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The Extraction of Bitumen from Western Oil Sands

**Quarterly Report
July - September 1993**

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By
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**QUARTERLY REPORT
PERIOD ENDING 30 SEPTEMBER 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 No. followed by brief restatement of each Task as stipulated in the contract, followed by Action this Quarter.

Task 1 NEPA Environmental Information Statement

Action this quarter: The draft report related to the environmental information required by the National Environmental Policy Act was prepared for submission to 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: A 1.5-hour shakedown test was performed with Whiterocks tar sand mixed with clean sand to check a new feeding procedure designed to avoid plugging of the feeder and the reactor. No plugging occurred when using the hollow feeding auger.

Task 2.2 Process the liquid products from the coupled fluidized bed system by hydrotreating

Action this quarter: An experimental hydrotreating apparatus used in a previous study was moved into the laboratory, but needs to be assembled.

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

Action this quarter: Theoretical development of an improved reactor control algorithm was completed and tested successfully on clean sand.

Task 2.4 Using the extensive data available, begin design economic studies (Commercial Scale Bitumen Recovery)

Action this quarter: A mathematical model for the pyrolysis reactor, which includes a new pyrolysis kinetic model, was completed and compared with good results to previously obtained experimental data.

Task 3 Water-based Recovery of Bitumen

Task 3.1 Fundamental research - continue studies of the surface chemistry as related to water-based recovery

Action this quarter: The surface tension values for toluene-extracted bitumens from Whiterocks, Sunnyside, PR Spring, Asphalt Ridge, and Circle Cliffs oil sands, were measured with the Wilhelmy plate technique and found to vary from 20.6 mNm^{-1} to 31.2 mNm^{-1} at 333 K. Also, the surface tensions of these bitumens at room temperature were calculated from contact angle measurements using Neumann's equation-of-state. Satisfactory agreement between the results for bitumen surface tensions as determined from contact angle data and direct surface tension measurements with the Wilhelmy plate technique were obtained for the Whiterocks, Sunnyside, and PR Spring bitumens. Larger discrepancies between the methods, $6.4\text{--}9.4 \text{ mNm}^{-1}$, were observed for the other two bitumens.

Task 3.2 Investigate the effect of mineral composition on bitumen concentrate

Action this quarter: In the continuation of resource characterization, studies were extended to one of the major oil sand deposits, viz., PR Springs. Bitumen was extracted in several batches from a freshly collected PR Spring oil sand, and the average bitumen saturation value was determined. After extraction of bitumen, a representative sample of the bitumen-free sand was ground to $<45 \mu\text{m}$ size, and mineralogical composition was determined by X-ray diffraction. The composition of the sand was found to be: quartz (50 wt%), plagioclase (18 wt%), K-feldspar (18 wt%), dolomite (3 wt%), calcite (3 wt%), pyrite (3 wt%), and mica and illite (3 wt%). The characterization of the extracted bitumen sample is in progress.

Task 3.3 Conduct process engineering studies related to bitumen separation by gravity separation and column flotation.

Action this quarter: Complete bitumen disengagement and displacement from oil sand during digestion is the determining but not sufficient condition for satisfactory bitumen recovery in the hot water process. The bitumen concentrate needs to contain an appropriate amount of gas to make it float. In this regard, the interaction of gas bubbles with bitumen during digestion were investigated. In preliminary tests carried out with a Denver-type flotation cell, an improved efficiency of bitumen recovery from Whiterocks oil sands was observed when the oil sand pulp was intentionally aerated by air sparging during digestion in addition to the natural aeration which occurs during vigorous agitation.

Task 3.4 Conduct diluent-oil sand penetration studies.

Action this quarter: Literature review aimed at 2D computer simulation of liquid in a capillary has been initiated.

Task 3.5 Investigate the chemical structure and properties of natural surfactants released from bitumen.

Action this quarter: Mass spectroscopy analysis of surfactants released to the aqueous phase during oil sand digestion using a standard GC-MS system and ion trap mass spectrometry were continued. Also, saturation of process water with surfactants was investigated during the hot water processing of the Whiterocks oil sands by examination of the surface tension of the aqueous phase. It was found that the aqueous phase becomes saturated with organic species extracted from bitumen after 5-7 cycles of water recycling and the surface tension of aqueous phase remains constant at a level of 52-55 mN⁻¹.

Task 3.6 Investigate underground recovery of oil sands.

Action this quarter: The concept of oil sand underground processing, resulting in complete bitumen separation from sand and transportation of the bitumen concentrate to the surface, is under further development. The behavior of the bitumen and the gas phase contained in the oil sand sample were monitored under a microscope during stagnant digestion experiments. Detailed measurements of bitumen and gas release were continued for an extended period of time. New Asphalt Ridge oil sand samples were prepared for mechanical strength measurements prior to and after treatment in an alkaline solution.

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.

Action this quarter: Task delayed in favor of additional unflighted kiln studies which were required to determine the relationship between solids mixing, residence time, and solids distribution (radial and axial) in the rotary kiln.

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

Action this quarter: PR Spring oil sands ore was substituted for the Asphalt Ridge oil sand due to feeding characteristics. Feeder problem delayed this work; however, the feeder has been modified and it is now possible to continuously feed a high saturation oil sand. The process studies have been initiated.

Task 4.3 Generate product distribution and yield data and product quality data to compare rotary and fluidized-bed tar sand pyrolysis reactors.

Action this quarter: Task has essentially been completed.

Task 4.4 Continue development of the process models for the rotary-kiln.

Action this quarter: Process model now includes "Turnover Number" concept. Residence time distribution studies have been conducted at ambient conditions. Studies have been conducted which indicated that there may be a significant "residence" time delay of the fresh oil sand feed in the inlet region of the kiln barrel.

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: Reactor design complete. Materials for construction ordered. After thorough design analysis 2" I.D. changed to 3" I.D.

Task 5.1.2 Determine the optimum design of the distributor for the 3-inch diameter reactor.

Action this quarter: Distributor design completed.

Task 5.1.3 Complete the design of the 3-inch diameter reactor system.

Action this quarter: The design of the 3-inch diameter reactor has been completed.

Task 5.1.4 Investigate the influence of process variables on the distribution.

Action this quarter: Small-diameter reactor is under construction. Process studies will commence after the reactor has been commissioned.

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: Preliminary process studies with Whiterocks oil sand will be conducted after reactor system is brought on-line.

Task 5.2.2. Conduct scaleup comparison with the 3-inch and 6-inch diameter reactors.

Action this quarter: see Task 5.1.4. Comparison of performance of Modified L-valves has begun.

Task 5.2.3 Conduct a production run in the 6-inch diameter reactor & produce feedstock for hydrotreating and hydrocracking studies.

Action this quarter: Redesign of heating system and temperature control for 6" I.D. reactor completed. Fabrication of propane burners initiated.

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

Action this quarter: Coked sand samples will be generated after both the 3-inch and 6-inch diameter reactors have been brought on-line.

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

Action this quarter: Initial testing of combustion reactor has been completed.

Task 5.3.2 Complete the analysis of the carbonaceous residues produced at various process operating conditions.

Action this quarter: Carbonaceous residues from pyrolysis of Whiterocks oil sands showed no variation in properties based on Pyrolysis temperature or residence time in the 6" fluidized bed reactor.

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: Pyrolysis studies P.R. springs oil sands in the rotary kiln were delayed due to feeder problems. The feeder problem has been corrected and the analysis of spent sands will be conducted following completion of process studies.

Task 5.3.4 Analyze carbonaceous residues produced for a fixed-feed sand source in different pyrolysis reactors.

Action this quarter: For Whiterocks oil sands, the wt% extractables and wt% combustibles on the spent sand was found to be greater for rotary kiln pyrolysis than for fluidized bed pyrolysis. Analysis of the non-extractable carbonaceous residue has been initiated.

Task 5.3.5 Design and construct a portable analytical system for produced gas analysis.

Action this quarter: Due to the high cost of this system as originally designed, a second effort was made to find less costly alternatives. Essential product information has been collected from suppliers and a redesign has been started.

Task 5.3.6 Complete the final design and construction of the riser-combustor reactor.

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.

Action this quarter: An appropriate subcritical extraction system design is in the planning/review stage.

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

Action this quarter: Subcritical: delayed pending construction of new apparatus. Supercritical: task completed.

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

Action this quarter: Task completed for CO₂ and propane at supercritical extraction conditions.

Task 6.4 Evaluate the effect of solvent type and extraction conditions on asphaltene rejection.

Action this quarter: Task completed for CO₂ and propane with the Whiterocks and PR Spring bitumens.

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

Task 7.1 Fixed Bed Hydrotreating of Bitumen and Bitumen-Derived Liquids

Task 7.1.1 Conduct hydrotreating process variable study with Whiterocks bitumen using a commercial HDN catalyst.

Action this quarter: The process variable study (temperature, pressure and space velocity) with the HDN catalyst has been completed.

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

Action this quarter: The bitumen-derived liquid has not yet been produced so this task has been postponed.

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

Action this quarter: The process variable study (temperature, pressure and space velocity) with the HDM catalyst has been completed.

Task 7.1.4 Characterize both fresh & used hydroprocessing catalysis used in the upgrading studies.

Action this quarter: The activity with respect to hydrogenation, hydrodenitrogenation, hydrodesulfurization and hydrodeoxygenation has been determined for fresh and deactivated HDN catalyst. The catalyst was deactivated by the Whiterocks bitumen in the process studies.

Task 7.2 Ebulliated-Bed Hydrotreating of Bitumen and Bitumen-Derived Liquids

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

Action this quarter: Work has been finished. Hydrodynamics data from literature was determined to be adequate for preliminary design purposes.

Task 7.2.2 Design the high pressure, high temperature ebulliated bed hydrotreating-hydrocracking reactor.

Action this quarter: The design of the laboratory scale ebulliated- bed reactor has been completed.

Task 7.2.3 Develop a mathematical model of the ebulliated bed reactor system.

Action this quarter: A hydrodynamic-kinetic model of ebulliated bed hydrotreating of bitumen has been partially developed.

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

Task 7.3.1 Complete process simulation of the primary process coking, visbreaking, catalytic cracking, hydropyrolysis.

Action this quarter: BASECASES for process simulation of coking, visbreaking, catalytic cracking, catalytic hydrotreating and hydropyrolysis have been completed. Process optimization is being conducted in concert with economic analysis.

Task 7.3.2 Provide direction re: pilot plant program. Candidate processes will be selected for further study.

Action this quarter: Initial profitability analysis for coking, visbreaking, catalytic cracking and hydropyrolysis has been conducted. Sensitivity to feedstock (bitumen) cost is used to address variability in upstream costs. Preliminary results are being reviewed for realism.

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 design and construction of high-pressure and high-temperature hydrotreating process screening unit.

Action this quarter: The design has been essentially completed and components have been ordered.

Task 8.1.2 Complete the design and construction of a high packed distillation column.

Action this quarter: The design and construction of the packed distillation column has been completed. A vacuum control device has been ordered.

Task 8.1.3 Complete the preparation of a series of asphalt base stocks for testing by UDOT.

Action this quarter: The completion of the distillation column will permit preparation of asphalt samples from PR Spring , Asphalt Ridge, Sunnyside and Whiterocks.

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

Action this quarter: The GC-MS data for the Asphalt Ridge bitumen is being analyzed and interpreted.

Task 9 Development of Mathematical Models for Bitumen Recovery and Processing

Task 9.1 Integrate available kinetic models of fluid-bed pyrolysis with model of rotary kiln pyrolysis.

Action this quarter: Experimental residence time distribution studies have been completed in the rotary kiln and have been initiated in the fluidized beds.

Task 9.2 Modify model for supercritical fluid extraction process. Adapt available models for supercritical fluid extraction to accommodate treating bitumen as a mixture of several lumped compounds.

Action this quarter: SFE thermodynamics models have been developed and tested with experimental SFE data for the Whiterocks and PR Spring bitumens.

Task 9.3 Develop a model for hydroprocessing of bitumen in a 3-phase ebulliated bed.

Action this quarter: The hydrodynamic design equations have been developed via similitude analysis. Kinetics have not yet been integrated into the design equations.

Task 9.4 Initiate the development of models for bitumen hydroprocessing in packed or trickle beds.

Action this quarter: The AMOCO resid conversion reaction network model has been successfully applied to bitumen resid conversion for the Whiterocks bitumen over both HDN and HDM catalysts.

Task 9.5 Develop a model for coke combustion in a transport reactor.

Action this quarter: TGA coke combustion kinetics will be integrated into the equations for the transport reactor. Residence time distribution and flow transition studies have been completed.

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

Task 10.1 Prepare a cost estimate for rehabilitation of the North Salt Lake Pilot Plant.

Action this quarter: The preliminary cost estimate has been completed in consultation with engineering staff from Ford, Bacon and Davis, Utah. Utility services (telephone, electrical, water and natural gas) have been restored to the North Salt Lake pilot plant facility.

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

Action this quarter:

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

Action this quarter: The classifier design has been completed.

Task 11.2 Conduct studies on feed aeration to increase bitumen recovery.

Action this quarter: This task will be conducted when the equipment has been constructed at the North Salt Lake Pilot plant.

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

Action this quarter: This task will be conducted when the equipment has been constructed at the North Salt Lake Pilot plant.

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

Action this quarter: This task will be conducted when the equipment has been constructed at the North Salt Lake Pilot plant.

Task 11.5 A study will be made of probable capital & operating costs of three-product classifiers employed at Suncor & Syncrude plants in Canada.

Action this quarter: This task will be conducted when the equipment has been constructed at the North Salt Lake Pilot plant.

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 for the pilot plant.

Action this quarter: This task will require samples produced during the experiments at the North Salt Lake Pilot Plant.

Task 12.2 Carryout studies to enable prediction of the performance of the primary classifier with respect to particle size.

Action this quarter: The task will be conducted as part of the classifier studies at the North Salt Lake Pilot Plant.

Task 12.3 Complete studies on secondary clarification and moisture content and handleability for conveying or pumping the product.

Action this quarter: This task will require samples produced during the experiments at the North Salt Lake Pilot Plant.

Task 12.4 Carry-out bench-scale studies on flocculation and thickening of the fine solids products.

Action this quarter: The task will be conducted as part of the classifier studies at the North Salt Lake Pilot Plant.

Task 12.5 Carry out bench-scale studies to determine the possibility of increasing solids concentration to toothpaste level.

Action this quarter: The task will be conducted as part of the classifier studies at the North Salt Lake Pilot Plant.

Task 12.6 Determine the possibilities for conveying & pumping the concentrate if Task 12.5 is successful.

Action this quarter: The task will be conducted as part of the classifier studies at the North Salt Lake Pilot Plant.

Task 12.7 Prepare specifications for a 3-product classifier for the pilot plant and outline plans for assembling and installing the equipment and testing it.

Action this quarter: This task has been completed.

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 North Salt Lake pilot plant.

Action this quarter: The site report prepared by the State agency responsible for hazardous waste sites has been obtained and is under review. The conclusions were that no remediation was required.

Task 13.2 Conduct an environmental impact evaluation of pilot plant operation.

Action this quarter: This task awaits the acquisition of funding from the State Of Utah.

Task 13.3 Prepare an assessment of commercial development of Utah's tar sands.

Action this quarter: This task awaits the acquisition of funding from the State of Utah.

Task 13.4 Assemble pertinent federal, state and local environmental rules and regulations pertaining to the commercial tar sand development.

Action this quarter: The environmental rules and regulations are being reviewed.

Task 13.5 Assemble and review all available environmental impact information for U.S. tar sand projects with particular emphasis on Uinta Basin deposits.

Action this quarter: Environmental Impact Assessments for the Sunnyside and Circle Cliffs oil sand projects have been acquired.

Task 13.6 Identify potential environmental concerns associated with possible surface recovery schemes.

Action this quarter: This task awaits the acquisition of funding from the State of Utah.

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

Action this quarter: Water and air pollution information has been obtained under State funded project.

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