfacial tension on the hot water process efficiency was continued. Microscopic observations of

bitumen release from several U.S. tar sands under stagnant conditions (the pH and ionic strength were varied by addition of electrolytes) showed that bitumen separation from mineral particles strongly depended on the bitumen-water interfacial tension. It was shown that bitumen recovery can be enhanced by appropriately controlling the bitumen-water interfacial tension.

The Circle Cliff tar sand sample was analyzed for the bitumen saturation value and mineralogical composition. The bitumen saturation value was found to be 2.8 weight percent. The mineralogical composition of the residual sand was determined to be: 44 wt% quartz, 18 wt% calcite, 11 wt% dolomite, 10 wt% kaolinite, 7 wt% K-feldspar, 4 wt% plagioclase, 3 wt% pyrite, 3 wt% illite, and 1 wt% amorphous material.

Task 3.1.2 Carry out process engineering studies related to bitumen separation from digested tar sand slurries by gravity separation and column flotation, and bitumen concentrate cleanup which will focus on characterization and utilization and the recycle of oily sludge produced in the cleaning process.

Action this Quarter: The study of air-sparged hydrocyclone flotation for the recovery of emulsified oil/bitumen from water was continued. The influence of flocculant addition (high molecular mass cationic polyelectrolyte) to an oil-in-water emulsion of various degrees of stability was investigated. The oil removal efficiency was found to correlate well with the zeta potential of the oil droplets.

Task 3.1.3 Carry out diluent penetration studies which will be aimed at the theoretical prediction of diluent penetration time, based on tar sand properties and process parameters. Determine the mechanical strength of tar sand saturated with diluent, and its impact on energy requirements during digestion.

Action this Quarter: Tar sand stagnant digestion experiments were continued, including blank experiments with model sand-oil mixtures. The behavior of the oil and gas was monitored by microscopic examination and important phenomena recorded on photographs. Regardless of tar sand origin, the mechanism of bitumen separation was found to involve two stages: a spontaneous retraction of the bitumen film from the sand surface into the sandstone pores and subsequent bitumen release from the pores due to the preferential wetting of sand by the aqueous phase.

Task 3.1.4 Investigate the chemical structure and properties of surfactants released from tar sand bitumen during water processing.

Action this Quarter: The surfactants released to the aqueous phase during Asphalt Ridge and Whiterocks tar sands digestion were submitted for MS analysis using a standard GC-MS system or direct insertion (DI) probe ion trap mass spectrometry (ITMS). The analyses showed that most fractions obtained from liquid chromatography still contained a mixture of many different compounds with a predominance of carboxylic and hydroxylic moieties. The presence of cationic surfactants cannot be excluded. These chromatographic fractions differed with respect to their aromatic nature, the length of hydrocarbon chains, and their sensitivity to oxidation/elevated temperature.

Task 3.1.5 Investigate the possibility for the recovery of bitumen by underground processing of oil sands.

Action this Quarter: A concept for underground processing is being developed to provide for diluent pretreatment, digestion and gravity separation steps. The method involves initial borehole-mining of a series of cavities which first provide the tar sand feed, later serve as the processing vessels for complete bitumen separation from sand, and finally are used to deposit the tailings. Only the bitumen concentrate is lifted to the surface for clean-up and further processing. If successful, such a process could be less expensive and would greatly lessen the environmental problems associated with

Task 4 - Rotary Kiln Process for Recovery of Bitumen and Combustion of Coke Sand

Task 4.1 Generate a comparison between the flighted and unflighted kiln barrels at a fixed set of operating conditions.

Action this Quarter: None (see previous quarterly report)

Task 4.2 Investigate the influence of process variables on the product distribution and yields and on the liquid product quality with the Asphalt Ridge tar sand with the unflighted kiln barrel.

Action this Quarter: Completed studies of residence time distribution in the solid phase using tracer materials.

Task 4.3 Generate sufficient product distribution and yield data and product quality data to compare tar sand pyrolysis in the rotary kiln and fluidized-bed pyrolysis reactors under comparable operating conditions with the Whiterocks tar sand.

Action this Quarter: Task has been completed

Task 4.4 Continue development of the process models for the rotary-kiln which incorporate plug-flow and plug-flow dispersion models for both the solid and gas phases.

Action this Quarter: Used residence time distribution data (Task 4.2) to update dispersion model of the rotary kiln.

Task 5--Recovery of Bitumen from Oil Sands Using Fluidized Bed Reactors and Combustion of Spent Sands in Transport Reactors

Task 5.1.1 Determine whether a 2-inch diameter reactor can be operated in the "pull" mode.

Action this Quarter: New reactor design (3-inch diameter) completed, materials purchased, components fabricated and construction started.

Task 5.1.2 Determine the optimum design of the distributor for the 2-inch diameter reactor and evaluate the tunability of the distributor for the control of slugging.

Action this Quarter: Design completed and distributor under construction.

Task 5.1.3 Complete the design of the 2-inch diameter reactor system based on the results of the distributor studies.

Action this Quarter: Task completed (3-inch diameter)

Task 5.1.4 Investigate the influence of process variables on the distribution, yields and product quality obtained with the Whiterocks tar sand in the new small-diameter reactor system.

Action this Quarter: None (deferred until reactor assembly completed)

Task 5.2.1 Complete preliminary process variables study of the pyrolysis of the mined ore from the Whiterocks tar sand deposit.

Action this Quarter: None (deferred until reactor assembly completed)

Task 5.2.2 Conduct scaleup comparison with the 2-inch and 6-inch diameter reactors with the mined and crushed ore from the Whiterocks tar sand deposit.

Action this Quarter: Studies in 6-inch diameter reactor complete. Small diameter reactor under construction.

Task 5.2.3 Conduct a production run in the 6-inch diameter reactor to produce bitumen-derived liquids for use in hydrotreating and hydrocracking studies.

Action this Quarter: Redesign of burner configuration completed.

Task 5.2.4 Generate spent sand samples for carbonaceous residue combustion studies.

Action this Quarter: Completed for Whiterocks oil sand

Task 5.3.1 Determine the fate of the metal constituents in the processing sequence from mining through pyrolysis of the mined ore and through combustion of the spent sand.

Action this Quarter: Completed for Whiterocks oil sand.

Task 5.3.2 Complete the analysis and characterization of the carbonaceous residues produced at various process operating conditions for a fixed-feed sand source.

Action this Quarter: Pyrolysis experiments completed. Coked sand analyses protocol being developed.

Task 5.3.3 Analyze and characterize the carbonaceous residues produced from different tar sands at a fixed set of process operating conditions.

Action this Quarter: None

Task 5.3.4 Analyze and characterize the carbonaceous residues produced for a fixed-feed sand source in different pyrolysis reactors at equivalent process operating conditions.

Action this Quarter: None

Task 5.3.5 Design and construct a portable analytical system for produced gas analysis for use with the fluidized bed, rotary kiln and riser reactors during combustion studies.

Action this Quarter: None (see previous quarterly report).

Task 5.3.6 Complete the final design and construction of the riser-combustor reactor. Initiate preliminary testing of the apparatus and the associated analytical systems.

Action this Quarter: Task completed

Task 6 -- Recovery of Bitumen from Oil Sand and Upgrading of Bitumen by Solvent Extraction

Task 6.1 Conduct a series of subcritical process studies with organic solvents and with bitumen-derived boiling range fractions produced during pyrolysis of tar sands.

Action this Quarter: None (student returned to India).

Task 6.2 Determine preliminary mass and energy balances for the solvent extraction process.

Action this Quarter: None (student returned to India).

Task 6.3 Evaluate the upgrading potential of various solvents and solvent blends at supercritical conditions.

Action this Quarter: Completed propane studies with PR Spring oil sand bitumen.

Task 6.4 Evaluate the effect of solvent type and extraction conditions on asphaltene rejection with Uinta Basin bitumen.

Action this Quarter: Asphaltene rejection studies completed with Whiterocks and PR Spring bitumen.

Task 7 -- Catalytic and Thermal Upgrading of Bitumens and Bitumen-derived Liquids

Task 7.1 Fixed bed hydrotreating of Bitumens and Bitumen-derived hydrocarbon liquids.

Task 7.1.1 Conduct a hydrotreating process variable study with the native bitumen from Whiterocks oil sand deposit using a commercial HDN catalyst.

Action this Quarter: Task completed.

Task 7.1.2 Conduct a hydrotreating process variable study with the bitumen-derived liquid from the Whiterocks oil sand deposit using a commercial HDM catalyst.

Action this Quarter: Task completed.

Task 7.1.3 Conduct a hydrotreating process variable study with the native bitumen from the Whiterocks oil sand deposit using a commercial HDM catalyst.

Action this Quarter: Task completed.

Task 7.1.4 Analyze and characterize the hydrotreated bitumen-derived hydrocarbon liquids, produced in the process variable studies. Evaluate the transportation fuel boiling range fractions distilled from the hydrotreated total liquid products.

Action this Quarter: Preliminary analyses (Sim D, elemental and metals analyses, and chemical and physical properties) completed on selected samples.

Task 7.1.5 Characterize both fresh and aged, spent hydrotreating and hydrocracking catalyst to establish regenerability and potential number of stream cycles.

Action this Quarter: Aged HDN and HDM catalyst analyses have been completed.

Task 7.2.1 Complete the hydrodynamic studies in the cold flow ebulliated bed reactor.

Action this Quarter: None

Task 7.2.2 Design the high pressure, high temperature ebulliated bed hydrotreating-hydrocracking reactor system based on the results of the cold flow reactor studies.

Action this Quarter: Task completed.

Task 7.2.3 Develop a suitable mathematical model of the ebulliated bed reactor system which is capable of predicting product yields and distributions.

Action this Quarter: None

Task 7.3 Technical and Economic Evaluation and Comparison of Bitumen Upgrading Alternatives

Task 7.3.1 Complete process simulation of the primary upgrading processes.

Action this Quarter:

1. The heart of the process simulation is the kinetic or extent-of-conversion models. Because all experimental work has been discontinued completion of the modeling has required estimates based on literature results.
2. The visbreaking kinetic model of Castellós (OGJ, 76, 1991) has been modified.
3. The visbreaking simulation approach of Washimi (Hyd. Proc. 69, 1989) was modified and completed.
4. The hydrolysis kinetic model of Ryu (Ph. D. Thesis, 1989) was modified to a five-component system to retain consistency with other kinetic models.
5. Simulation of HP without heat recovery was completed. Simulation with heat recovery is in progress.

6. Simulation of the HT has incorporated the hydrogen consumption data of Brechtel (M. S. Thesis, 1981).

Task 7.3.2 Provide direction re pilot plant program. Candidate processes will be selected for further study. The basis for the selection will be documented, making note of any significant differences in product yield and quality. A narrow list of process candidates will be subjected to profitability analysis, varying the important economic variables. The variables include, but are not limited to bitumen cost and product price, process costs, process scale and capital costs, and transportation and marketing costs.

Action this Quarter:

1. The comparative analysis has been further broken down to a weighted criteria (in addition to the economic analysis). The criteria are ranked in terms of order of increasing risk. Categories are commercially practiced, demonstrated (semi-works) pilot plant (continuous pdu), batch or semi-continuous bench scale, exploratory, and conceptual. The criteria are applied to the general state of technology development and the state of specific development on bitumen. The criteria values may be used as a coefficient to adjust projected economics of less mature technology to conventional economics of more mature technology.
2. Cost analysis based on simulated results is continuing.

The student working on these tasks is beginning his dissertation preparation. Completion of the dissertation and resulting work is expected before the end of 1993.

Task 8 -- Evaluation of Utah's Major Oil Sand Deposits for the Production of Asphalt, High Energy Jet Fuels and Other Specialty Products

Task 8.1.1 Complete the design and construction of a high pressure, high temperature hydrotreating process screening unit having a minimum catalyst loading of 10 cubic centimeters. This small-scale unit will be designed for single-pass hydrogen and single-pass liquid flow.

Action this Quarter: Design completed.

Task 8.1.2 Complete the design and construct a packed distillation column capable of fractionating bitumen, bitumen-derived liquids and product streams produced by upgrading and processing bitumens and bitumen-derived liquids.

Action this Quarter: Task completed.

Task 8.1.3 Complete the preparation of a series of asphalt base stocks from the PR Spring, Sunnyside and Hill Creek bitumens for evaluation by the Utah Department of Transportation for road asphalt potential.

Action this Quarter: PR Spring bitumen has been extracted and distillation has been initiated.

Task 8.2 Characterizaion of the Native Bitumens and Reservoir Rocks from the Uinta Basin Tar Sand Deposits

Task 8.2.1 Complete the characterization and analysis of the Asphalt Ridge bitumen samples.

Action this Quarter: Standard chemical and physical property determinations have been completed.

Task 9 -- Development of Mathematical Models for Bitumen Recovery and Processing

Task 9.1 Rotary-kiln pyrolysis process model integrated with kinetic models from fluid-bed studies.

Action this Quarter: Solid dispersion model of rotary kiln updated to include solids residence time distribution data.

Task 9.2 Supercritical Fluid Extraction - Multicomponent, multiphase thermodynamic models will be used to validate experimental data.

Action this Quarter: Modeling with Peng-Robinson equation-of-state completed.

Task 9.3 Bitumen Hydrotreating in 3-phase Ebulliated Beds - Develop a laboratory hydrodynamic model reactor that replicates the conditions in the commercial reactor.

Action this Quarter: Task completed.

Task 9.4 Bitumen Hydrotreating - Initiate research on process models in packed or trickle beds.

Action this Quarter: Amoco resid conversion reaction scheme for residual fractions successfully applied to bitumen hydrotreating.

Task 9.5 Coke Combustion - Reexamine models already developed to determine the regime of experimental operation: whether intrinsic kinetics or mass transport controlled.

Action this Quarter: None

Task 10 -- Completion of the Cost Estimation Study of the Pilot Plant Restoration

Task 10.1 Complete cost estimates for 100-ton per day operation.

Action this Quarter: None

Task 11 -- Development Studies of Equipment for Three-Product Gravity Separation of Bitumen and Sand

Action this Quarter: Study completed

Task 11.1 Design a classifier for the pilot plant to process at least 100 tons/day of tar sand.

Action this Quarter: After completing task 11, we are now designing this unit. Should be completed August 31, 1993

Task 11.2 Determine if aeration of the feed would increase bitumen recovery.

Action this Quarter: Cannot do this until the pilot plant is completed and installed.

Task 11.3 Determine the probable separation of solids by particle size.

Action this Quarter: Hope to install pilot plant by October 31, 1993. Can then make the determinations.

Task 11.4 Summarize the solids split by making plots of particle-size split between overflow and underflow as a function of size distribution in tar sands from various tar sands sources in Utah.

Action this Quarter: Cannot complete this until Autumn Quarter at earliest. Depends on the installation of the pilot plant.

Task 11.5 Re the Centrifugal force unit, compare probable capital and operating costs of the present three-product classifiers as employed at Suncor and Syncrude in Canada and the centrifugal unit.

Action this Quarter: Will need minimum time of Autumn Quarter to complete pilot plant and run. Can complete in Winter Quarter if pilot plant is installed by the end of the summer.

Task 12 -- Development Studies of Disposal of Sand by Conveying or Pumping of High Solids Concentration Sand-Water Slurries

Task 12.1 Determine thickener requirements.

Action this Quarter: If pilot plant is installed by October 31, we can start this work in Winter Quarter and complete in Spring Quarter by May 31, 1994.

Task 12.2 Predict performance of the primary classifier re particle size. Study bitumen recovery with air added to classifier feed.

Action this Quarter: Cannot start this until Winter Quarter. If we can start by Jan. 1994, we can expect completion by May 1994.

Task 12.3 Complete studies on secondary clarification and moisture content prediction as it affects conveying or pumping.

Action this Quarter: Expect to start this task in Winter Quarter and complete by end of Spring Quarter.

Task 12.4 Carry out bench-scale studies on flocculation and thickening of the fine solids. Prepare specifications for a conventional thickener and estimate solids content.

Action this Quarter: Expect to start this work during Spring Quarter and complete by June 30, 1994.

Task 12.5 Working with super thickener equipment manufacturers devise and carry

out bench scale studies to determine the possibilities of dramatically increasing solids concentration.

Action this Quarter: Plan to build the necessary equipment during Winter Quarter and complete by June 30, 1993.

Task 12.6 If high concentration products are obtained in the 12.5 studies determine the possibilities for conveying and pumping this material.

Action this Quarter: Plan to initiate this work in Spring Quarter and complete by June 30, 1994

Task 12.7 Prepare specifications for a three product classifier for the pilot plant. Outline assembly, installation, and evaluation procedures.

Action this Quarter: Will complete this in Summer Quarter 1994.

Task 13 -- Environmental Studies of the North Salt Lake Pilot Plant Rehabilitation and Eventual Operation and those Environmental Problems Associated with Eventual Commercial Products

Task 13.1 Evaluate and develop a plan for the environmental remediation needed prior to refurbishing the pilot plant.

Action this Quarter: None (awaiting award of State funding).

Task 13.2 Prepare environmental impact assessment of pilot plant operation using material and energy balance flowsheets.

Action this Quarter: None (awaiting award of State funding).

Task 13.3 Develop detailed flowsheets for various production and upgrading strategies to assess the impact of a commercial development.

Action this Quarter: Completed pyrolysis-hydrotreating upgrading sequence evaluation.

Task 13.4 Assemble all pertinent federal, state and local environmental rules and regulations related to tar sand development.

Action this Quarter: Assembly of documents continued.

Task 13.5 Assemble and review all environmental impact statements that have been prepared for proposed tar sand projects in the United States with particular reference to Uinta Basin tar sand deposits.

Action this Quarter: Sunnyside, Circle Cliffs, and Tar Sand Triangle EIS's reviewed.

Task 13.6 Identify all potential environmental concerns associated with each of the possible surface recovery schemes.

Action this Quarter: Surface recovery schemes narrowed to two probable candidates: thermal and aqueous recovery.

Task 13.7 Assemble all pertinent tests and sampling protocols related to identified environmental concerns for tar sand development.

Action this Quarter: Assembly of documents continued.

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