Third Annual Oil Shale, Tar Sand and Mild Gasification Contractors Review Meeting

Agenda, Project Synopses, List of Participants

Held at
Sheraton Lakeview Conference Center
Morgantown, West Virginia 26505

Sponsored by
U.S. Department of Energy
Office of Fossil Energy
Morgantown Energy Technology Center
P.O. Box 880
Morgantown, West Virginia 26507-0880

July 19-21, 1988
AGENDA

TUESDAY, JULY 19, 1988

00 a.m.  Registration, Ballroom Foyer

00 a.m  Speakers Briefing Breakfast

30 a.m  Morning Coffee and Danish

00 a.m  Introduction
    Dr. Theodore C. Bartke, Chief,
    Laramie Branch, METC

9:15 a.m.  Welcome
    Dr. John Wilson, Deputy Director,
    METC

9:30 a.m.  Oil Shale Prospects
    Dr. Raymond L. Zahradnik, President,
    Occidental Oil Shale, Inc.

10:00 a.m.  Oil Shale Program Overview
    Mr. Carl E. Roosmagi,
    Oil Shale Project Manager
    Laramie Branch, METC

10:30 a.m.  Mild Gasification Program Overview
    Dr. Madhav R. Ghate, Chief,
    Gasification and Combustion Branch, METC

11:00 a.m.  Tar Sand Program Overview
    Dr. T. Robert McLendon,
    Tar Sand Project Manager
    Laramie Branch, METC

LUNCH

Oil Shale I
SESSION 1A
Oil Shale Properties and Behavior

Chairperson:  Carl E. Roosmagi
1:00-5:00 p.m.

1A.1 Oil Shale Reference Sample
Bank
    Dr. Lawrence B. Owen
    Terra Tek, Inc.

Mild Gasification
SESSION 1B

Chairperson:  Dr. Madhav R. Ghate
1:00-5:00 p.m.

1B.1 Mild Gasification: Product
Characterization
    Dr. Ron L. Graves
    Martin Marietta Energy Systems
    Oak Ridge National Laboratory
1A.2 Thermal Decomposition of DOE Reference Shales
Dr. Francis P. Miknis
Western Research Institute

1A.3 Rapid Pyrolysis Mechanisms and Product Characterization
Dr. Lawrence J. Shadle
METC

1A.4 Improved Analytical Methods and Measurements of Rapid Pyrolysis Kinetics for Western and Eastern Oil Shale
Dr. Robert W. Taylor
Lawrence Livermore National Laboratory

1A.5 Pyrolysis Modeling of Oil Shale
Mr. Scott M. Klara
METC

1A.6 Water Generation During Pyrolysis of Oil Shales
Dr. Thomas T. Coburn
Lawrence Livermore National Laboratory

1A.7 Rate of Cracking or Degradation of Oil Vapor in Contact with Oxidized Shale
Dr. Robert W. Taylor
Lawrence Livermore National Laboratory

IB.2 Evaluation of Coal-Derived Liquids for Locomotive Engine
Dr. Richard A. Wolfe
United Coal Company Research Corporation

IB.3 Upgrading of Condensibles from Coal Devolatilization
Dr. John E. Mrochek
Oak Ridge National Laboratory

IB.4 Advanced Fuels Research
Mr. E.B. Smith
Western Research Institute

IB.5 Product and Precursor Properties of Condensable Hydrocarbons
Dr. Peter A. Hesbach
METC
Oil Shale II
SESSION 2A
Oil Shale Surface Processes, R&D

Chairperson: Catherine E. Gregoire
8:30 a.m.–12:00 p.m.

2A.1 Recent Progress in Solid-Recycle Retorting and Related Laboratory and Modeling Studies
Dr. Robert Cena
Lawrence Livermore National Laboratory

2A.2 KENTORT II Process Development
Mr. Scott D. Carter
Kentucky Energy Cabinet Laboratory

2A.3 Surface Retorting Studies
Dr. Chang Yul Cha
Western Research Institute

2A.4 Research in Process Development for Hydroretorting of Eastern Oil Shales
Mr. Raymond C. Rex, Jr.
Hycrude Corporation

BREAK

2A.5 Beneficiation-Hydroretort Processing of U.S. Oil Shales - Project Review and Conclusions
Mr. Carl Rampacek
University of Alabama Mineral Resources Institute

2A.6 Pressurized Fluidized-Bed Hydroretorting of Eastern Oil Shales
Mr. Dharam V. Punwani
Institute of Gas Technology

2A.7 Conceptual Design of Commercial Oil Shale Plants
Mr. Roger J. Gaire
Foster Wheeler USA Corporation

2A.8 Comparison of Western and Eastern Oil Shale Conversion in a Staged Fluid Bed System
Ms. Catherine E. Gregoire
METC

LUNCH

Tar Sand I
SESSION 2B

Chairperson: T. Robert McLendon
8:30 a.m.–12:00 p.m.

2B.1 Property Determination and Reference Resource Selection
Dr. Kenneth P. Thomas
Western Research Institute

2B.2 Fundamental Chemistry and Physics of Recovery Processes
Dr. Kenneth P. Thomas
Western Research Institute

2B.3 The Effect of Recovery Methods on Bitumen and Heavy Oil Composition
Dr. Kenneth P. Thomas
Western Research Institute

2B.4 Recycle Oil Pyrolysis and Extraction Process
Dr. Chang Yul Cha
Western Research Institute

2B.5 Fuels Precursors and Product End Use Potential
Mr. E.B. Smith
Western Research Institute

2B.6 Effluent Characterization of Tar Sand Laboratory Recovery Processes
Dr. John Nordin
Western Research Institute

LUNCH
Oil Shale III
SESSION 3A
Mining, Material Handling and
In-Situ Processes

Chairperson: Carl E. Roosmagi
1:30-5:00 p.m.

3A.1 Oil Shale Mining and Material
Handling Technology Assessment
Dr. Carroll F. Knutson
EG&G Idaho, Inc.

3A.2 Technology Development for
Blasting Based Mining Operations
Dr. Paul J. Hommert
Sandia National Laboratories

3A.3 Evaluation of Water-Jet-Assist
Mechanical Cutting of Oil Shale
Mr. Wilhelm J. Kogelmann
Alpine Equipment Corporation

3A.4 In Situ Retorting Studies
Mr. Norman Merriam
Western Research Institute

3A.5 Retorting Using Electromagnetic
Energy
Professor Ramarao Inguva
University of Wyoming

3A.6 An In-Situ Process to Recover
Hydrocarbons from Eastern Shales,
Results of Horizontal Retort
Preparation
Mr. Victor Carr
Eastern Shale Research
Corporation

Tar Sand II
SESSION 3B

Chairperson: T. Robert McLendon
1:00-5:00 p.m.

3B.1 Modified Hot Water Separation
Technology
Dr. Jan D. Miller
University of Utah

3B.2 Dual Fluidized Bed Pyrolysis
and Combustion
Dr. Francis V. Hanson
University of Utah

3B.3 Heat Pipe Pyrolysis and
Combustion
Dr. John D. Seader
University of Utah

3B.4 Tar Sand Bitumen Upgrading
Dr. James W. Bunger
University of Utah

3B.5 Rotary Kiln Pyrolysis of
Bitumen-Impregnated Sandstone
from the Tar Sand Deposits
of Utah
Dr. Francis V. Hanson
University of Utah

3B.6 USBM Resource Assessment in Utah
Dr. James W. Bunger
University of Utah

3B.7 Solvent Extraction of Southern
U.S. Tar Sands
Dr. R.E. Babcock
University of Arkansas

THURSDAY, JULY 21, 1988

7:30 a.m. Registration, Ballroom Foyer

8:00 a.m. Morning Coffee and Danish
Oil Shale IV
SESSION 4A
Environmental R&D

Chairperson: Carl E. Roosmagi
8:30 a.m-12:00 p.m.

4A.1 Industry, University and Government Cooperative Research to Evaluate Stability and Behavior of Processed Oil Shale Waste Embankments
Professor Quentin D. Skinner
University of Wyoming

4A.2 Field and Laboratory Leaching Studies of Retorted Kentucky Oil Shale
Dr. Thomas L. Robl
Kentucky Energy Cabinet Laboratory

4A.3 Organic Geochemical Characterization of Solid Waste
Dr. John M. Bowen
Western Research Institute

4A.4 Inorganic Geochemical Characterization of Solid Waste
Dr. Michael E. Essington
Western Research Institute

BREAK

4A.5 Evaporation Process Analyses and Emissions from Oil Shale Retort Waters
Dr. Tim L. Reeves
University of Wyoming

4A.6 Groundwater Studies at Rio Blanco
Ms. Florence Barker
Western Research Institute

4A.7 Oil Shale Plant Siting Methodology
Dr. John Nordin
Western Research Institute
Oil Shale I
SESSION 1A
Oil Shale Properties and Behavior

Chairperson: Carl E. Roosmagi
ABSTRACT

A National Oil Shale Reference Sample Bank (OSSB) has been established to provide DOE researchers with well characterized and well mixed material of uniform particle size for use in experimental programs. Availability of carefully documented and homogeneous samples will enable investigators to more accurately compare experimental results especially in the case of comparative studies of process technology. The sample bank also provides a more convenient way for researchers to quickly obtain materials for laboratory programs at no cost to the program. Samples are distributed primarily to DOE contractors. Non-DOE contractors may obtain reference samples pending DOE approval.

A primary objective of the OSSB program is to obtain bulk Oil shale samples from locals in the Eastern and Western United States. Terra Tek's responsibilities include identification of potential bulk sampling sites, verification of oil yield, bulk sampling, shale processing, distribution of reference shales to DOE contractors and maintenance of the shale inventory for the duration of the program.

Oil yield is the primary criterion for bulk sampling site selection. Eastern U.S. shales must have a minimum Fischer assay of 10 gpt. Western oil shales must satisfy a requirement of 20 gpt minimum oil yield. Bulk sampling involves removal and transport of up to 24 tons of material from outcrops or mines. Material is recovery as appropriate for a specific site and then transported by truck to a crushing site in Utah. Shale processing consists of crushing bulk samples to a uniform size
between 3/8 and 3/4 of an inch. Finer material is rejected. The crushed shale is homogenized in a large capacity rotary mixer. Samples for distribution are packaged in 55-gallon and 5-gallon capacity drums under an inert argon gas blanket. Approximately 10 tons of processed material from each site is stored in drums.

During shale processing, ten sample splits are recovered for analysis to document bulk sample homogeneity and sample characteristics. A comprehensive report describing shale sampling, processing and results of shale characterization is provided to each reference shale sample recipient. The analytical program generates the following data set for each bulk sample:

<table>
<thead>
<tr>
<th>NUM. OF ANALYSES</th>
<th>DESCRIPTION</th>
<th>NUM. OF ANALYSES</th>
<th>DESCRIPTION</th>
</tr>
</thead>
<tbody>
<tr>
<td>10</td>
<td>FISCHER ASSAY</td>
<td>5</td>
<td>HYDROGEN</td>
</tr>
<tr>
<td>5</td>
<td>MASS BALANCE ASSAY</td>
<td>5</td>
<td>NITROGEN</td>
</tr>
<tr>
<td>5</td>
<td>-60 MESH MOISTURE</td>
<td>5</td>
<td>TOTAL SULFUR</td>
</tr>
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<td>TOTAL CARBON</td>
<td>5</td>
<td>ORGANIC SULFUR</td>
</tr>
<tr>
<td>5</td>
<td>MINERAL CARBON</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

ACCOMPLISHMENTS

The current reference shale inventory includes shales from the EXXON Colony Mine, Parachute, Colorado (average oil yield =27.3 gpt), a Tipton oil shale from an outcrop near Rock Springs, Wyoming (average oil yield =22.8 gpt) and a New Albany (Clegg Creek Member) shale from a quarry near Louisville, Kentucky (average oil yield =14.9 gpt). A new Eastern shale will be acquired from an outcrop of New Albany Shale in Southern Indiana during the fourth quarter of 1988. The inactive White River Oil Shale Mine in Eastern Utah is a potential source for the next Western reference shale.

The shale inventory is repurged with nitrogen and then argon gas every four months. Samples in storage include one 55-gallon drum of shale from each sampling site which were not blanketed with inert gas. These samples are resubmitted for mass balance assays once per year. The data are used to assess the potential for shale degradation in storage. Data for the first year of storage indicate negligible alteration of two non-purged samples suggesting that the purged samples are adequately protected.
ABSTRACT

The objective of this research effort is to explore the chemistry of kerogen thermal decomposition in relationship to kerogen structure. Since the kerogen in oil shales exists in a variety of structural assemblages, understanding the chemistry of kerogen pyrolysis requires investigations of the pyrolysis kinetics of several different types of kerogens. Knowing the chemistry of the pyrolysis of shales having different aromatic and aliphatic carbon distributions is necessary for the development of a general chemical kinetics model of kerogen decomposition. Also, by understanding these relationships it may be possible to improve the efficiency of the conversion processes or to develop novel approaches for greater conversion of kerogen to oil. Despite the large number of studies that have been conducted on oil shale thermal decomposition, a paucity of data exists on the composition of the reaction products. In particular, there is little information on the thermal behavior of the bitumen intermediate which, although often used in kinetic models of oil shale decomposition, is seldom measured. However, understanding the mechanisms of formation and destruction of the bitumen, and relationships between kerogen structure and bitumen formation can lead to development of novel oil shale processes, as well as improvements in conventional oil shale retorting processes. An isothermal reaction system has been developed specifically to study kerogen decomposition. This retort uses a fluidized sand bath to heat rapidly a reasonably large shale sample, enclosed in a thin-walled reactor, to reaction temperatures as high as 932°F (500°C). Typically, 20-gram samples of shale are used to provide sufficient product for analysis. A sweep gas is used to carry reaction products efficiently from the reactor into a dry ice-cooled trap, with volatiles passing on to the gas analysis system. The composition and rate of the sweep gas flow can be varied to investigate the effects of the gaseous environment on the kerogen decomposition reactions. Elemental analyses of the solids and liquids are obtained using standard techniques. Liquid state NMR measurements are made on the shale oils. Solid-state NMR measurements are made on the retorted shales.

ACCOMPLISHMENTS

The isothermal decomposition of western and eastern reference oil shales has been studied in the range of 375°C to 440°C. Because the two oil shales have significant differences in their kerogen structures, the results obtained from
this study allow for some relevant comparisons to be made on the formation of the bitumen intermediate.

The amount of extractable bitumen appears to be inversely proportional to the carbon aromaticity of the kerogen. This study showed that the eastern reference shale, which has a high carbon aromaticity, produced small amounts of bitumen, whereas the western reference shale, which has a low carbon aromaticity, produced significantly more bitumen during pyrolysis. The oil and bitumen evolution data, coupled with the differences in the oil shale kerogen structures, suggest that the ability to form bitumen is related to the parent kerogen structure.

For both oil shales, the maximum amount of recoverable bitumen increased with the pyrolysis temperature. This observation is compatible with an oil shale decomposition model in which the activation energy for kerogen decomposition is greater than the activation energy for bitumen decomposition.

Recovery of the bitumen intermediate can lead to enhanced recovery of soluble products. By quenching the reaction after 30 minutes at 425°C and recovering the bitumen and oil, 85% of the kerogen in the western reference shale can be recovered as soluble products. This is significant improvement over the 65-70% conversion of kerogen to oil obtained from western oil shale during most pyrolysis processes.

PUBLICATIONS


The objectives of the rapid pyrolysis research on oil shale at the Morgantown Energy Technology Center (METC) are to identify reaction mechanisms at various heating conditions and to provide the understanding needed to predict shale conversion and product distribution under rapid heating conditions. Research results from the flash lamp reactor indicate that kerogen decomposes on a very rapid time scale (1 ms), but the temperatures (900-1200°C) necessary for rapid conversion on this time frame tend to favor gasification instead of pyrolysis. More moderate temperatures (650°C) and heating rates (125°C/sec) in a wire grid reactor result in conversion levels of greater than 90% of the organic matter, principally to liquids. Solvent swelling experiments on shales suggest that kerogen is a highly cross-linked macromolecule which after rapid heating yields high molecular weight, heteroatomic components, with only slightly greater aromaticity but with significantly more aromatic ring systems per molecule than observed in the feed. Analysis of the flash lamp liquid products indicates that the cleavage of ester linkages and alkyl-aryl linkages occurs between 1-2 J/cm² incident energy. The condensation of aromatic ring systems was dramatic at higher incident energies. Apparently, the vaporization of large molecules is favored at low energies, but as the energy increases the molecular weight of the liquid product decreases and gaseous products are favored. These findings provide insight into the nature of the reactions important at rapid heating rates and which are necessary to evaluate the effects on product quality, distribution, and overall conversion. Such information can be used to assess process alternatives by coupling mechanistic models based on such experimental data with systems models.
ABSTRACT

The purpose of this project is to find out how long oil shale must be heated to recover the oil. We heated Green River and New Albany shales in a laboratory fluidized bed. The volatile pyrolysis products were transferred to a combustion tube and burned. The rate of pyrolysis was calculated from the concentrations of carbon dioxide and steam in the burned pyrolysis gas as measured by means of an on-line mass spectrometer. The amount of carbon and hydrogen released during pyrolysis was calculated from the flow rate of the fluidizing gas and the concentrations of carbon dioxide and steam.

ACCOMPLISHMENTS

We have determined the rate of shale pyrolysis over the temperature range 450° to 525°C. We find the rate of pyrolysis of Green River Shale at 500°C is approximately three times faster than we previously thought.

We found that all of the experimental apparatus that the pyrolysis gas contacts must be at pyrolysis temperature to avoid condensation of heavy oil which subsequently form coke and secondary products. These reactions impede the flow of some pyrolysis products to the pyrolysis detector and have apparent pyrolysis rates which are longer than the true rates.

We accomplished carbon and hydrogen balances during these rate measurements by burning the residual char immediately after pyrolysis.

There is no change in the mechanism of kerogen thermal decomposition over the temperature range investigated.
We have modeled the kinetic data and find that the Green River shale pyrolysis occurs in two steps, an initial fast step that releases 17% of the oil and gas, and a second slower step.

Because the first step is so much faster than the second, the time for any degree of pyrolysis at a given temperature (T) can be expressed by a single first-order rate constant \(k\) as follows:

\[
t = -\ln(\text{FR})/k + \ln(0.83)/k
\]

where

\[
t - \text{time of pyrolysis in minutes}
\]

\[
\text{FR} - \text{fraction of recoverable organic carbon remaining after } t
\]

\[
k = 3.0 \times 10^{14} \exp\left(\frac{49000}{RT}\right) \text{ minutes}^{-1}
\]

We find that pyrolysis and char combustion are not congruent processes in Green River shale. The rapid initial part of kerogen pyrolysis is rich in hydrogen.

FUTURE PLANS ARTICLES AND PRESENTATIONS

ABSTRACT

The Oil Shale Program at the Morgantown Energy Technology Center (METC) is investigating feasible/practical methods for the conversion of oil shale to useful liquid products. The present focus is on pyrolysis processes with fast to rapid heating rates. As part of the METC Program, the Pyrolysis Modeling Project is responsible for developing the predictive capability required to support the in-house experimental and system analysis research. Presently, modeling activities are concentrated on a flash lamp reactor system (rapid heating regime) and a laminar, entrained flow reactor (LEFR) system (fast heating regime). The modeling approach has focused on reaction kinetics, heat and mass transfer, and multiphase fluid flow. This approach, thus far, has led to the development of the Xenon Flash Lamp (XELAMP) code for the rapid heating, flash lamp reactor system, and the modified Pulverized Coal Gasification and Combustion, two-dimensional (PCGC-2D) code for the fast heating, LEFR system.

Flash pyrolysis of oil shale in a xenon flash lamp reactor is an extremely rapid process that allows very fast heating and quenching of the shale (up to $10^6^\circ$ C/s) to increase the product yields. XELAMP is a transient, one-dimensional code which predicts radiant energy and temperature distributions across the shale layer and reactor using the concept of successive reflection and absorption of photo-radiative energy. The code also predicts the extent of reaction for kerogen decomposition, mineral' (carbonate) reactions, and bound water release. Thermal contact resistance between the shale particles and the reactor wall is also considered. Preliminary validation of the code shows a maximum deviation of less than 10 percent between the predicted and measured results for a polystyrene/graphite experiment conducted in the flash lamp reactor. Parametric studies were conducted to examine the effects of particle size, lamp pulse width, input energy flux, and cooling medium on system performance. Present and future work involves identifying and enhancing the model for the important kinetic reactions in the process. Two reaction pathways for kerogen decomposition during rapid pyrolysis are currently being considered. The feasibility of these pathways is being examined utilizing experimental data, and attempts are being made to determine the appropriate kinetic parameters.

PCGC-2 is an axially symmetric, two-dimensional, steady state code originally developed by Brigham Young University (BYU) to simulate pulverized coal gasification and combustion under entrained flow conditions. The code was modified at METC to handle oil shale pyrolysis processes, emphasizing the LEFR system. The modified code is presently being used to test alternative designs of low turbulence particle injectors. Low turbulence is important so that the residence times of the oil shale particles can be accurately determined, thus yielding accurate values for the devolatilization rates. The code is also used to analyze the factors that influence the amount and
type of devolatilization products. This, in-turn, provides important infor-
mation about the kinetics involved for fast heating rate, oil shale pyrolysis
processes. The gas phase chemistry package in the original code was replaced
with METC's Chemical Equilibrium Code (METC- CEC) to obtain a better estimate
of product gas components. The code has been used to study the solid-gas
mixing at the vicinity of solid and gas feeding to the LEFR. Particle
trajectories as well as gas streamlines can also be generated from the code.
Future work involves estimation of pathways and kinetics for the dominant
pyrolysis reactions.

Both the XELAMP and PCGC-2 codes provide a basic framework for the develop-
ment of more generic (independent of reactor shape) codes. The development
of a generic single-particle model (SPM) is underway and the model can be
applied to large-scale pyrolysis processes with different heat sources and
reactor configurations. Examples of possible applications are: fluidized-
bed retorting, entrained flow reactor, microwave pyrolyzer, and others.
The SPM model is a key link in merging the mechanistic information obtained
from the flash lamp and LEFR studies for the development and optimization
of feasible, commercial scale oil shale pyrolysis processes.
Water Generation During Pyrolysis of Oil Shales

1. FTPA NUMBER: W-7405-ENG-48
   CONTRACTOR: Lawrence Livermore National Laboratory
   P.O. Box 808
   Livermore, CA 94550
   FTS: 532-6450
   415-422-6450
   CONTRACTOR PROGRAM MANAGER: Robert N. Schock
   PRINCIPAL INVESTIGATOR: Arthur E. Lewis
   METC PROJECT MANAGER: Theodore C. Bartke
   CONTRACT PERIOD OF PERFORMANCE: FY87, FY88

2. ABSTRACT: Thomas T. Coburn, Myongsook S. Oh,
   Richard W. Crawford, and Kenneth G. Foster

   A triple quadrupole mass spectrometer (TQMS) built in house has been used to study
   chemistry and kinetics of specific compounds generated by retorting oil shales. We
   will focus on our use of the TQMS to monitor water and present details of our
   studies along with a quality assurance investigation.

   Water formation affects both the chemistry and energy balance in an oil shale
   retort. Quantitative, well-resolved, real-time measurements of water released as oil
   Shale is heated have not previously been available. We developed a mass
   spectrometric method and applied it to the study of water evolution during batch
   pyrolysis of oil shale in a small thermal-gradient vessel. We investigated a
   Devonian oil shale (Kentucky, New Albany Shale) and a Green River Formation oil
   shale (Colorado, Mahogany Zone). Precursors of pyrolysis water are different for
   these oil shales since they differ in mineralogy and kerogen structure. Each of the
   following types of water contribute to total evolved water to a greater or lesser
   extent: a) free water, b) water from hydrated salts, c) bound mineral water, d)
   water from the reaction of iron compounds with R^S, e) water from elimination of
   organic oxygen, and f) equilibrated water. The major sources of water are
   eliminated when Green River oil shale is acid leached. Our methods have been
   tested on an Israeli oil shale and results compared to a previous report. Future
   work will include the study of pressure effects and predictions based on mineralogy.
ACCOMPLISHMENTS:

Objectives of the project are to provide a technical base which will contribute to the advance of oil shale retorting technology including the mitigation of undesirable environmental effects. Our water evolution studies provide data that have been used in enthalpy calculations that result in a satisfactory energy balance for both Green River and Devonian oil shales; we can predict conditions where self-fluidization resulting from rapid water release in the early stages of flash pyrolysis might pose difficulties; the details of quantitative water release allow us to confidently use a new detector to obtain isothermal oil shale decomposition kinetics while simultaneously obtaining elemental carbon and hydrogen balances.

PUBLICATIONS FROM OUR TQMS STUDIES OF OIL SHALE PYROLYSIS:


ABSTRACT

The purpose of this project is to find out the rate and amount of oil loss due to exposure of shale oil vapors to oxidized shale. The data are needed to develop a model of cracking and coke formation essential to predict the extent of these reactions in scaled up solid-recycle oil shale retorts. Our approach has been to conduct laboratory experiments and also pilot retort runs.

One set of laboratory experiments was designed to measure the rate of reaction of oil vapors with oxidized shale. In these experiments we injected shale oil vapors into a bed of oxidized shale and measured the distribution of carbon between solid (char), gas and oil as a function of residence time, bed temperature and bed particle size.

Another set of laboratory experiments were designed to measure the distribution of raw shale organic carbon between solid and gas phases under the conditions of flash pyrolysis. In these experiments a sample of shale was flashed pyrolyzed in a fluidized bed containing oxidized shale. A carbon balance was obtained, but not reaction rates. The amount of carbon released as oil and gas was determined by burning the pyrolys products and measuring the resulting carbon dioxide content. The fraction of the total organic carbon that remained in the bed was measured by burning the pyrolyzed shale. The principal variables in these experiments were the ratio of raw shale to oxidized shale, shale type and pyrolysis temperature.

He have measured the carbon distribution and oil yield for Green River and New Albany shales in our solid recycle pilot retort, variables have been pyrolysis time and temperature, recycle ratio and combustion temperature.
ACCOMPLISHMENTS

We have measured the kinetics of shale oil thermal decomposition. Without oxidized shale, but passing the oil vapors over quartz sand, the products of the thermal decomposition of Green River shale oil are mostly light gases with minor amounts of coke. The fraction of the oil cracked to gas and coked to solid carbon approaches a limit of approximately 40X. The time to this limit depends on temperature according to an Arrhenius expression.

When oil shale vapors were exposed to oxidized shale, the maximum fraction of oil cracked was approximately double the fraction cracked over sand without oxidized shale, and the fraction of coke is much higher.

In flash pyrolysis in laboratory fluidized beds we have found that the fraction of organic carbon in the raw shale recovered as oil and gas is higher than for slower pyrolysis, for example Fischer assay.

We have also found that oxidized shale in the fluidized bed decreases the yield of oil plus gas and increases the yield of coke. When the concentration of oxidized shale in the fluidized bed is equal to 4 times the amount of raw shale pyrolyzed the yield of oil plus gas falls to the values recovered by Fischer assay, and this observation is true for Eastern US shale as well as shale from Western US.

We also observe that those shales that by flash pyrolysis demonstrate the best improvement in yield compared to Fischer assay, are the very shales which suffer the highest yield loss in the presence of oxidized shale.

We have learned that out pilot retort, operated with recycled Green River or New Albany shale, can recover the same amount of oil as recovered by Fischer assay, but little or no more. This disappointing result is consistent with our laboratory data.

FUTURE PLANS ARTICLES AND PRESENTATIONS

The laboratory experiments on coking and cracking are continuing in order to understand the mechanism of coke formation and seek to find ways to utilize oxidized shale for beneficial cracking reactions while minimizing coke formation.

Work needs to be done on the rate of reaction with oxidized shale of oil vapors produced by the flash pyrolysis of Eastern US shale.
Mild Gasification
SESSION IB

Chairperson: Madhav R. Ghate
MILD GASIFICATION: PRODUCT CHARACTERIZATION

FIELD WORK PROPOSAL: FEAA310

CONTRACTOR: Martin Marietta Energy Systems, Inc.
Oak Ridge National Laboratory
Oak Ridge, TN 37831

CONTRACTOR PROJECT MANAGER: R. L. Graves

PRINCIPAL INVESTIGATORS: R. L. Graves, C. S. Daw

METC PROJECT MANAGER: Sophia Lai

PERIOD OF PERFORMANCE: April 15, 1988 to September 30, 1990

ABSTRACT

The purpose of this investigation is to evaluate the products of mild gasification to provide data which can be used in systems studies of the relationships between process conditions, product yields, and the product quality. Of particular interest is whether the oil and char from mild gasification are of suitable quality to be used as fuels for diesel engines and boilers with little or no upgrading.

From previous studies it is known that light liquids can be produced through mild devolatilization or mild gasification of coal. However, the yield and quality of these liquids vary with processing conditions. In general, higher yields at higher temperatures result in lower quality products. Some liquids upgrading may be required even under mild-gasification conditions. However, the severity and cost of the treatment will vary with the raw liquid quality. A similar situation exists for the char product in that severe devolutilization tends to reduce the char's suitability as a boiler fuel (but may be satisfactory for other uses). These tradeoffs between product quality, yields, and upgrading requirements determine the overall viability of the mild gasification approach.

With the cooperation of METC and industry participation, samples of mild gasification products will be acquired from existing pilot plant operations. Small quantities of typical products have been previously produced at ORNL, but at present there are no plans to reactivate this bench-scale equipment. Rather, samples are to come from private industry groups who are involved in process development. The liquids will be characterized in a laboratory using consistent test procedures. The analyses will include elemental analysis, viscosity, gravity, heating value, aromatic/aliphatic content, and others as appropriate. Determinations of the blending characteristics are to be achieved as well as product stability in the latter stages of the program. NMR and distillation data are to be generated as well. The data will be compared to those for liquids from other coal liquefaction processes such as the Exxon Donor Solvent (EDS) process and the current pilot plant operations at Wilsonville, Alabama. Existing data from previous low-temperature
carbonization experiments will also serve as reference points. The characterizations of the liquids will be coordinated with companion DOE projects concerning liquids upgrading.

Physical and chemical characterizations give only a rough idea of what potential a specific liquid fuel has. To better understand the utilization of these materials, it is customary that tests be conducted with selected fuel samples on a standard Cooperative Fuel Research (CFR) test engine (to determine cetane number). If sample quantities are sufficient, this will be done outside ORNL. In addition, tests in a more modern research engine are to be conducted to compare the performance of the fuels in an engine combustion system more realistic than a CFR engine. These experiments will be performed for both untreated, blended, and upgraded liquids contingent on the availability of sufficient quantities. Because the amount of liquids generated is small, it is further planned to use blends of a reference diesel fuel and mild gasification oils to determine their combustion performance.

Representative mild gasification char samples will be characterized in terms of fundamental parameters which have been previously measured for conventional coals and linked to performance in large-scale combustors. During the first year of this effort, the following parameters will be measured for two or three selected chars: 1) ultimate and proximate analysis, 2) ash analysis (including trace metals), 3) BET surface area and pore-size distribution, 4) true (helium density) and hydrodynamic particle density, 5) heating value, 6) ignition temperature, 7) ignition delay, and 8) intrinsic combustion kinetics at low, medium, and high-temperature. Previously well-characterized coals, such as Kentucky No. 9, Pittsburgh No. 8, or anthracite, will be compared with the mild gasification chars as controls.

If small-scale char characterization results are promising, future large-scale combustion testing of the char will be needed to confirm the predicted performance. In this case, it is recommended that combustion tests be conducted at char feed rates of 10-100 lb/hr on at least one bench-scale facility. Depending on the results of the fundamental characterizations, ORNL will identify the most suitable combustor type or types (e.g., pulverized combustor, fluidized bed, circulating bed, stoker fired grate) and the most important test issues to be addressed.

ACCOMPLISHMENTS

The project has been active only for two months and efforts have focused on acquiring samples and planning/establishing the test program. Arrangements have been made to acquire oil and char samples from United Coal Company and SGI International, Inc. Other reference fuels and coal liquids are being collected also. A single-cylinder diesel engine is being reactivated for combustion tests on oil products and a thermogravimetric analyzer has been identified for use in the initial studies of the char. In addition, a subcontract with Babcock & Wilcox is being placed for char combustion tests.

PUBLICATIONS

None.
ABSTRACT

The objective of this project is to evaluate the mild coal gasification liquid for locomotive fuel application. Emphasis has been focused on directly using the coal liquid as a locomotive diesel fuel with minimum upgrading. Recent laboratory results show that the mild gasification liquid can indeed be used directly as Diesel No.2 or No.3 fuels after only minor beneficitation procedures. It appears that expensive hydrotreating processing, such as hydrocracking, can be avoided.

Using only simple beneficitation procedures, such as alkaline washing and distillation, a quarter of one barrel No.2 petroleum-based diesel fuel equivalent was produced from one barrel of mild coal gasification liquid. Furthermore, when the coal liquid was upgraded by a suitable catalyst, the yield increased to 80%. That is, one ton of coal can yield almost one barrel of diesel fuel for the existing locomotive engine. Analytical data of this fuel have shown similarities to petroleum diesels and successful performance is expected.

A small diesel engine test of this fuel has been demonstrated successfully. We are currently generating large enough quantity of this fuel to conduct locomotive engine tests.

The process for locomotive coal diesel production is under development at UCC Research Corporation's new R&D Center in Bristol, Virginia. It is expected that this process will successfully produce locomotive fuel from mild gasification coal liquid and in the near future we will be able to totally rely on coal derived diesel fuel to power our trains.
ACCOMPLISHMENTS

• Acquired 20,000 gallons of raw coal liquids

• Completed the coal liquid analysis

• Based on laboratory beneficiation test findings, established the processing scheme for the 20,000 gallons of coal liquids

• Determined product properties

• Completed the purchase and erection of a liquid coal refinery facility (not DOE funded) capable of processing up to 42,000 gallons per day of coal liquids
UPGRADING OF CONDENSIBLES FROM COAL DEVOILATILIZATION

FWP NUMBER: FEAA323

CONTRACTOR: Oak Ridge National Laboratory
Chemical Technology Division
Oak Ridge, TN 37831-6226
(615-574-6775)

CONTRACTOR PROJECT MANAGER: R. A. Bradley

PRINCIPAL INVESTIGATORS: John E. Mrochek
Charles D. Scott

METC PROJECT MANAGER: Sophia Lai

PERIOD OF PERFORMANCE: March 17, 1987 to September 29, 1988

ABSTRACT:

The purpose of this project is to develop energy-efficient upgrading methodologies for the condensible products of mild coal devolatilization. Upgrading entails the removal of heteroatom-containing (N, S, and O) "contaminants" which are deleterious to the use of these products as fuels. Emphasized in this program is the development of chemical separations technologies such as solvent extraction and solid-phase adsorption as alternative or adjunct technologies to the more conventional, but expensive, hydrotreatment.

Acidic reagents, including acetic, formic, chloroacetic, trichloroacetic, and phosphoric acids, have been tested for their efficacy in removing nitrogen-containing "contaminants" from condensible products by two-phase solvent extraction. Extractions with chloroacetic and trichloroacetic acids were performed to determine the effect of acid strength on nitrogen removal; however, the principal reagents employed were acetic and formic acids. Raw condensible liquids and some distilled fractions (<650°F) were studied. Condensible liquids from the United Coal Company Research Corporation in Bristol, VA, SGI International, Pittsburgh, PA, and the Allis-Chalmers KILnGAS process, East Alton, IL, were employed in the extraction studies.

ACCOMPLISHMENTS:

Raw condensible liquids from UCC and Allis Chalmers were highly aromatic with H/C ratios ranging from 0.73 to 0.88, while the second-stage product from SGI International was about 21% aromatic with a H/C ratio of 1.73. Nitrogen contents ranged from a low of about 0.4% for the SGI product to 1.5% for one of the UCC products, while sulfur ranged from virtually none (0.07%) in the SGI liquid to 3.38% in the KILnGAS product. Solvent extractions were performed with aqueous acetic and formic acids in concentrations of 25, 50, 75, and 90 vol %, with the greatest removal of nitrogen generally observed for 75 and 90 vol % concentrations. Extractions of the UCC product were complicated by their higher viscosities and subsequent retention of water in the organic phase. Generally, removal of nitrogen from a raw condensible product from UCC was about 51%, from the KILnGAS product about 67%, and from the SGI second-stage product about 98% (the latter appeared to be more like a distillate than a raw condensible
product) using 75 vol % acetic acid. Distillates (<650°F) were prepared from a UCC and a KILnGAS product, improving their quality and removing more than 50% of their organonitrogen content. Extraction of these distillates with 75 vol % acetic acid reduced their N assays to 0.09 and 0.13%, respectively. More acidic chloroacetic acids provided no improvement over acetic acid in the extraction of nitrogen.
ABSTRACT

The object of this project is to examine the potential of crude oils derived from coal, oil shale or tar sand as sources of aviation turbine fuels. Three oils will be selected from a group consisting of (1) a coal liquid produced by mild gasification (2) an eastern shale oil, (3) a western shale oil, and (4) a tar sand oil. The selected oils and their distillate fractions will be characterized in detail, and appropriate fractions will be processed to candidate fuels. Processing steps will include dewaxing (for highly paraffinic materials), acid/base extraction for heteroatom removal, thermal treatment for boiling-range conversion, and hydrogenation for heteroatom removal and aromatics saturation. Candidate fuels will be analyzed in detail for comparison with fuel specification requirements. Since no experimental work has been conducted in FY88 pending receipt of crude oil samples, it is useful to review recent processing experiments with Great Plains by-product liquids. This work provides some insights into processing coal pyrolysis liquid to make military jet fuels.

The Great Plains Gasification Project (GPGP) produces three liquid by-products from lignite gasification - tar oil, crude phenols and a light naphtha. Neither the crude phenols nor the naphtha are suitable sources for jet fuels production. The naphtha is too low-boiling for any fuel except gasoline. The crude phenols could make a minor contribution to JP-4 production, but at an extremely high processing cost. The tar oil however represents a source of 2,000-3,000 barrels per stream day of either JP-4, the principal military jet fuel, or JP-8, the military version of commercial jet fuel. Our hydrogenation experiments demonstrated that tar oil could be a suitable source for either of these conventional fuels or a high-density JP-8. The experiments also demonstrated the advantages of pre-distillation to reject particulates and high-boiling (>800°F) aromatic material, and the feasibility of extracting phenols with caustic before hydrogenation. These experiments have alerted us to some of the processing requirements in the forthcoming work with mild gasification coal liquids. Elemental composition of pyrolysis tars from coals
of various ranks and of the GPGP tar oil are quite similar. We expect mild gasification liquids to resemble these. Atomic hydrogen to carbon ratios are mostly in the 1.2 to 1.4 range indicating high aromaticity. Nitrogen, sulfur and oxygen compounds are present in significant amounts. Exploring practical ways of removing these by means other than catalytic hydrogenation such as acid base extracting is another objective in advanced fuels research. Such methods are expected to reduce but not avoid catalytic hydrogenation as the most practical way of removing residual heterocompounds and saturating aromatics.

Typical characteristics of shale oils and tar sand oils permit some predictions about their jet fuel potential. Western shale oil has been studied extensively and has been the subject of numerous processing studies. These studies demonstrate that it is a suitable source for conventional jet fuels. Its highly paraffinic nature makes it a poor source for fuels of higher density. Eastern shale oil may be a much better source for higher density fuels because of its more aromatic composition. Tar sand oils from some deposits have enough aromatic and alicyclic components to be potential sources for fuels of higher density. The potential of some tar sand oils as sources of conventional military fuels has been demonstrated in previous studies.

ACCOMPLISHMENTS

Performed a series of hydrogenation experiments demonstrating that a coal pyrolysis liquid could be a source of jet fuels.


FUTURE PLANS, ARTICLES, AND PRESENTATIONS

Begin the work planned for FY88.


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Oil Shale II
SESSION 2A
Oil Shale Surface Processes R&D

Chairperson: Catherine E. Gregoire
ABSTRACT

The purpose of the Livermore oil shale project is to contribute to the development of a basic understanding of oil shale, which will lead to efficient processing and contribute to an economically-viable and an environmentally-acceptable industry. The approach is a combination and iteration between mathematical modeling, laboratory measurements, and operation of a 1 tonne-per-day oil shale retort and other components, as required.

The LLNL pilot retort consists of two primary units: a dense phase plug-flow pyrolyzer and a combination air lift pipe and delayed fall cascading bed combustor. The raw and recycled shales are combined before entering the pyrolyzer in a simple mixer equipped with a series of inclined chutes, through which solids fall. Once mixed, the solids enter the plug-flow pyrolyzer where the raw shale reaches a retorting temperature of 500°C within 20 seconds and pyrolysis is complete within 2 minutes. (Hot gas retorts require 15-30 minutes to achieve the same degree of conversion.) The spent shale leaves the retort and is cycled to a combustor which burns the residual carbon and heats the shale. With a recycle ratio of 4 to 1, a combustor exit temperature of 640°C is required to maintain the process.

In the following I discuss pilot retort results and show correlations with laboratory experiments and model predictions. Using the retort, we have investigated high throughput, yield loss through coking and cracking reactions, shale mixing requirements, and a new scheme for spent shale cooling. We have found that for processing Western shale, more rapid throughput is possible, consistent with recently measured kinetics for
Western shale pyrolysis. Fischer assay yields were obtained for Western and Eastern shales, which correspond with laboratory fluidized bed experimental measurements, when oxidized shale was present in the bed. Mixing limits for layers of hot and cold shale were investigated in the retort and modeling studies. Layers in the 1-2 cm range were found not to reach thermal equilibrium in the 2-3 minute pyrolyzer retorting time. Shale cooling via solid recycle was demonstrated in the pilot retort. Wetting of shale cooled by this method will require additional work. Sulfur emissions are an important concern in shale processing. We have measured a number of sulfur species on-line and via grab samples, processing both Western and Eastern shales. 

Our recent work in study the pathways by which light gases are formed during oil shale pyrolysis has led to a concept that suggests that it may be possible to minimize the gas production route in favor of oil production. We find that under some conditions the radical precursors to light gases can be trapped and polymerized back to oil. A demonstrated increase in oil production has been accomplished by the introduction of an olefin sweep gas during pyrolysis with a catalyst present.

ACCOMPLISHMENTS

• We determined operating parameters, sulfur and nitrogen distribution and oil yield during solid-recycle retorting of several Western and one Eastern oil shale.

• We investigated the degree of mixing required for heat transfer in dense phase solid-recycle systems.

• We investigated the use of solid-recycle to cool spent shale as a first step in solid waste disposal.

• We developed easily used enthalpy relations for Western and Eastern shales.

• We improved the model for carbonate decomposition based on a survey of the literature and reanalysis of experimental results.

• We performed a parameter study of a generic commercial-scale hot-solids recycle retorting process consisting of a fluidized-bed pyrolyzer and a lift-pipe combustor.

REFERENCE


KENTORT II PROCESS DEVELOPMENT

COOPERATIVE AGREEMENT: DE-FC21-86LC11086

CONTRACTOR: Kentucky Energy Cabinet Laboratory
P.O. Box 13015
Lexington, KY 40512

CONTRACTOR PROJECT MANAGER: Dr. Thomas L. Robl

PRINCIPAL INVESTIGATORS: Scott D. Carter
Aurora M. Rubel
Darrell N. Taulbee

METC PROJECT MANAGER: Carl E. Roosmagi

PERIOD OF PERFORMANCE: September 30, 1986 to September 30, 1989

ABSTRACT

The purpose of this program is to develop on a small scale a process to improve process efficiency, process economics, and resolve environmental problems involved in the utilization of eastern U.S. oil shale as an energy source. The KENTORT II process has been conceived as a means of meeting these criteria. KENTORT II arose from extensive fluidized bed pyrolysis research which showed that oil yields in excess of 150% of Fischer assay were possible for Eastern shales. In addition to a fluidized bed pyrolysis zone, the process includes gasification and combustion zones with combined solid and gas heat transfer among the sections. The gasification section produces synthesis gas from unused carbon in the char while removing inorganic sulfur as H2S which facilitates flue gas clean-up, provides a raw material for sulfur production, and reduces potential acid drainage from disposed solids. The program is divided into three main areas: (1) the evaluation of important gasification parameters for oil shale char and application to the development and operation of an integrated pyrolysis/gasification reactor system; (2) the evaluation of combustion rates of oil shale materials with application to the design and operation of a complete KENTORT II prototype; and (3) the evaluation of the leaching characteristics of the processed solids from the system. Two experimental systems are used in the program: (1) a 3.8-cm diameter, single-stage fluidized bed reactor and (2) a 7.6-cm diameter, multi-stage fluidized bed reactor. The 3.8-cm diameter unit is used to obtain the fundamental rate data for the gasification and combustion of the materials to be used in the integrated reactor system. The gasification kinetics study included two experimental matrices to evaluate the effects of bed temperature, mean char residence time, and fluidizing medium on the gasification characteristics of a pyrolyzed oil shale sample stored under argon. A study in the 3.8-cm unit to evaluate the combustion of raw, pyrolyzed, and gasified oil shales is currently in progress. The integrated pyrolysis/gasification system is operational and is progressing through test matrices to: (1) evaluate the gasification kinetics of nascent oil shale char; and (2) evaluate the effects of solid and gas heat transfer from the gasifier to the pyrolyzer. The char in the integrated unit gasified more readily compared to the stepwise pyrolysis, cooling, and gasification procedure utilized for the 3.8-cm diameter reactor. Once combustion kinetics and cold-flow modelling of reactor fluid/solid dynamics are complete, a combustion zone will be added and the integrated KENTORT II prototype will be tested.
ACCOMPLISHMENTS

A 3.8-cm diameter fluidized bed reactor was used to investigate the gasification kinetics of pyrolyzed Kentucky oil shale. A test matrix comprised of bed temperature (750 to 850°C) and mean solid residence time (10 to 60 minutes) was completed.

A gasifier design for an integrated 7.6-cm diameter pyrolysis/gasification reactor system was developed based on the results from the above gasification kinetics study and a cold-flow modelling study.

Fabrication and shakedown of the 7.6-cm diameter pyrolyzer/gasifier was completed. A severe corrosion problem in the gasifier was solved after a metallurgical evaluation indicated that aluminized stainless steel withstood the gasification environment.

A 10 ton sample of the Cleveland Member of the Ohio Shale was mined and processed into a master sample for use in the process study.

The integrated pyrolyzer/gasifier test matrices comprised of bed temperature, mean solid residence time, and char recirculation rates were started.

The 3.8-cm diameter fluidized bed reactor was converted into a combustor and a combustion kinetic study of Kentucky oil shale materials was started.

PUBLICATIONS AND PRESENTATIONS


SURFACE RETORTING STUDIES

CONTRACT NUMBER: Cooperative Agreement DE-FC21-86MC11076

CONTRACTOR: Western Research Institute
Box 3395, University Station
Laramie, Wyoming 82071

CONTRACTOR PROJECT MANAGER: Mr. Verne Smith

PRINCIPAL INVESTIGATORS: Dr. Chang Yul Cha
Mr. Norman W. Merriam
Dr. Frank D. Guffey

METC PROJECT MANAGER: Mr. Carl Roosmagi

PERIOD OF PERFORMANCE: October 1986 to September 1992

ABSTRACT

The objective of this work is to develop new oil shale processing technologies that (1) produce specialty products such as BTX Feedstocks, advanced jet fuel feedstocks, and improved asphalt binders, (2) produce upgraded oil, and (3) produce greater oil yields than other pyrolysis processes. Preliminary data from the ROPE© (Recycle Oil Pyrolysis and Extraction) concept suggest that processing oil shale with product oil recycling produces upgraded oil that may be used as feedstocks for BTX and advanced jet fuel production. Preliminary data from the inclined fluidized-bed (IFB) suggest that retorting oil shale in the IFB will produce higher yield of improved asphalt binder. The ROPE© process consists of two pyrolysis steps: (1) retorting oil shale at a lower temperature (T < 420°C) with the recycled product oil, and (2) completing the pyrolysis of the residue at a higher temperature (T > 500°C) in the absence of product oil recycle. Three oil shale resources, New Albany, Israel, and AMAX oil shales, have been investigated using the ROPE© concept. Preliminary results indicate that oil yields from the ROPE© process are greater and oil quality is much improved compared with other oil shale processes. The product oil from the ROPE© process was evaluated as feedstock for production of BTX and advanced jet fuel.

The inclined fluidized-bed is designed to provide a plug-flow for solids with the uniform residence time of particles having different sizes and a cross-flow for gas to achieve a short oil vapor contact time with solids that minimizes secondary reactions. Tests conducted by retorting eastern and western oil shales with steam produced very heavy, viscous oil, which may be used as asphalt binders. Oil yields from recycle gas retorting of western and eastern oil shale were 103X and 114X of Fischer assay, respectively. In combustion tests using raw oil shale, 99.2% and 96.1X of the organic carbon was burned from western and eastern oil shales, respectively. Combustion efficiency of retorted shale was also high (97J carbon removal). The ROPE©
process has a great potential to provide feedstocks for the advanced jet fuel from eastern oil shale with oil yield greater than the Fischer assay. Also, the ROPE® process will assist existing oil shale processes in improving their economics by coprocessing shale with their rejected oil shale fines and producing light oil product which can be used as diluent to decrease the pour point of shale oil for pipeline transportation. The inclined fluidized-bed reactor has been shown to be capable of producing a high yield of asphalt binder from oil shale and combusting retorted shale to very low organic content.

ACCOMPLISHMENTS

• Preliminary tests were conducted using New Albany, Israeli, and AMAX oil shales in the 2-inch diameter screw reactor (ROPE®).

• A 10-day shakedown test was completed using New Albany oil shale in the 6-inch diameter bench-scale unit (ROPE®).

• Western and eastern oil shales were retorted with steam in the first stage inclined fluidized-bed and the retorted shales were burned in the second stage inclined fluidized-bed reactor.

• Eastern and western shales were combusted in the inclined fluidized-bed reactor.

FUTURE PLANS, ARTICLES, AND PRESENTATIONS

ROPE© Process Development

• Conduct 10-day test using New Albany oil shale and Colorado oil shale in a 6-inch bench-scale unit

• Obtain process data using 2-inch screw reactor to find optimum process conditions

• Techno/economic evaluation of ROPE© process

Inclined Fluidized-Bed Development

• Conduct retorting tests with eastern and western oil shale to find optimum process conditions for maximum production of asphalt binder

• Investigate the possibility of using the IFB reactor to desulfurize the oil shale product gas and to burn oil shale fines with product gas
ABSTRACT

The overall objectives of this program are to expand the process and environmental data base for moving-bed hydroretorting of Eastern oil shales and to investigate novel concepts which have the potential for increasing process efficiency and resource utilization.

In Task 1, hydroretorting characteristics, a hydroretorting data base was developed for the six eastern oil shales: Indiana New Albany, Alabama Chattanooga, Tennessee Chattanooga, Michigan Antrim, Ohio Cleveland, and Kentucky Sunbury. The objective of Task 2, process efficiency improvement tests, is to test the concept of using synthesis gas retorting and mist-generation as ways to improve the overall process economics. The objective of Task 3, fines utilization tests, is to investigate the potential for increasing resource recovery through fines utilization. The technical viability of fines gasification was demonstrated and analysis of the economic viability is being conducted. The objective of Task 4, process designs/environmental mitigation, is to investigate the potential environmental impact of a hydroretorting facility utilizing various eastern oil shale feedstocks. HYCRUDE has developed a computer program to simulate the operations of a conceptual commercial hydroretorting plant, generated designs for six shales and conducted site-specific environmental analysis. The objective of Task 5 is to obtain representative and geologically documented shale samples needed for this program. This program provides data base which can be utilized by all researchers. Additionally, development of conceptual commercial designs assists in the direction of future research by identifying process areas where additional research may significantly add to the viability of eastern oil shale processing. This program has advanced the moving-bed hydroretorting technology to the point where a demonstration plant is the next logical step.
ACCOMPLISHMENTS

TASK 1. The response of six eastern oil shales to hydroretorting at various pressures and temperatures was determined in HAU. HAU data was utilized in specific run conditions for tests conducted in MBU. Two BSU tests were conducted to ascertain the validity of results in the MBU. The work conducted under this task provided a data base on the six shales.

TASK 2. Synthesis gas retorting is being studied as a possible technique for improving process efficiency. Conceptual designs are being developed to incorporate the results for these tests into a hydroretorting facility for an analysis of the potential for utilizing synthesis gas retorting.

Task 3. Oil shale fines gasification for utilization of the fines that can not be fed into a moving bed retort is being investigated. A conceptual gasification plant design is being developed to determine the economics of gas production in this manner. Cold-flow modeling of the moving-bed retort is also being conducted to determine the maximum amount of fines which can be fed to the retort.

Task 4. The MBU data from Task 1 forms the basis for development for the conceptual HYTORT designs used for the environmental mitigation analysis. Six conceptual HYTORT plant designs were developed, one for each of the shales tested. A site-specific analysis was conducted by Tennessee Technological University.

Task 5. Representative and geologically documented oil shale samples were collected from the states for Alabama, Indiana, Kentucky, Ohio, Michigan and Tennessee. Samples were collected by R. D. Matthews, Inc.

PUBLICATIONS


ABSTRACT

The University of Alabama and HYCRUDE Corporation are completing the third and final year of a joint advanced research and development project, funded by DOE, to evaluate the technical, environmental, and economic feasibility of processing U.S. oil shales by combined beneficiation-hydroretorting. The objectives of the program were to:

- Assess the response of various U.S. oil shales to beneficiation-hydroretorting processing.
- Optimize the beneficiation and hydroretorting process variables in tests of several selected shales and to study the environmental impacts of the two processes.
- Design conceptual commercial plant flowsheets for the beneficiation and hydroretorting processes and prepare an economic evaluation of the combined beneficiation-hydroretorting processing approach to for maximizing oil product extraction from Eastern oil shales.

During the first year the response of 14 U.S. oil shales to beneficiation-hydroretorting was assessed. Nine of the shales were from the Eastern and Central United States, and five were from Western deposits including one from Alaska. Eight of the Central and Eastern shales responded favorably to beneficiation and hydroretort processing. Four of the Western shales were amenable to flotation and two responded to combined beneficiation-hydroretorting.

Based on the results of the assessment study, three Eastern oil shales were selected for more comprehensive tests during year 2. Optimizing the flotation step was a major MRI effort. The research resulted in development of a regrind procedure that produced kerogen flotation concentrates yielding 45 to 71 gallons of oil per ton as determined by the Fischer Assay procedure with accompanying oil recoveries of 90 to 93 percent. Hydroretorting research by HYCRUDE concentrated on
assessing the hydroretorting characteristics of the beneficiated shales in a continuous 1 lb per hour Mini-Bench Scale Unit (MBU). Hydroretorting increased the oil extractions from the concentrates by factors of 1.6 to 1.9 times.

Environmental impact studies as they related to the beneficiation process were concerned within organic ionic species released in the process waters during grinding and flotation. The tests indicated that the ionic species dissolved during processing should not present a serious environmental problem.

Research currently underway is concerned with developing capital and operating cost estimates for beneficiation-hydroretorting facilities recovering 20,000 and 50,000 bbl/day of oil product from different grades (Fischer Assay) of raw and beneficiated Alabama, Kentucky, and Michigan oil shales. Preliminary capital and operating costs have been determined for conceptual plants producing 20,000 bbl of oil product from the Alabama and Kentucky shales by combined beneficiation-hydroretorting. Similar information is being developed for the Michigan shale. Continued engineering studies are expected to refine the economic analysis prior to establishing final plant designs, and capital and opportunity figures for plants producing 20,000 and 50,000 bbl/day of oil from the three shales by combined beneficiation-hydroretorting.

ACCOMPLISHMENTS

The research completed to date has demonstrated the merit of combined beneficiation-hydroretorting for maximizing oil recoveries from "low yield" Eastern oil shales.

A preliminary economic study has shown that processing beneficiated Alabama and Kentucky oil shales by the combined processes reduces the projected selling price of oil by as much as 23 percent when compared to hydroretorting the raw shales alone.

The technology developed during the term of this project has enhanced the importance of Central and Eastern oil shales as a potential national future energy resource.

FUTURE PLANS. ARTICLES, AND PRESENTATIONS

The merit of beneficiation-hydroretort processing of Eastern oil shales to maximize oil extraction has been demonstrated on a bench and semi continuous basis. Future research should include installation and operation of a completely integrated beneficiation pilot plant operation to obtain continuous operating data relative to designing a commercial plant, and to produce kerogen enriched concentrates for processing in IGT continuous bench scale or pressurized fluid bed hydroretorting units.

Twelve presentations and publications resulted from this project. Because the large number of publications they are listed in the attached addendum.
ADDENDUM


M. Misra and Frank Hilleke, "Power Requirements for Ultrafine Grinding of Oil Shale", 1987 Eastern Oil Shale Symposium, November 18-20, Lexington, KY.

PRESSURIZED FLUIDIZED-BED HYDROTERTORTING OF EASTERN OIL SHALES

CONTRACT NUMBER: DE-AC21-87MC11089

CONTRACTOR: Institute of Gas Technology
3424 S. State Street
Chicago, IL 60616
(312) 567-3713

CONTRACTOR PROJECT MANAGER: Dharam V. Punwani

PRINCIPAL INVESTIGATORS: W. P. Bonner (TTU), S. H. Chiang (UP), H. S. Fogler (UM), T. M. Knowlton (IGT), F. S. Lau (IGT), M. C. Mensinger (IGT), R. M. Pfister (OSU), C. Rampacek (UA/MRI), M. J. Roberts (IGT), and D. T. Wasan (IIT)

METC PROJECT MANAGER: Carl Roosmagi

PERIOD OF PERFORMANCE: September 30, 1987 to November 30, 1990

ABSTRACT

The overall objective of the program is to perform the research necessary to develop a pressurized fluidized-bed hydroretorting (PFH) process for producing oil from Eastern oil shales. The integrated PFH process offers the potential for improved process economics compared with those of state-of-the art Eastern shale retorting technologies. The program also incorporates research on technologies in the areas of raw shale preparation, beneficiation, product separation, and waste disposal that have the potential to improve the economics and or environmental acceptability of the PFH process. The program is divided into six major tasks: 1) Determine the hydroretorting characteristics of one eastern oil shale in a lab-scale fluidized-bed retort; 2) Optimize the PFH process for six eastern shales and determine the effects of scale-up from lab- to bench-scale using one oil shale; 3) Develop data for several process improvement concepts (in-bed desulfurization, electrostatic desulfurization and denitrification, electrostatic separation of fines from shale oil, and restricted-pipe discharge system for non-mechanical pressure letdown of solids) to improve the overall economics of the PFH process; 4) Conduct oil shale beneficiation research, including shale grinding, and beneficiation waste treatment and disposal, to increase kerogen concentration in the feedstock for the PFH process; 5) Develop a PFH system for the ultrafine beneficiated shales; and 6) Obtain environmental data and analyze the potential environmental impact of the integrated PFH process. Even though the prime contract was issued on September 30, the six subcontracts were finalized between December 1987 and April 1988. In most of the tasks, major accomplishments to date are design and construction of the experimental equipment and preliminary results of a few initial tests.
ACCOMPLISHMENTS

- Designed and constructed a lab-scale 200-gm batch PFH experimental unit capable of operating at temperatures and pressures up to 1200°F and 1000 psig. A few shakedown tests conducted with Indiana New Albany oil shale show carbon conversion up to 77% (compared to about 35% for conventional retorting) at 1000 psig and preheat and retorting temperatures of 400° and 1000°F, respectively. Also completed the design of a lab-scale continuous PFH experimental unit.

- Measured electric charges on the oil shale particles to be 4 to 6 times higher than those on pyrite particles indicating potential favorable separation by electric fields. Assembled an electrostatic sieve separator system and conducted shakedown tests with Indiana New Albany oil shales ground to 40 microns. SEM analysis indicate the need for particle size to be less than 10 microns for good pyrite liberation. Isolated microorganisms from Eastern oil shales and river bed sediments for microbial desulfurization and denitrification tests. Initial test results showed sulfur reductions in the oil shales treated with the microorganisms of up to 77% in sulfur peak (2.3 KeV) obtained in energy dispersion x-ray flourecense. Designed and constructed a restricted-pipe flow discharge system to develop data for a solids depressurization system that is potentially more economical than the conventional lockhoppers.

- Ordered a stirrred-ball mill for ultrafine grinding of oil shale, and developed the concept for the test equipment for the pressure-cycling comminution of oil shale. Conducted several tests in a 1-meter tall column (10 cm diameter) flotation unit. The quality of the concentrate produced by a single-stage cleaning in this column cell is significantly better than the concentrate obtained by five stage cleaning in a conventional cell. Installed a lab-scale (5-cm diameter) air-sparged hydrocyclone for kerogen concentration tests.

- Assembled a 30-cm diameter Plexiglas fluidization column and conducted fluidization tests with ultrafine beneficiated shale from Alabama with and without inerts in the bed. Fluidization tests indicate that the effective particle size of the ultrafine beneficiated shale is much larger than the individual particle sizes.

- Began mapping shale deposits and water sources in Tennessee to determine a match of shale with water availability for siting a PFH plant. Similar work for the other five states was begun. Assembled the reactor for catalytic removal of heavy metals from shale. Initiated work for the preparation of a computer model for a PFH system.

FUTURE PLANS, ARTICLES, AND PRESENTATIONS

Proceed with the program as planned.
ABSTRACT
Under this contract effort, Foster Wheeler developed conceptual designs and cost estimates for commercial scale oil shale plants. The primary objective of this work was to provide a technical and economic data base for use by DOE in systems modeling and analysis of oil shale processing routes. Another objective of these conceptual designs was to identify process alternatives which could potentially reduce costs, improve operability, or minimize environmental impacts.

The overall contract scope included five conceptual design cases, to be executed at DOE's option, for the production of a nominal 50,000 BPD of syncrude based on processing run-of-mine shale in a grassroots facility.

- **Base Case:** Fluidized-bed retorting/combustion of Colorado Mahogany zone shale.
- **Option 1:** Alternate base case design employing an FCC concept of fluidized-bed retorting/combustion of Colorado Mahogany zone shale.
- **Option 2:** Directly heated moving vertical-bed retorting of Colorado Mahogany zone shale.
- **Option 3:** Fluidized-bed retorting/combustion of eastern U.S. New Albany Shale.
- **Option 4:** Directly heated moving vertical-bed retorting of eastern U.S. New Albany shale.

Foster Wheeler conducted and completed the first three case studies on Colorado shale. For each case study, the following technical definition was developed.
Selection of process units for the overall Integrated plant.

- Process unit designs, including process flow diagrams, material and energy balances, equipment specifications, and utilities requirements.
- Identification of process design uncertainties and recommendations for process alternatives.
- Specifications for plant support systems and description of the overall plant layout.
- Estimated capital investment for the Integrated plant, detailed by Individual process unit.
- Estimated overall operating requirements and annual operating costs for the plant.

Foster Wheeler's contract effort is essentially complete. Due to funding constraints, DOE has elected not to proceed with the Option - 3 and Option - 4 case studies.

ACCOMPLISHMENTS

Based on non-confidential information, designs were developed for commercial size retort modules employing fluidized-bed retorting and vertical moving-bed retorting of 30 gpt Colorado oil shale. Corresponding designs for downstream processing units were also prepared. The overall processing sequence produced a syncrude product having a maximum nitrogen content of 1000 ppm and a pour point less than 30°F.

Specifications for the major process equipment items were prepared for all plant sections from which detailed capital cost estimates were developed. Details of the conceptual plant designs and cost estimates for each case study were summarized in separate topical reports.

CONTRACT REPORTS


Tar Sand I
SESSION 2B

Chairperson:  T. Robert McLendon
Based on developed selection criteria three tar sand deposits were selected, samples from which were suitable for in situ and surface recovery process development. The sources of these samples were the Uintah county quarry near Vernal, Utah (Asphalt Ridge deposit); the Great Northern Corporation mine site near Price, Utah (Sunnyside deposit); and an outcrop near Edna, California (Arroyo Grande deposit). The following amounts of material were obtained from each of the deposits: Asphalt Ridge - six tons, Sunnyside - seven tons, and Arroyo Grande - eighteen tons.

The factors that were considered prior to the deposit selection included size of the deposit, oil saturation, and selected chemical and physical properties of the tar sand and the bitumen it contains. Other factors included presence of appropriate outcrops and current surface activity in the area.

With these considerations, deposits in California, Kentucky, New Mexico, Texas, and Utah were identified as possible sources of material. The Asphalt Ridge deposit was chosen first because of easy access to material and abundance of information available on the deposit. The county quarry near Vernal, Utah, is currently used as a source of nonspecification grade asphalt. The Sunnyside deposit was chosen second because of the size and richness of the deposit. The chemical and physical properties of the bitumen are similar to that at Asphalt Ridge but the tar sand material is consolidated while that at Asphalt Ridge is unconsolidated to semi-consolidated.

The deposits in Kentucky and New Mexico were eliminated as possible sources of samples. This was because the available sampling sites contained material that was too lean, averaging less than 6 wt % bitumen. The Anacacho deposit in Texas was eliminated as a possible source of a sample because the rock matrix is composed of limestone. Thus, this material is inappropriate for recovery processes involving combustion and possibly hot gas ingestion. The Casmalia deposit in California was eliminated as a possible source of a sample
because the rock matrix is composed of diatomite. Even though this reservoir rock has high porosity and oil saturation, the very low permeability (less than one millidarcy) make it unsuitable for in situ recovery processes.

The Arroyo Grande (Edna) and Zaca-Sisquoc deposits of California remained as possible sources of samples. Both deposits were considered to be of approximately equal ranking. However, the Arroyo Grande deposit contains a somewhat greater amount of measured resource than the Zaca-Sisquoc deposit. Consequently, the Arroyo Grande deposit was the third deposit selected.

Bitumen samples from these deposits were obtained by solvent extraction, and numerous chemical and physical properties were measured. The properties measured satisfy the needs of both process engineers and the people developing the mathematical models.

ACCOMPLISHMENTS

Selection criteria were developed and used to identify tar sand deposits that are suitable for in situ and surface-recovery process development.

Three deposits were selected (Asphalt Ridge, Sunnyside, and Arroyo Grande), and samples were obtained from each of them. The chemical and physical properties of these samples were measured.

FUTURE PLANS, ARTICLES, AND PRESENTATIONS


The continuation of this work in the future is necessary because it supplies samples and base-line data used in the development of thermal recovery processes.
ABSTRACT

The principle objective of this investigation was to acquire preliminary data that can be used to describe the chemistry and physics of the Recycle Oil Pyrolysis and Extraction (ROPE©) concept. This data will be used by the engineering staff responsible for conducting, evaluating and modeling the process demonstration unit (PDU). The screw feed PDU was conceptually divided into five processing zones; 1) tar sand pretreatment zone, 2) recycle oil pyrolysis zone, 3) recycle oil reflux zone, 4) residual bitumen pyrolysis zone, and 5) residual carbon combustion zone. The first three processing zones were targeted for the preliminary investigation performed under this project. The experiments were performed in a glass reaction system using Asphalt Ridge tar sand and an Asphalt Ridge tar sand derived recycle oil from previous PDU experiments. The recycle oil was spiked with four model compounds (perhydrofluorene, 9,10-dihydroanthracene, phenanthrene, and n-octadecane) to act as markers to better follow the chemistry of the concept. All of the experiments that were used to evaluate the concept had material balances closures between 99.52 and 100.42. Samples were collected during the course of each experiment and selected samples were submitted for analyses.

The tar sand pretreatment zone functions to solubilize the bitumen in hot recycle oil and distill a volatile product. This zone operates between 204 and 260°C (400 and 500°F) with residence times on the order of 15 minutes. The results from the experiments conducted in the bench scale reaction system indicate that the tar sand bitumen goes into solution within five minutes and that chemical reactions are minimal. The recycle oil pyrolysis zone functions to initiate the pyrolysis of the recycle oil/bitumen solution. This zone of the PDU operates at temperatures between 371 and 427°C (700 and 800°F). The residence times vary between 30 and 45 minutes. The results from the bench scale experiments indicate that pyrolysis does begin in this zone and that
coke and gas production are minimal. The recycle oil reflux zone of the PDU functions as an area where the recycle oil/bitumen solution is disengaged from the spent sand. Further pyrolysis reactions are also expected to occur. The recycle oil reflux zone operates at temperatures between 399 and 454°C (750 and 850°F) with residence times on the order of 20 to 30 minutes. The results from the bench scale experiments indicate that the majority of the coke and gas production occurs in this zone and that pyrolysis of the recycle oil and the bitumen continue at an accelerated rate.

ACCOMPLISHMENTS

A series of experiments were performed in a glass, bench scale reactor to provide preliminary data to describe the more important processing zone of the ROPE concept.

The results from these experiments indicate:

• that the solubilization of the bitumen in the recycle oil is relatively fast and will not be a limiting factor in processing Asphalt Ridge tar sand using this concept.

• the pyrolysis that occurs in the recycle oil pyrolysis zone occurs with a minimum of gas and coke production.

• the recycle oil reflux zone appears to act as both a disengaging area for liquid and solids and as an area for accelerated pyrolysis. The major gas and coke yields occur in this zone of the PDU.

FUTURE PLANS, ARTICLES, AND PRESENTATIONS


Future bench scale research efforts should be directed at performing experiments to:

• develop more detailed kinetics of the reactions that occur in each of the reaction zones.

• investigate the possible co-processing mechanisms (hydrogen transfer etc.) in more detail.

• investigate extinction of the recycle oil.

• investigate the effects of variation in the ratios of tar sand and recycle oil.

evaluate resources other than Asphalt Ridge tar sand.
THE EFFECT OF RECOVERY METHODS ON BITUMEN AND HEAVY OIL COMPOSITION

1. CONTRACT NUMBER: DE-AC20-85LC11071

CONTRACTOR: Western Research Institute
Box 3395
Laramie, Wyoming 82071

CONTRACTOR PROJECT MANAGER: Dr. Ken Thomas

PRINCIPAL INVESTIGATORS: Dr. K. P. Thomas

METC PROJECT MANAGER: James D. Westhoff

PERIOD OF PERFORMANCE: October 1, 1985 to December 31, 1987

ABSTRACT

The objectives of this investigation were three-fold: (1) to determine the chemical and physical properties of three sets of oil samples, each set composed of a primary and a thermally produced oil from the same pattern area; (2) to determine the potential of producing specification-grade asphalt from the three thermally produced oils; and (3) to compare the properties of the thermally produced oils with refiners' requirements. Data from numerous field projects were evaluated and, eventually, three projects were chosen for study. These projects were the Mobil Oil Corporation's steamflood at San Ardo, Santa Fe Energy Company's steamflood at Kern River, and also their fireflood at Midway Sunset. The first two projects were classified as heavy oil recovery projects and the third as a tar sand recovery project.

In all cases the chemical and physical properties of the three sets of oils were determined and they were related to the status of the thermal recovery projects. At the Mobil steamflood project the process is in transition. A majority of the oil bank has been produced and steam distillation of the remaining oil is becoming a more important production mechanism. The properties of the produced oil reflect this. The oil is improved in quality relative to the primary oil. The viscosity, specific gravity, and percentage of oil distilling above 538°C (1000°F) are all lower. However, at the Santa Fe steamflood the properties of the produced oil are degraded relative to the primary oil. In this case the viscosity, specific gravity, and percentage of oil distilling above 538°C (1000°F) are all higher, because steam breakthrough has occurred, the injection pressure has been reduced, and a hot water flood to recover additional oil is being conducted. This oil has fewer low boiling components because they were produced, to a certain extent, when steam distillation was occurring. The Santa Fe fireflood is a rather immature project. Consequently, few changes in the properties of the thermally produced oil were detected.
Distillation residues of the three thermally produced oils were prepared. All of the residues meet the ASTM D-3381 Table 1 requirements for viscosity-graded asphalts. However, the residues are too temperature susceptible to meet the more restrictive Table 2 requirements. In general, the results of the specification and non-specification tests indicate that the residues are very similar to normal petroleum asphalts.

The properties of the thermally produced oils are outside the acceptable range for direct refining. Consequently, they must be blended with lighter crudes before processing. As these lighter crudes become depleted, refineries will be forced to process heavier and heavier crudes. It is suggested that a refinery dedicated to the production of a syncrude suitable for existing refineries could fulfill an important role.

ACCOMPLISHMENTS

Identified and obtained three sets of oil samples (primary and thermally produced) from three in situ thermal recovery projects.

Analyzed the sets of samples in detail and correlated their properties with the status of each of the projects.

Prepared distillation residues from the thermally produced oils and evaluated their potential to produce specification-grade asphalts.

Compared the properties of the thermally produced oils with refiners' requirements.

FUTURE PLANS, ARTICLES, AND PRESENTATIONS

RECYCLE OIL PYROLYSIS AND EXTRACTION PROCESS

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PRINCIPAL INVESTIGATORS: Dr. Chang Yul Cha
Dr. Frank D. Guffey

METC PROJECT MANAGER: Dr. T. R. McLendon

PERIOD OF PERFORMANCE: May 1, 1986 to September 30, 1988

ABSTRACT

The objective of this work is to develop a process that produces (1) greater oil yields than other pyrolysis processes, (2) an upgraded feedstock, (3) a diluent to decrease the pour point of bitumen for pipelining, and (4) a solvent for extraction processes. Recent preliminary data from the ROPE© (Recycle Oil Pyrolysis and Extraction) concept suggest that processing tar sand with product oil recycling may achieve this objective.

The concept consists of two pyrolysis steps: (1) retorting tar sand at a lower temperature \( T < 420^\circ C \) with the bitumen and product oil, and (2) completing the pyrolysis of the residue at a higher temperature \( T > 420^\circ C \) in the absence of product oil recycle.

Experimental results from three tar sands, Asphalt Ridge (Utah), Sunnyside (Utah), and Athabasca (Alberta), indicate that oil yields from the ROPE© process will be greater than those from other pyrolysis processes. Differences were observed in the characteristics of the distillable liquid products from the three resources. These differences were evaluated in relation to the potential end uses of the products.

The ROPE© process will contribute to the mission of DOE by improving the oil recovery from tar sand and oil shale resources. The ROPE© process will assist existing surface and in situ recovery process economics by producing a diluent to decrease the pour point of bitumen produced from existing processes for pipeline transportation. The ROPE© process also produces a solvent for existing solvent extraction processes. The ROPE© process will produce the feedstocks from tar sand for the production of an advanced aviation fuel.

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ACCOMPLISHMENTS

Following is a list of major accomplishments in 1986 and 1987:

• Constructed a 6-inch diameter bench-scale unit and an inclined liquid fluidized-bed reactor system

• Eight tests were conducted using the Asphalt Ridge tar sand in a 2-inch PDU to determine the effects of pyrolysis temperature and residence time on the oil yield and product distribution.

• Two 48-hour tests were completed using the Asphalt Ridge tar sand in a 2-inch diameter PDU to measure the operating time required to reach steady-state with respect to product compositions.

• A 24-hour test was conducted using the Athabasca oil sand to obtain the preliminary data and to produce oil samples for the evaluation of using ROPE© product oil as a diluent to reduce the pour point of bitumen for transportation.

• The potential use of the distillable products was evaluated for the products obtained from the processing of Asphalt Ridge and Sunnyside tar sands.

FUTURE PLANS, ARTICLES, AND PRESENTATIONS

• Conduct 6-inch bench-scale reactor tests using Asphalt Ridge and other tar sand resources

• Investigate coprocessing of tar sand with heavy oil and bitumen using the ROPE© process

• Techno/economic evaluation of the ROPE© process

• Evaluate ROPE© product oil as a solvent for solvent extraction processes and diluent for bitumen transportation


FUELS PRECURSORS AND PRODUCT END USE POTENTIAL

CONTRACT NUMBER: DE-FC21-83FE60177
DE-FC21-86MC11076

CONTRACTOR: Western Research Institute
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CONTRACTOR PROJECT MANAGER: Mr. Lei and C. Marchant

PRINCIPAL INVESTIGATORS: Dr. K. P. Thomas
Dr. F. D. Guffey

METC PROJECT MANAGER: James D. Westhoff

PERIOD OF PERFORMANCE: October 1, 1985 to September 30, 1987

ABSTRACT

The primary purpose of these projects was to evaluate the product oils from three different experimental recovery processes. The first project evaluated liquid products from a process developed at Western Research Institute (WRI) called ROPE® (Recycled Oil Pyrolysis and Extraction). The second project evaluated the product oil from a simulated in situ wet forward combustion process. The third project evaluated the product oil (called TS-1S oil) from the DOE in situ tar sand steamflood project at Vernal, Utah. The first two projects used Asphalt Ridge tar sand and the third project used Northwest Asphalt Ridge tar sand.

The ROPE® process produced a light product oil and a heavy product oil from each of six different runs. In three of these runs, the light product oil was analyzed for fuel potential. Heavy product oil was not analyzed. The light oil products from these tests are significantly upgraded compared to the native bitumen or oil products from conventional processes. The light product oils are best suited for the production of diesel fuels.

The wet forward combustion oil was evaluated with respect to its potential to produce a specification grade asphalt and a high-density aviation turbine fuel. The distillation residue meets all of the ASTM D-3381 Table 1 specification tests for an AC-10 asphalt. The residue also has an unusually low aging index. This indicates not only that it may not set properly, but also it may be resistant to rapid age hardening.

The chemical and physical properties of the distillate are better than those of the original bitumen and the thermally produced oil. Analysis of the neutral fraction from the distillate indicates it is composed of predominantly aromatic structures of the 2- and 3-ring type. The saturate structures are primarily of the 3-ring type.
The TS-IS oil was evaluated for its potential to produce specification-grade asphalts and also as a potential feedstock for aviation turbine fuels.

Two distillation residues were produced, one at +316°C (+600°F) and one at +399°C (+750°F). However, only the lower boiling residue met ASTM specifications. The original oil sample met specifications as an AC-5 asphalt. These residue samples showed some unique properties in the area of aging; however, these properties need to be investigated further to determine the implications.

Two distillate samples were produced, one at IBP-316°C (IBP-600°F) and one at IBP-399°C (IBP-750°F). The chemical and physical properties of these samples were determined, and it was concluded that both samples appear to be potential feedstocks for the production of aviation turbine fuels. However, hydrogenation studies need to be conducted and the properties of the finished fuels determined to verify the prediction.

ACCOMPLISHMENTS

Analyzed a light product oil from the ROPE© process to determine its fuels potential.

Prepared and analyzed a distillate and a residue prepared from the product oil from a simulated in situ wet forward combustion process for the potential to produce high-density aviation fuels and a specification grade asphalt.

Prepared and analyzed two distillates and two residues from TS-IS oil produced at the DOE in situ tar sand steamflood project at Vernal, Utah for the potential to produce aviation turbine fuels and specification grade asphalts.

FUTURE PLANS, ARTICLES, AND PRESENTATIONS


EFFLUENT CHARACTERIZATION OF TAR SAND LABORATORY RECOVERY PROCESSES

CONTRACT NUMBER: DE-FC21-86MC11076

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METC PROJECT MANAGER: Dr. T.R. McLendon

PERIOD OF PERFORMANCE: April 1983 - Sept 1988

ABSTRACT

The effluents from several laboratory extraction process experiments of tar sand are being characterized. This program is part of an effort to obtain a data base of environmentally significant compounds resulting from tar sand processing, as defined by existing and proposed regulations under the Clean Air Act, Clean Water Act, Resources Conservation and Recovery Act, National Environmental Policy Act, and other legislation.

During previous years, effluents from a series of wet forward combustion tests that simulated in situ processing of Utah Asphalt Ridge and Sunnyside tar sands were analyzed for trace metals. In addition, sulfur species emissions were measured in the off-gas. In May 1988 effluents from wet forward combustion tests simulating in situ processing of Asphalt Ridge tar sand were characterized for trace metals and volatile and semi-volatile organics. The simulations were performed in WRI's tube reactor (one-dimensional combustion) or block reactor (three-dimensional combustion).

Hydrogen sulfide concentrations in the off-gas for the Asphalt Ridge test were as high as 8% by volume. The average equivalent sulfur dioxide emission for four Asphalt Ridge combustion tests was calculated to be 9.2 pounds per barrel of oil produced. The average equivalent sulfur dioxide emission for three Sunnyside combustion tests was 6.2 pounds per barrel of oil. Average nitric oxide concentrations in the off-gas were 0.11 and 0.016 pounds per barrel of oil for the Asphalt Ridge and Sunnyside tests, respectively.

Mass balances were completed on 28 elements for the in situ simulation tests. In most cases, elemental closure was between 80% and 120%. Generally, almost all of the trace metals remained in the combusted tar sand. A small amount of the trace metals also appeared in the process wastewater and produced oil. The off-gas contained measurable quantities of trace metals, but almost all of the trace metals were deposited in the off-gas lines and in the particulate filter ahead of the gas sampling train. An exception was gaseous mercury, where an
average concentration of 0.02 milligrams per standard cubic meter (equivalent to 2 milligrams per barrel of oil) was measured in the off-gas during Asphalt Ridge tar sand processing. The combusted tar sand from the tests passed the Environmental Protection Agency's extraction procedure toxicity test by at least an order of magnitude. This is a metals leachate test to determine whether a waste is hazardous as defined by the Resources Conservation and Recovery Act.

Volatile and semivolatile organic compounds were measured in the off-gas and in the produced water from a May 1988 wet forward combustion test of Asphalt Ridge tar sand. Toxicity characteristic leaching procedure tests were also completed on combusted and partially combusted Asphalt Ridge tar sand. The organic compounds analyzed were those elements of environmental concern under the Clean Water Act as defined by EPA test procedures 624 and 625.

ACCOMPLISHMENTS

Sulfur and nitrogen species emissions in the off-gas were measured during the course of eight tests simulating wet forward in situ combustion of tar sand. Trace metals were measured in the off-gas, process waters, and oils from five tests. Volatile and semivolatile organic species concentrations were measured in the off-gas and process water from one test.

ARTICLES AND PRESENTATIONS


FUTURE PLANS

Characterize gaseous, solid, and process water effluents of surface processing of tar sand, and relate emissions to resource and processing characteristics.
Oil Shale III
SESSION 3A
Mining, Material Handling and In-Situ Processes

Chairperson: Carl E. Roosmagi
OIL SHALE MINING AND MATERIAL HANDLING TECHNOLOGY ASSESSMENT

CONTRACT NUMBER: DE-AC07-76ID01570

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CONTRACTOR PROJECT MANAGER: Ms. Susan G. Stiger

PRINCIPAL INVESTIGATORS: Dr. Carroll F. Knutson
Dr. Richard P. Smith

DOE-ID PROJECT MANAGER: Mr. Dave M. Blanchfield

PERIOD OF PERFORMANCE: November, 1986, to March, 1988

ABSTRACT
This assessment was requested and funded by the U.S. Department of Energy (DOE) in order to analyze research needs and opportunities in oil shale mining and material handling (M&MH) technology. In addition, this assessment suggests areas of M&MH research that may warrant DOE support. Part of the assessment provides a review of costs associated with M&MH and production of shale oil. A major portion of this document (Section 4) reviews available M&MH technologies, including mechanical mining machines. Information was obtained through literature searches; attendance at symposia; and discussions with personnel in academia, government agencies, and the mining industry. Based on the information obtained, 58 M&MH research needs/opportunities were identified. The needs/opportunities were then presented to a committee of industry representatives, the probable users of successful research. These representatives prioritized the research needs/opportunities and suggested areas of research for DOE support.

ACCOMPLISHMENTS
The eastern and western oil shale resources, geometries and mechanical properties were reviewed.

The available cost estimates for oil shale M&MH were reviewed and evaluated as to the fraction they contributed to total project.

The oil shale M&MH technology was reviewed and evaluated through information obtained in literature searches, contacts with representatives of industry, manufacturers, service companies, state and federal government mining and ancillary research organizations, academia, and contacts at M&MH technical meetings and symposia.
A tabulation of perceived M&MH needs/opportunities was prepared from the technical information review, from a mining and excavation Research Institute (MERI) brainstorming session, and from an industry M&MH panel input.

An estimate of the economic impact of a research success was made for most of the needs/opportunities, via calculations involving surface or underground M&MH for a hypothetical 31,500 bbl/day shale oil project.

An industry oil shale M&MH panel representing interested segments of the energy industry, was convened. This panel evaluated the (1) probability of success; (2) probability of being carried out by industry without direct government support (under ordinary circumstances); (3) expected duration of Research and development effort; (4) probability that the estimate of economic impact of successful research was high, low, or reasonable; (5) impact on resource recovery; (6) environmental impact; and (7) overall research priority.

With the aid of the industry panel, M&MH needs/opportunities were prioritized from a DOE standpoint, and five priority M&MH research areas were suggested for DOE support.

- Oil shale/explosive (bit, cutter, etc.) interactions
- Oil shale cement
- M&MH model evaluation
- Geologic/hydrologic characterization
- Enhanced beneficiation studies.

FUTURE PLANS ARTICLES AND PRESENTATIONS


TECHNOLOGY DEVELOPMENT FOR BLASTING BASED MINING OPERATIONS

CONTRACT NUMBER: DE-AC04-76DP00789

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METC PROJECT MANAGER: Carl Roosmagi

PERIOD OF PERFORMANCE: October 1, 1986 to Present

ABSTRACT
A key step in the utilization of either coal or oil shale is the initial mining. Most mining operations are either entirely or partially based on rock fragmentation using chemical explosives. Despite the importance of the blasting step in mining, there remains considerable uncertainty concerning the fundamental mechanisms controlling blast results. The lack of fundamental understanding limits the amount of optimization that can be done for blasting since trial and error must be used and that is often cost prohibitive in large scale mining operations.

This research proposes to develop an overall computational model of the blasting process. Processes to be modeled include: detonation of the explosive, expansion of the explosive gases, fracture of the rock mass adjacent to the explosive including the calculation of particle size, translation of the fragmented material and final settling of the fractured rock. These features of the blast must be calculated as functions of the key blast design parameters: blastwell diameter and spacing, explosive type and loading, burden, and interwell spacing.

The approach being taken to this research involves the combination of engineering model development coupled closely with experimentation. The experimental program is particularly important because the complexity of the overall problem requires engineering assumptions that must be calibrated. The experimental program consists of large scale experiments in both single and multiple hole geometries as well as laboratory scale blasts where very accurate particle size distributions and damage zone profiles can be obtained.

The successful completion of this research effort will impact a number of energy related areas. The efficiency of oil shale resource development can be improved by providing a basis for optimizing blasting based mining operations. Similarly, coal resource recovery can be made more economical through improvements in blast designs. Also, recent research has shown the
potential for significantly improving in situ oil shale retorting. However, these improvements are only possible if precise control of the blasting results can be maintained. The development of computer models for blast design is one of the necessary components to achieving that control.

ACCOMPLISHMENTS

Significant accomplishments in the past year include:

A submodel for the direct prediction of particle size from the wave code was developed and implemented. This model incorporates elements of percolation theory and the known relationship of particle size to strain rate to provide a sound basis for predicting particle size distributions.

Another major advance was the completion of the first fully three dimensional fragmentation calculations. Since even the simplest practical blasting geometries (single well in a bench geometry and two adjacent boreholes) are three dimensional the development of the ability to perform routine three dimensional calculations is a significant advance.

In the experimental area as part of a joint program with industry, an extensive sequence of highly controlled cratering experiments was completed. Instrumentation on all the shots included detonation velocity measurements, high speed photography, surveyed muck and excavated crater profiles, particle size determination and some coring. This data base is currently being used to validate the two dimensional versions of both the fragmentation and motion codes.

FUTURE PLANS ARTICLES AND PRESENTATIONS


Future work will concentrate on completing the cooperative research program with industry. The second phase of this work calls for full scale instrumented experiments. In addition, direct experimentation in an oil shale mine is being planned. In the modeling area, further developments are needed to develop a three dimensional motion code, which includes gas pressure effects and extensive comparison of the fragmentation and motion codes with experimental data.
EVALUATION OF WATER-JET-ASSIST MECHANICAL CUTTING OF OIL SHALE

COOPERATIVE AGREEMENT NUMBER: DE-FC13-88GJ30000

CONTRACTOR: Alpine Equipment Corp.
             P. O. Box 767
             State College, Pennsylvania 16804
             (814) 238-1114

CONTRACTOR PROJECT MANAGER AND
PRINCIPAL INVESTIGATOR: Wilhelm J. Kogelmann

D.O.E. PROJECT MANAGER: Joseph E. Virgona

PERIOD OF PERFORMANCE: March 1, 1988 to February 28, 1990

ABSTRACT
The U.S. Department of Energy's Grand Junction Projects Office has entered into a $1.5 million Cooperative Agreement with Alpine Equipment Corp. of State College, Pennsylvania and Astro International Corp. of Beliefonte, Pennsylvania, for the evaluation of water-jet-assist (WJA) mechanical cutting of oil shale under actual mining conditions. Under the 24-months, three-phase Technology Project the contractor will design, assemble and field test a commercial-sized WJA mechanical mining machine. Field testing of the machine will be conducted at Exxon's Colony Pilot Mine located in the Piceance Basin of Western Colorado. Additional support will be provided by the Colorado Mining Association and the U.S. Bureau of Mines Pittsburgh Research Center.

OBJECTIVES
The evaluation under actual mining conditions will provide field data that can be utilized by the private sector to develop and commercialize water-jet-assist mechanized mining machines for use in oil shale. The main objectives for application of a WJA machine are (1) to improve safety and health conditions in underground mines through dramatically reduced dust levels and (2) to reduce excavation cost through reduced cutter bit (pick) consumption and lower repair and maintenance cost through reduced cutting vibrations. Furthermore, both water and energy consumption are to be minimized in order to achieve cost-effective oil shale extraction.

TECHNICAL PROGRESS AND PROBLEMS
Oil shale, a dolomitic limestone containing kerogen, has physical properties such as relatively high compressive and tensile strengths which made excavation with presently available production mining machines ineffective and expensive.

Improved mechanical production excavation technologies could make oil shale more competitive with other energy sources.

The following is a short outline of the current investigations performed in the project in Phase I "Equipment Design and Selection" which comprises a four months period.
Cutting tools (bits, picks) are the highest cost consumable in mechanized oil shale excavation. Various tool types and designs were evaluated in this project for WJA cutting of oil shale. The contractor will test tungsten-carbide, PDC, and thru-flush bits.

Water phasing to the cutter head, i.e., to restrict the flow of high-pressure-water (HPW) is applied only to jets which assist bits which are engaged in rock cutting. No water and energy must be wasted on bits which are not cutting rock. The lack of availability of a reliable, mine-tested HPW phasing system was until now a major obstacle in WJA cutting. The contractor selected a novel phasing system which has proven itself to be both energy-efficient and reliable.

High-pressure-water pumps are an essential part of a WJA cutting system. The suitability of plunger (piston) pumps and hydraulic pressure intensifiers for underground rock cutting applications was investigated. Furthermore a new system where the HPW pump is an integral part of the cutter head (drum) was evaluated.

Modification of an existing cutting machine is part of the project. The contractor selected a heavy-duty American-made roadheader (boom-type continuous miner) to be modified for WJA oil shale mining. Due to the high strength and toughness of the oil shale the existing machine's components such as cutter head transmission, boom structure, stabilizers, etc. had to be redesigned for large-scale underground cutting tests at the Colony shale oil project.

ACCOMPLISHMENTS

The contractor selected a flexible cutting system which will permit testing of WJA bits (picks) under a plurality of machine and HPW criteria.

A modern, mine-tested water-phasing system was selected for reliable and energy-efficient WJA cutting of oil shale.

The contractor studied extensively European-made WJA mining machines. The American-made machine designed under this D.O.E. contract is expected to exhibit improved performance compared to presently available WJA mining equipment.
IN SITU RETORTING STUDIES

CONTRACT NUMBER: Cooperative Agreement DE-FC21-86MC11076

CONTRACTOR: Western Research Institute
Box 3395, University Station
Laramie, Wyoming 82071

CONTRACTOR PROJECT MANAGER: Mr. Verne Smith

PRINCIPAL INVESTIGATORS: Mr. Norm Merriam
Dr. Chang Yul Cha

METC PROJECT MANAGER: Mr. Carl Roosmagi

PERIOD OF PERFORMANCE: May 1986 to December 1987

ABSTRACT

Four retorting tests were conducted to study the increase in oil yield resulting from the use of external fuel in in situ oil shale retorts having nonuniform permeability.

The results were compared to a baseline test where no external fuel was used.

Zones of contrasting permeability were created by loading particles of one size distribution into part of the retort and those of another size distribution into the remainder. Shale for the five tests was loaded using identical shale grade, particle size, and bed geometry to create duplicate shale beds. In the baseline test, S78, the shale was retorted using a gas flux of 0.875 scf of air and 0.375 scf of steam per square foot of bed cross section per minute. The remaining four tests, S80 through S83, were retorted using only the air flux of 0.875 to ensure that fuels could be burned.

Test S81 was conducted by injecting propane into the retorting air at the retort inlet. Test S82 was conducted by injecting fine coal particles into air flowing through the shale bed and retorting the bed after the coal was blown in the bed. Test S83 was conducted by injecting coal dust into the top of a zone of high permeability while retorting was in progress.

All four tests using external fuel produced higher yields than the test without added fuel. The tests using external fuel resulted in heating values of the incremental oil and gas produced exceeding the heating value of the fuel consumed. Three of the four tests using external fuel produced an incremental heating value of the oil exceeding the heating value of the fuel used. The net energy ratios for the four tests varied from 1.1 to 3.5.
ACCOMPLISHMENTS

Results of the retorting tests using external fuel showed that a low-cost fuel such as coal (and perhaps oil rich shale) can be used as fuel and that solid fuel can be blown into a retort by injecting dust into the inlet air stream.

FUTURE PLANS, ARTICLES, AND PRESENTATIONS


Merriam, N. W., Cha, C. Y., and Fahy, L. J. December 1987. Use of External Fuel to Improve Yields from In Situ Oil Shale Retorts with Nonuniform Permeability. Submitted to DOE.
3A.5

RETORTING. USING EUEX2IKCMAGNETIC ENERGY

CONTRACT NUMBER: DE AC20 85IC11060

CONTRACTOR: Professor Ramarao Inguva
University of Wyoming, Physics Department
Laramie, WY 82070

CONTRACTOR PROJECT MANAGER: Professor Ramarao Inguva

PRINCIPAL INVESTIGATORS: Professor Ramarao Inguva

METC PROJECT MANAGER: Carl Roosmagi

PERIOD OF PERFORMANCE: September 30, 1985 to October 1, 1988

ABSTRACT

The primary purpose of this project was to provide an in depth investigation (theoretical and experimental) of the feasibility of in situ electromagnetic heating of oilshales. The experimental work included setting up a microwave power delivery system, a data acquisition system, and construction of a reactor. A generator at 2.45GHZ and 3000 watts was purchased and a hybrid tuner was acquired. Data was stored on a H.P. 300 series computer which was connected to a Fluke data logger. High power heating tests were carried out on blocks of shale of dimensions (0.5 x 1.0 x 1.0) ft$^3$. The microwave power was applied by a monopole antenna. Temperatures, oil yields and gas analysis were obtained.

A theoretical model was developed to describe the heating process and an electromagnetic model of the monopole was developed. The model predicts temperatures, pressures, oil flow as a function of time. A dielectric measuring device was constructed using an open circuited transmission line. The device measures dielectric constants as a function of frequency and temperature. Both liquids and solids can be measured. A permeability device was also developed to measure absolute permeability of the shale as a function of temperature. The device uses the KLinkenburg technique for transient response. A model of fluid flow in weakly porous media has been developed and the model is currently being tested.

ACCOMPLISHMENTS

A microwave power system was developed along with a data acquisition system and a reactor was constructed. Electromagnetic heating experiments were performed and oil yields with monopole applicators were found to be on the order of 25%.

A numerical model of the fields due to a monopole was developed and interfaced with a heat-mass transfer model. The model results were compared to experiment and the results were in reasonable agreement with experiment.
The dielectric measuring device was constructed and was used to measure
dielectric constants of solid and liquid samples.

A permeability measuring device was constructed and various shale samples have
been measured.

FUTURE ARTICLES AND PRESENTATIONS

heating of oilshales, Fuel, June.

with a monopole antenna. Submitted JMP.

Baker-Jarvis, J. and R. Inguva. 1988. calculating current distributions on

AN IN-SITU PROCESS TO RECOVER HYDROCARBONS FROM EASTERN SHALES,

RESULTS OF HORIZONTAL RETORT PREPARATION

Mr. Victor Carr

Eastern Shale Research Corporation

This abstract was not available at the time of printing.
Tar Sand II
SESSION 3B

Chairperson: T. Robert McLendon
MODIFIED HOT WATER SEPARATION TECHNOLOGY

CONTRACT NUMBER: I. DE-FG22-84LC11057  
II. DE-FG21-88MC25046

CONTRACTOR: University of Utah  
Salt Lake City, Utah 84112.

CONTRACT PROJECT MANAGER: Dr. Alex G. Oblad

PRINCIPAL INVESTIGATOR: Jan D. Miller  
Alex G. Oblad

LETC PROJECT MANAGER: T. Robert McLendon

PERIOD OF PERFORMANCE: I. October 1, 1984 to December 31, 1987  
II. May 9, 1988 to June 28, 1988

ABSTRACT

Since the inception of the project our research efforts were mainly directed to the hot water recovery of bitumen from domestic tar sands. Tar sand deposits occurring in Utah are quite varied both in chemical properties and physical characteristics. The important property which greatly influences the recovery process is viscosity. Some Utah tar sands contain bitumens of highest viscosity known. Viscosity characteristics and other structural differences in domestic tar sands required development of an array of process strategies for the recovery of bitumen. Among these is the modified hot water process, which is very versatile and can be used for most deposits.

The most important factor involved in the modified hot water process is control of the bitumen viscosity in tar sands. This is accomplished by the addition of a diluent prior to the processing of tar sands. It has been determined that recoveries are vastly improved when the bitumen viscosity in the tar sand is adjusted to a value in the range 0.5-1.5 Pa.S. Kerosene has been the commonly used diluent. This method has been found to be extremely successful for tar sands containing bitumens of low and medium viscosities.

Application of the modified hot water process to tar sands containing high viscosity bitumen has not resulted with the same level of success. In an attempt to make this process more efficient, the nature and effect of diluent on the bitumen recovery is being evaluated. For this purpose a number of commonly used industrial solvents have been chosen and their relative efficiencies were determined from the viscosity measurements of bitumen-diluent mixtures. It has been found that of the diluents employed, SC#150, an aromatic hydrocarbon solvent, was the most efficient. Batch hot water
separation experiments were conducted on 4 kg samples of the highly viscous Sunnyside tar sand to verify the results obtained from viscosity experiments. In these tests SC#150 and kerosene were used as diluents.

In most of our earlier work a high shear energy reactor was used for the digestion of tar sand slurry. Since this type of reactor may not be desirable for industrial operations, a low shear energy digestion reactor was tested.

ACCOMPLISHMENTS

Successfully developed a technology to recover bitumen from tar sands containing lbw and medium viscosity bitumen.

Preliminary pilot plant studies by the private industry adopting this technology has been successfully performed.

In recognition of research contributions made in this field, the Taggart Award has been received from the Society of Mining Engineers.

Options on patents have been taken by private industry from the University of Utah for the evaluation of the tar sand technology.

PUBLICATIONS

/Hanson, F.V., Miller, J.D. and Oblad, A.G., "Process for Obtaining Products from Tar Sands," U.S. Pat. 4,337,143 (June 29, 1982).


Miller, J.D. and Sepulveda, J.E., "Separation of Bitumen from Dry Tar Sands," U.S. Pat. 4,120,776 (October 17, 1978).


3B.2

DUAL FLUIDIZED BED PYROLYSIS AND COMBUSTION

CONTRACT NUMBER:  I. DE-FG20-84LC11057
II. DE-FG21-88MC25046

CONTRACTOR:  Department of Fuels Engineering
University of Utah
• Salt Lake City, Utah 84112
(801) 581-6591

CONTRACT PROJECT MANAGER:  Professor Alex G. Oblad

PRINCIPAL INVESTIGATORS:  Francis V. Hanson
Alex G. Oblad

LETC PROJECT MANAGER:  T. Robert McLendon

PERIOD OF PERFORMANCE:  I. October 1, 1984 to December 31, 1987
II. May 9, 1988 to June 28, 1988

ABSTRACT

The influence of process operating variables on the product distribution and yields for the pyrolysis of bitumen-impregnated sandstone in a fluidized bed reactor have been investigated in both laboratory (1-1/2 inch diameter) and pilot scale (4-1/2 inch diameter) reactors. The quality of the liquid products has also been determined and related to the process operating variables.

The process variables investigated included pyrolysis reactor temperature, sand retention time in the pyrolysis zone of the reactor, the fluidizing gas velocity, and the average feed sand particle size. The ranges of the variables were as follows: temperature 723-973 K (450-700°C), feed sand retention time 15-35 minutes, fluidizing gas velocity 1-4 times the minimum fluidization velocity, and feed sand particle sizes from 359 micron up to 1/2 inch. In the case of the bench scale experiments, the reactor pressure and feed sand particle size were constant; the reactor pressure was maintained at atmospheric pressure and the average feed sand particle size was 359 microns. In the pilot scale experiments the feed sand particle size varied from 359 microns up to 1/2 inch. A variety of physical, chemical, and spectroscopic analyses was used to characterize the native bitumens and the bitumen-derived hydrocarbon liquids produced in the fluidized bed pyrolysis experiments. The tar sand deposits investigated included Sunnyside, Whiterocks, PR Spring, Tar Sand Triangle, and Circle Cliffs in the laboratory bench-scale reactor and Whiterocks and Circle Cliffs in the pilot-scale reactor.

The sand retention time appeared to be the most significant variable affecting the product distribution and yield of the bitumen-derived hydrocarbon liquid,
whereas the fluidizing gas velocity had little effect on the product
distribution, yields, and liquid product quality for the range of values
studied in the laboratory bench-scale reactor. The liquid product yield
increased with decreasing sand retention time; however, the coke-on-sand yield
was insensitive to changes in sand retention time. The gas yield increased
with increasing temperature while the liquid yield decreased. The coke yield
decreased as the reactor temperature increased up to 723 K and remained
constant as the reactor temperature increased above 723 K. The bitumen-
derived hydrocarbon liquid yield approached 55–60 wt% based on bitumen fed to
the reactor for the pilot-scale reactor when processing the crushed and sized
material from the Whiterocks tar sand deposit. This yield is consistent with
the data obtained for the Whiterocks tar sand in the laboratory bench-scale
reactor.

The liquid product quality and yield pattern indicated that the optimum
operating conditions should be those approximating visbreaking rather than
coking.

The kinetic parameters for the bitumen pyrolysis and the carbonaceous residue
combustion reactions have been determined for the PR Spring, Sunnyside and
Whiterocks tar sands. These data were obtained by TGA analysis for the case
in which the bitumen and the carbonaceous residue remained in contact with the
sand-mineral matrix during the pyrolysis and combustion reactions.

ACCOMPLISHMENTS

Completed the PR Spring Tar Sand tar sand deposit process variable study and
the characterization of the native bitumens and the produced bitumen-derived
hydrocarbon liquids.

Initiated the process variable study with the material from the Circle Cliffs
tar sand deposit and completed the characterization of the native Circle
Cliffs bitumen. Investigated the nature and composition of the Circle Cliffs
sand-mineral matter substrate to determine the source of the high yield of
water in the product slate.

Determined the kinetic parameters; that is, the activation energies and pre-
exponential factors for the pyrolysis of bitumen which remained in contact
with the sand-mineral matter substrate for the Whiterocks, Sunnyside, and PR
Spring Rainbow I tar sands.

Determined the kinetic parameters; that is, the activation energies and pre-
exponential factors for the combustion of the carbonaceous residue formed on
the sand grains during the fluidized bed pyrolysis of the Whiterocks,
Sunnyside, and PR Spring Rainbow I tar sands.
Completed preliminary testing of the large diameter (4.5-inch) fluidized bed pyrolysis reactor using the masons sand and the crushed and sized material from the Circle Cliffs and Whiterocks tar sand deposits.

Developed a mathematical model for the pyrolysis of bitumen-impregnated sandstone particles for the regime in which diffusion is the dominant transport mode (T > 600 K)

FUTURE PLANS, ARTICLES, AND PRESENTATIONS


HEAT PIPE PYROLYSIS AND COMBUSTION

CONTRACT NUMBER: DE-FG20-84LC11057

CONTRACTOR: Department of Chemical Engineering
University of Utah
Salt Lake City, Utah 84112
(801) 581-6916

CONTRACTOR PROJECT MANAGER: Professor Alex G. Oblad

PRINCIPAL INVESTIGATOR: Professor J. D. Seader

METC PROJECT MANAGER: James D. Westhoff

PERIOD OF PERFORMANCE: October 1, 1984 to December 31, 1987

ABSTRACT

The primary purpose of this project is to develop and demonstrate an efficient, economical, and operable process and apparatus, based on advanced heat-pipe technology, for recovering bitumen from tar sands. More specifically, the process is a fluidized-bed thermal process that produces a cracked bitumen, which, after hydrotreating for nitrogen removal, is a high-grade synthetic crude oil suitable for processing in a petroleum refinery. The process does not require solvents, process water, or the recycle of large amounts of sand as in other processes for bitumen extraction. The reaction apparatus consists of a single vessel divided into pyrolysis and combustion zones, each zone operating as a fluidized-bed reactor. Tar sand feed at ambient temperature and pressure is fed by a conveyor from a hopper down into the pyrolysis bed, operating at 450-525°C. These temperature levels are achieved by energy transfer from the combustion reactor to the pyrolysis reactor by heat pipes, which extend into and thermally couple the two reactors. In the pyrolysis bed, bitumen is thermally cracked to about 80 weight percent vapors and 20 percent coke, which remains deposited on the sand particles. The vapors pass to a condensing system, where approximately 90 percent of the vapor is condensed to oil. The coked sand flows downward to the combustion bed, where the coke is burned with air at 550-600°C, leaving clean sand. Energy remaining in the sand is used to make steam and preheat the air for combustion. The clean byproduct sand can be returned to the environment or used for other purposes, such as drilling or for making glass. The basic features of the process are covered in U.S. Patent 4,160,720 Issued to Seader and Jayakar. Some of the features of the process were demonstrated by operating a small 2-inch diameter by 10-foot high laboratory apparatus equipped with a single 3/4-inch diameter heat pipe. A total of 70 runs of about one-hour duration each were conducted with three different Utah tar sands at feed processing rates averaging four pounds of tar sand per hour. These tests demonstrated the ability of the heat-pipe concept to permit the
process to generate its own energy internally and to achieved predicted yields of synthetic oil. To study longer-term process operability, controllability, and the effect of multiple heat pipes, a 4-inch diameter apparatus with three 3/4-inch diameter heat pipes in parallel was constructed to process up to 15 pounds of tar sand feed per hour. To date, 15 test runs have been conducted with this larger apparatus on tar sands form the Sunnyside, Whiterocks, and PR Springs deposits in Utah. Yields of synthetic oil as high as 71 weight percent have been achieved. Temperature of the pyrolysis bed and bed levels have been controlled using a digital PID control. The pyrolysis bed has been maintained at a sufficiently high temperature solely by heat transfer from the combustion bed by the three heat pipes.

Based on the experimental results from the two laboratory units, preliminary process designs and economic evaluations have been prepared for plant capacities of 15,000 and 50,000 barrels per day of synthetic crude oil. For a feed of 10 weight percent bitumen, the production cost of the bitumen-derived oil, prior to hydrotreating, ranges from about $12 to $19 per barrel depending upon tar sand mining costs. The significance of the heat pipe process lies in its advanced technology, which overcomes many of the economic, environmental resource requirement limitations, and other problems hindering the commercialization of tar sand production in the United States. No major technical barriers exist for its successful implementation, but it is necessary to determine the optimal reactor configuration and operating conditions and demonstrate the process on a modest scale with several representative tar sands. Future activities involve detailed computer modeling of the process to predict the most efficient implementation, followed by design, construction, and operation of an intermediate-size unit based on the results of the modeling studies.

ACCOMPLISHMENTS

Developed a concept, utilizing heat pipes, for the thermal processing of tar sands in coupled fluidized beds, to produce a synthetic crude oil from bitumen. The process avoids the need for solvent, process water, and the recycle of large amounts of sand. Designed, constructed, and operated two laboratory processing units with four different Utah tar sands. Demonstrated the ability of the process to achieve oil yields of 70 percent. Demonstrated the ability of the heat pipe(s) to transfer the necessary heat from the combustion bed to the pyrolysis bed. Demonstrated the ability to control the operation of the beds. Prepared preliminary process designs and economic evaluations of commercial-sized plants to produce synthetic crude oil from Utah tar sands at from $12 to $19 per barrel.

ARTICLES AND PRESENTATIONS

TAR SAND BITUMEN UPGRADING

CONTRACT NUMBER: DE-FG21-88MC25046

CONTRACTOR: Department of Fuels Engineering
University of Utah
306 William Browning Building
Salt Lake City, Utah 84112-1183
(801) 581-8627

CONTRACT PROJECT MANAGER: Dr. James W. Bunger

PRINCIPAL INVESTIGATOR: Dr. Alex G. Oblad
Dr. James W. Bunger

METC PROJECT MANAGER: T. Robert McLendon

PERIOD OF PERFORMANCE: May 9, 1988 to May 8, 1989

ABSTRACT

The principal objective of this project is the development of engineering data required for pilot plant bitumen upgrading processes. It also has as its objective the support of the thermal and water extraction recovery technology under research at the University of Utah. Previous research has shown that hydropyrolysis is a prime candidate for upgrading of native bitumen such as that recovered by water extraction. Catalytic hydroprocessing has been shown to give satisfactory results for processing of thermally recovered products and for hydropyrolyzate. A pipeline acceptable synthetic crude oil can be produced by this sequence. Coking and visbreaking, while more conventional, result in unacceptably high losses to coke or insufficient upgrading in the case of visbreaking. The principal upgrading objective is to reduce the molecular weight without major losses of bitumen to less valuable coke or to gas. Research in hydropyrolysis has progressed to a point where a 2-liter per hour process development unit has been operated and results have shown that bitumen can be upgraded in 90+% yield to distillate products with low coke and low gas production. Hydrogen consumption is low and is in direct relationship to the amount of gas produced. The chemistry of the hydropyrolysis process has been examined. Kinetic pathways have been elucidated and reported in previous contract periods. What remains to be done is the development of an overall process model which takes into account the reaction kinetics and the physical phenomena associated with the heat and mass transfer within the reaction vessel. More extended runs are required to confirm operability of the process over longer periods of time. The combined hydropyrolysis/hydrotreating concept must undergo further design and testing to show its process viability relative to commercial application. The lowest amount of gas throughput which will still afford a low coke production has yet to be completely determined. This will be determined in the next phases of
our research. Preliminary economic analysis have shown that hydropyrolysis is a viable option because the high cost of mining and recovery of bitumen place a premium on high distillate yields. Current plans call for engineering design over a period of two to three years, resulting in a pilot plant design and field fabrication in a period of three to five years from now. Additional requests for funding for the out years have been made to complete the engineering design tasks indicated.

ACCOMPLISHMENTS

The contract is in its inception; however, the equipment and know-how of the previous 13 years' research will be duly utilized in the course of the current year's work.

PUBLICATIONS

All publications date to previous contracts and have been previously reported. Refer to:
3B.5

ROTARY KILN PYROLYSIS OF BITUMEN-IMPREGNATED SANDSTONE FROM THE TAR SAND DEPOSITS OF UTAH

CONTRACT NUMBER: DE-FG21-88MC25046

CONTRACTOR: Tar Sands Research Group
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Salt Lake City, Utah 84112
(801) 581-6591

CONTRACT PROJECT MANAGER: Professor Alex G. Oblad

PRINCIPAL INVESTIGATORS: Francis V. Hanson
Alex G. Oblad

LETC PROJECT MANAGER: T. Robert McLendon

PERIOD OF PERFORMANCE: May 9, 1988 to June 28, 1988

ABSTRACT

The rotary kiln pyrolysis of bitumen-impregnated sandstone from the tar sand deposits of Utah is being investigated as a complement to the fluidized-bed pyrolysis research program at the University of Utah. The objectives of the program are to generate a direct comparison between the product distributions and product qualities of the bitumen-derived hydrocarbon liquids produced by the two pyrolysis processes. The tar deposits to be evaluated include Whiterocks, PR Spring, Sunnyside, Tar Sand Triangle, Circle Cliffs, and Asphalt Ridge. The rotary kiln will be operated in a continuous-feed mode under isothermal conditions to facilitate comparisons to the fluidized-bed process.

The influence of process operating variables on the product distribution and product quality will also be determined. The rotary kiln operating variables of primary interest will be the pyrolysis temperature, the feed sand retention or residence time in the kiln, the vapor or sweep gas residence time in the kiln, and the kiln surface velocity. The operating pressure of the kiln will be several psia above the ambient pressure to prevent the introduction of air into the kiln.

Preliminary batch rotary kiln experiments have been conducted outside of this contract in cooperation with another investigator for a number of tar sand deposits at different heating rates and rotation velocities. Completion of the rotary kiln research program will enable those skilled in the art of process design to select the optimum operating conditions for both the rotary kiln and fluidized-bed pyrolysis processes. These data, coupled with complete characterization of the produced liquids, gases, and carbonaceous residues
will permit identification of the superior mining-surface thermal recovery process for each of the Utah tar sand deposits amenable to surface mining.

ACCOMPLISHMENTS

We have made arrangements to test the rotary kiln pyrolysis concept on a laboratory scale at a commercial facility prior to purchasing a rotary kiln apparatus. The kiln and tar sand feed system will be provided by the fabricator; however, we must provide a suitable product recovery system.

The product recovery train is being designed and fabricated at the University of Utah at the present time.

FUTURE PLANS, ARTICLES AND PRESENTATIONS

The pyrolysis runs will be conducted during a two-week period in September 1988. The tar sands to be evaluated in these tests will include Whiterocks, Sunnyside, PR Spring and/or Asphalt Ridge. The influence of temperature, feed sand retention time, sweep gas residence time, and kiln rotation velocity will be determined in a series of exploratory experiments. The ranges of the operating variables to be employed are being determined at the present time.

More than half of the estimated 50 billion barrels of U.S. tar sands are located in the state of Utah. Most of these resources are located on federal or state lands. The current state of knowledge with regard to Utah tar sands has been gained primarily by geologic field work supplemented by sparse, but important core drilling data. Many deposits have little core data to confirm the geologic estimates, while others, Asphalt Ridge for example, have considerably more core data available. The available information shows that the tar sand resource is a rather nonhomogeneous ore body varying considerably in grade and characteristics on a foot-to-foot basis. These variations will have a major, but currently not quantified, impact on the approach to recovery and upgrading technology and the economics of commercialization of this resource. Until these resources can be further assessed, commercial development will not occur. This project has as its primary objective the development of resource characterization and cost analysis data to support public land management decisions and to reduce the barriers to commercialization that are attendant with the lack of information on the resource base. The project is expected to be of a multi-year duration. It will assemble the known data on Utah tar sands in a single database location, prioritize the information requirements of interest to the federal government, and will update and modify the economic evaluation model developed at the University of Utah to reflect the current information. The model will be operated to assess the impact of specific public policy such as taxation, lease, royalty, and permitting issues on commercial viability. The model will be operated to determine the logical development units for specific technologies. In the initial phase of this work, available and pertinent data will be assembled, field samples will be gathered on deposits of high priority, and examined relative to currently known properties of interest to determine insofar as possible the recovery, processing, and upgrading characteristics of the samples. This information will be added to the economic evaluation model. The final year's results will provide the government with recommendations for future activities. It should be noted
that a single plant of 25,000 bbls/day of $25.00/bbl of asphalt or syncrude located on public resources would return to the federal government, by virtue of the 12.5% royalty, approximately $750 million over the life of the project. This appears to be an important incentive for the government to develop the required information on public resources it manages.

REFERENCES


SOLVENT EXTRACTION OF SOUTHERN U. S. TAR SANDS

Contract No: DE-FG21-88MC25043

Contractor: Board of Trustees
University of Arkansas
Department of Chemical Engineering
Fayetteville, Ar 72701

CONTRACT PROJECT MANAGER: Don Ousterhout

Principal Investigator: J. R. Couper

METC Project Manager: T. Robert McLendon

Period of Performance: May 15, 1988 to May 14, 1989

ABSTRACT

The Department of Chemical Engineering at the University of Arkansas in cooperation with Diversified Petroleum Recovery, Inc. (DPR) of Little Rock, Arkansas have been developing for the past four years a fatty acid solvent extraction process for the recovery of bitumen from tar sands. The fatty acid solvent is recovered for recycle purposes by an amphiphilic phase behavior process known as the Wood-Beaver process.

The Mineral Research Institute (MRI) at the University of Alabama, Tuscaloosa, has been developing a pretreatment beneficiation process for domestic tar sands that upgrades relatively low grade tar sands to relatively high grade tar sands. The basis of the beneficiation is primarily fine grinding followed by flotation in which a large fraction of the original mineral content of the feed tar sand is rejected.

The overall purpose of the proposed project herein is to initiate a study of both the economical and technical feasibility of merging these two technologies with specific application of the integrated process to Northern Alabama Tar Sand deposits. The specific objectives of the work plan are to: (1) conduct batch scale solvation studies of various solvent blends with beneficiated and raw Alabama tar sand samples with emphasis on solvent retention on the spent sand and tar sand feed/solvent contacting methods, (2) conduct grinding and flotation studies on the samples, (3) conduct bench-scale Wood-Beaver solvent recovery studies to ascertain the effect of process variables on the amount and characteristics of the bitumen recovered, (4) to evaluate the economic potential of the process with a variety of market values of the derived product, and (5) the amount of solvent retained in the product bitumen.

It is important to make the studies mentioned above in order to properly evaluate the integrated process in terms of quality of product bitumen recovered, solvent losses to spent sand, and process enhancement due to beneficiation. It is anticipated that additional funding will be necessary to actually develop a bench-scale unit of the fully integrated process.
ACCOMPLISHMENTS

Project personnel have been recruited and signed-on to the project. The laboratory has been cleaned and purchase orders for the necessary supplies and equipment have been issued. An in-depth organizational meeting has been held. We are currently awaiting the arrival of the first tar sand samples from Northern Alabama in order to proceed with the laboratory testing. A thorough literature search has been conducted on solvent extraction and solvent separation from sand and initial experiments in this area have been designed.

FUTURE PLANS, ARTICLES AND PRESENTATIONS

Two papers have been accepted for presentation from previous work prior to this contract which will include results achieved this upcoming summer. They are:


Oil Shale IV
SESSION 4A
Environmental R&D

Chairperson: Carl E. Roosmagi
A mature oil shale industry would generate over 10 billion ft$^3$ of solid waste per year. This could cover an area 75 mi* to a depth of 150 ft after 30 years. To be environmentally and economically acceptable, processed shale waste disposal must: result in physically stable structures, prevent or minimize release of toxic compounds, and provide an economically acceptable post land use. This research program was designed with this goal in mind.

The purpose of this study is to physically model and observe three interacting factors which would affect the disposal and behavior of processed oil shale embankments and to compare these observed physical data to computer generated data in an evaluation of model performance. These three factors are: 1) hydrology, 2) geotechnical stability, and 3) geochemistry. An associate purpose of this research is to conduct these investigations at a scale that is sufficient to describe commercial scale embankment behavior. There are five major objectives: 1) Assess the unsaturated movement and redistribution of water and the development of potential saturated zones in disposed processed oil shale waste; 2) Assess the unsaturated movement of solubles and major chemical constituents in disposed processed oil shale; 3) Assess the physical and constitutive properties (index properties, shear strength, stress-strain properties, and compressibility of processed oil shale and determine potential changes in these properties caused by disposal and weathering; 4) Assess the use of previously developed computer model(s) to describe the infiltration, unsaturated movement, redistribution, and drainage of water in disposed processed oil shale; and 5) Evaluate the stability of theoretical, commercial
scale, processed oil shale waste embankments using computer models. This program is being conducted in cooperation with Rio Blanco Oil Shale Co., Inc., a subsidiary of Amoco Corp., Exxon USA, (Colony Group), the University of Wyoming, and the Department of Energy.

The research program combines the operation of three large scale field lysimeters (24 ft x 10 ft x 10 ft deep), the operation of two large scale laboratory lysimeters (24 ft x 10 ft x 10 ft deep) enclosed by a large scale environmental chamber, and a bench top research and testing program. Field lysimeters are located at Rio Blanco Oil Shale Co., Inc. Tract C-a, in the Piceance Basin of Colorado. The laboratory lysimeters with the combined environmental chamber are located at the University of Wyoming, Laramie. Besides providing temperature and humidity control, this environmental chamber includes rainfall simulation and solar radiation. All lysimeters are monitored, in situ, for soil water, vertical and horizontal stress, leachate chemistry, and temperature at several depths. The bench top testing program has been designed to characterize the processed waste for initial hydrologic properties, initial soil mechanical properties with geochemical interactions, initial chemistry of solubles, and to evaluate on going or potential changes in these properties caused by disposal and weathering in the lysimeters.

ACCOMPLISHMENTS

Identified a 500 ton source of decarbonized processed oil shale waste material with Rio Blanco Oil Shale Co., Inc. and formalized a contract for cooperative research between this company and the University of Wyoming in July 1987.

Built and installed three lysimeters at Rio Blanco Tract C-a, emplaced processed oil shale and instrumentation monitoring in situ data, and installed the remote meteorologic station and data logging system. (September 1987 - December 1987).

Initialized bench top testing to define initial hydraulic and soil mechanical properties (September 1987).

Completed construction and modification of the indoor lysimeters and environmental chamber at the University of Wyoming; equipment testing has been initialized. (July 1988).

FUTURE ACTIVITIES

Emplacement of processed oil shale in the University of Wyoming indoor lysimeters and environmental facility.

Continuation of bench scale testing program in hydrology, soil mechanics, leachate chemistry, and geochemistry.

Initialization of objectives and tasks associated with computer modeling.
FIELD AND LABORATORY LEACHING STUDIES OF RETORTED KY OIL SHALE

COOPERATIVE AGREEMENT: DE-FC21-84MC21144

CONTRACTOR: University of Louisville,
Kentucky Energy Cabinet Laboratory
P.O. Box 13015, Lexington, KY 40512-3015

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METC PROJECT MANAGER: Carl Roosmagi

PERIOD OF PERFORMANCE: June 1, 1983 to June 1, 1988

ABSTRACT
The purpose of this project is to provide data on the leaching characteristics of raw and retorted oil shale from the eastern U.S. in a variety of disposal configurations. The data have been obtained from laboratory column techniques, and from field lysimeters located in the oil shale outcrop belt of Kentucky. These data will aid understanding of mechanisms and kinetics of leaching of eastern U.S. oil shales and related materials, will form a baseline of data for evaluation of disposal and reclamation options for oil shale operations, and will compare the leaching characteristics of materials produced by advanced retorting technologies. The objective of the laboratory studies was to develop techniques which produce results comparable to those from the field. Laboratory tests are important in the present energy-research environment, where large retorting tests are quite rare. Furthermore, with environmental evaluation of new retorting technologies as an important impetus to design, bench-scale leaching tests are required. In almost all cases, the leachate parameters from EPA and ASTM batch tests met RCRA requirements. Batch tests on Kentucky oil shale are inadequate because of low solid-to-liquid ratios and short duration. Thus, they underestimate extractable ions. The column tests used packing densities watering regimes which closely simulated field conditions. Properties of the leachates from the column tests were in good agreement with those from the field lysimeters. In addition, elemental release patterns from the laboratory columns followed closely those from the field. In 1983, approximately 1200 tons of 1/4" by 1" oil shale (33% Sunbury Shale, 67% Cleveland) was retorted in the Dravo traveling grate pilot plant in Cleveland, OH. The retorted oil shale and the rejected fines were returned to Montgomery County, KY, near the mine. The oil shale, soil and overburden materials were placed in eight 640-cubic-foot concrete lysimeters which were constructed especially for this project. Slotted drainage tubes connected to collection vessels in two central sample chambers were implaced as the lysimeters were filled. Several fill plans based on varying amounts of raw and retorted oil shale, overburden and soil were
used to evaluate effects of different disposal options. Sample weight and pH were determined in the field, and a small aliquot was taken for laboratory analyses. Lab work consisted of a second pH measurement, conductivity, As, B, Ca, Cd, Cr, Cu, Fe, K, Mg, Mo, Mn, Na, Ni, Pb, Se, Zn, Al, and sulfate. All elements were present above detection limits except Pb (0.5 ppm detection limit), As (0.5 ppm), Mo (0.2 ppm), and Cu (0.1 ppm). All of the material combinations produced acidic leachates with pH's ranging from 2.5 to 3.6 over the study period (July 1984 - May 1988). The leaching patterns of the raw shale declined comparatively rapidly in concentration, but concentrations of the retorted and mixed shales declined more gradually. The leachate composition from the retorted oil shale had markedly high concentrations of Al, K, B, and Na compared to the leachate from the raw oil shale. Acid-mine drainage problems would arise very rapidly from all combinations of materials used in this study if not properly handled.

ACCOMPLISHMENTS

Demonstrated a laboratory leaching test using glass columns, field densities, and natural watering regimes, closely replicating elemental release patterns of the field lysimeters with close approximations to quantitative results.

Provided four years of leachate data from retorted Kentucky oil shale, reject fines, overburden, and soil materials in several mine reclamation disposal configurations using eight concrete lysimeters located in the oil shale outcrop belt.

From the above data base we concluded that all of the disposal configurations produced acidic leachates with high concentrations of dissolved ions, the elemental release pattern for the retorted oil shale is more complex than for raw, and retorting apparently changes the structure of the clays, and in so doing allows for readier dissolution and the release of K and Al.

FUTURE PLANS ARTICLES AND PRESENTATIONS


ABSTRACT

The primary objectives of the research conducted under this task have been to directly determine the sorption behavior of soluble organic species on mineral surfaces by identifying the bonding mechanisms using vibrational spectroscopy and chromatographic modeling. The research strategy has been to utilize model organic compounds and mineral phases to determine the specific sorption mechanisms between the organics and minerals' based on the functional group and moiety interactions and competition. The information derived from these studies can be used to help predict the behavior of organic compounds of similar conformation on minerals in more complex natural mixtures as well as creating a greater understanding of specific sorption behavior on solid waste. The instrumentation utilized to accomplish this task has been Fourier transform Infrared spectroscopy and high performance liquid chromatography, and investigations in the use of Raman spectroscopy, differential scanning calorimetry, and thermogravimetric analysis for this purpose. To meet these objectives, several studies have been undertaken utilizing mineral phases and organic model compounds either resembling in functional group composition or will be actual organic compounds found in raw and/or spent oil shales. First, a study was undertaken to determine what, if any, effect the various infrared sample preparation techniques had on the analysis of the sorption interactions, a subject of conflict in the literature. Studies into the chemisorption mechanisms between montmorillonite clay and pyridine, isopropylamine and dimethyl methylphosphonate, benzene, 2,2,4-trimethylpentane, benzoic acid or sodium benzoate were performed. The same series was performed on the mineral hematite. Results from the analysis of the infrared band shifts indicated that sorption took place between montmorillonite and pyridine, isopropylamine, and dimethyl phosphonate, and between hematite and isopropylamine, indicating that the primary sorption between the bare mineral and organic compounds took place with the more polar organic compounds. The use of FTIR spectroscopy indicated that the newer instrumental methods of photoacoustics and diffuse reflectance were especially suited for this type of study, although contrary to previous literature.
conducted on dispersive instruments, there was no significant difference in the use of any sampling technique. High performance liquid chromatography indicated some interaction between pyridine and montmorillonite clay. However, this is a difficult experimental method requiring further development. Raman spectroscopy, using a customized instrument was found to be better suited for a remote, in situ method for monitoring organic contaminants in water, and for which a patent was applied for. Thermogravimetric and differential scanning calorimetry were found to be too insensitive for this application. The results of these studies have indicated that FTIR is especially suited to this study but that further instrumental and technique development is required.

ACCOMPLISHMENTS

In addition to the results previously mentioned, it is apparent with the use of FTIR techniques and with further development of the aqueous-mineral HPLC columns that specific information on the organo-mineral sorption mechanisms can be meaningfully generated and can be used to provide basic information to be utilized into the transport of the organic compounds in solid waste with groundwater flow.

FUTURE PLANS, ARTICLES, AND PRESENTATIONS

With the use of FTIR/PAS and DRIFT the sorption mechanisms between more appropriate minerals and other organic species will be examined. The instrumental techniques and software abilities now available will enable better resolution and more data to be accumulated for this project. Further, studies into the use of mixtures of organics and mineral phases can now be done. Further developmental work into the aqueous-mineral HPLC columns will continue.


INORGANIC GEOCHEMICAL CHARACTERIZATION OF SOLID WASTE

CONTRACT NUMBER: Cooperative Agreement DE-FC21-86MC11076

CONTRACTOR: Western Research Institute
Box 3395, University Station
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PRINCIPAL INVESTIGATORS: Dr. Michael E. Essington
Dr. Katta J. Reddy

METC PROJECT MANAGER: Mr. Carl Roosmagi

PERIOD OF PERFORMANCE: October 1986 to September 1992

ABSTRACT

The primary objectives of research conducted under this task were to provide a chemical characterization of spent oil shale and spent oil shale aqueous extracts, to evaluate experimental and geochemical modeling techniques that would identify the processes controlling major, minor, and trace element concentrations in spent oil shale leachates, and to evaluate methods of predicting spent oil shale leachate quality. To meet these objectives, several studies have been undertaken using retorted and combusted western and eastern reference oil shales. The mineralogy of spent oil shales have been examined through the utilization of nondestructive mineral preconcentration techniques, X-ray diffraction analysis, and scanning electron microscopy. The chemical characteristics of spent oil shale aqueous extracts have been examined using the equilibrium solubility method. This characterization, in addition to providing information on the solubility of chemical constituents, has allowed for the indirect characterization of minor mineral phases through the application of chemical thermodynamics. The distribution of elements into various spent oil shale physicochemical separates and selective extraction solutions has provided information on the mineralogical residencies of trace elements. The adsorption of arsenate and selenite by spent western oil shales was correlated to some of the basic physical and chemical properties of the solids, such as, specific surface area iron oxide content. Further, these experiments indicate that arsenate and selenite are irreversibly adsorbed by the spent oil shale solids and that precipitation reactions may also occur. The use and development of predictive models requires a critically reviewed thermodynamic database. To date, the thermodynamic data for strontium, molybdenum, fluoride, and selenium have been reviewed and included for use in geochemical models. In addition, barium arsenate solubility studies have been completed. Currently, the thermodynamic data on alkaline earth and alkali metals, and the equilibrium solubility of calcium molybdate in spent western oil shales, is in progress. A preliminary study to identify the types and concentrations of secondary minerals forming in hydrated Rio Blanco oil shale
has just been completed. The data, obtained by X-ray diffraction and scanning electron microscopy, suggest that secondary mineral formation and the physical strength of the cores are dependent on the length of time the spent oil shale is saturated with water. The results of this preliminary experiment were used to design an experiment to correlate mineralogy and spent oil shale physical strength.

ACCOMPLISHMENTS

In addition to those results listed above, the information gained through the chemical and mineralogical characterization of spent western oil shales has been used to develop a method to predict trace element concentrations in spent oil shale leachates. Experimental data has indicated that the soluble concentrations of fluoride are controlled by the precipitation of calcium fluoride. Using this information, the concentrations of major anions and cations, the ion association characteristics of fluoride in spent oil shale leachates, and a geochemical model, fluoride concentrations in retorted and combusted western oil shales were predicted. The resultant concentration values were comparable to the analytically determined concentrations.

FUTURE PLANS, ARTICLES, AND PRESENTATIONS

The mineralogical and solubility characterization of secondary mineral formation and weathering reactions that result from hydration and recarbonation of spent oil shales will be conducted. Further, the influence of these weathering reactions on leachate chemistry will be examined. Geochemical model evaluation will continue, as will the generation and review and selection of quality thermodynamic data. Chemical and mineralogical support will also be provided to the University of Wyoming on the Rio Blanco field and laboratory lysimeter studies.


EVAPORATION PROCESS ANALYSES AND EMISSIONS FROM OIL SHALE RETORT WATERS

CONTRACT NUMBER: DE-AC20-84LC11049

CONTRACTOR: CIVIL ENGINEERING DEPARTMENT
UNIVERSITY OF WYOMING
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METC PROJECT MANAGER: RICHARD MCQUISTEN


ABSTRACT

The primary objective of this research program was to study chemical, micro-climatological, and interactive effects on the evaporation of low-quality oil shale retort waste waters to develop more applicable evaporation models and evaporation design criteria for the disposal of oil shale process waters. A supplementary goal of this project was to analyze the processes associated with the release of potentially toxic emissions from these low-quality effluents. The research program incorporates field and laboratory studies analyzing microclimatic and chemical effects on the evaporation of oil shale process waters.

Field Studies were designed to continuously monitor microclimatological conditions and the evaporation from three low-quality effluents using Class A evaporation pans. Fresh water evaporation was monitored as a control. Process waters were being routinely monitored for concentrations of organic and inorganic constituents. Laboratory studies were designed to isolate and describe significant climatic, chemical, and interactive effects on evaporation rates. Results from the above studies were utilized to develop a regression model to predict evaporation from these low quality effluents. This model was then compared to commonly utilized models to estimate evaporation. A stochastic model was developed using a first order markov process to generate 1000 20 year climatological records. Mass balance techniques were then used to evaluate the new data sets for evaporation processes and determine critical design parameters for evaporation disposal ponds. Laboratory studies were conducted to determine significant effects on the Henry's Law Constants for 7 organic compounds in two process waters. Henry's Law Constant is a necessary component to estimate or model the emission of organics from
these low quality effluents.

ACCOMPLISHMENTS

- Field studies analyzing the evaporation rates from oil shale process waters and determining microclimatic effects have been concluded.
- Laboratory studies used to determine significant microclimatic and chemical effects have been concluded.
- A regression model was developed and compared to historical models for accuracy in predicting evaporation rates from oil shale process waters. This model was compared to historical evaporation models for accuracy in predicting evaporation from process waters.
- Laboratory studies to determine significant effects on Henry's Law Constants for 7 major organic constituents found in oil shale process waters has been completed.
- Significant evaporation pond design parameters were determined using stochastic modeling (first order markov process) and a mass balance technique to estimate evaporation on the generated climatic data.

ARTICLES AND PRESENTATIONS


GROUNDWATER STUDIES AT RIO BLANCO

CONTRACT NUMBER: Cooperative Agreement DE-FC21-83FE60177
Cooperative Agreement DE-FC21-86MC11076

CONTRACTOR: Western Research Institute
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METC PROJECT MANAGER: Carl Roosmagi

PERIOD OF PERFORMANCE: October 1984 to September 1988

ABSTRACT

In 1974, the Rio Blanco Oil Shale Project was granted a lease for tract C-a near Rifle, Colorado, for the purpose of developing the Green River oil shale formation. Mine and surface retorting facilities were constructed, and two in situ retort experiments were conducted in 1980 and 1981. In an effort to understand the environmental impacts of in situ oil shale retorting, and to assess mitigation strategies for these activities, Western Research Institute has conducted several studies related to Tract C-a. These studies include:

Characterization of the retort water by gas chromatography/mass spectrometer;
Characterization of the retort water before and after treatment;
Evaluation of baseline conditions at the site;
Toxicological evaluation of the groundwater samples;
Evaluation of microorganism located at tract C-a;
Identification of indicator species by gas, liquid, and ion chromatography.
ACCOMPLISHMENTS

A number of tasks were undertaken in the Rio Blanco studies.

The chemical, physical, and toxicological characteristics of the retort water were measured. By studying the microbiological activity in the water and soil at the site, WRI assessed the potential for biological degradation of organic constituents.

Toxicity assays and gas, liquid, and ion chromatography were used to evaluate the migration of organics away from the retort.

The baseline groundwater-quality data collected from lease tract C-a were also evaluated.

FUTURE PLANS, ARTICLES, AND PRESENTATIONS


OIL SHALE PLANT SITING METHODOLOGY

CONTRACT NUMBER: Cooperative Agreement DE-FC21-83FE60177
Cooperative Agreement DE-FC21-86MC11076

CONTRACTOR: Western Research Institute
P.O. Box 3395, University Station,
Laramie, WY 82071

CONTRACTOR PROJECT MANAGER: Mr. Verne Smith

PRINCIPAL INVESTIGATORS: Dr. John Nordln

METC PROJECT MANAGER: Mr. Carl Roosmagi

PERIOD OF PERFORMANCE: October 1986 to September 1988

ABSTRACT

The objective of the Initial work on this task was to develop a plan for addressing issues relating to oil shale plant siting. The second objective is to develop a guide for the numerous permits and approvals required to locate, construct, and operate an oil shale plant.

The plan for the oil shale plant siting methodology has been completed. The guide to permits and approvals that is preparation categorizes Institutional requirements into (1) land use, mineral leases, and rights-of-way permits; (2) acquisition of a water supply; (3) major multipurpose permits such as the environmental Impact statement; (4) environmental permits for protecting air quality, water quality, wildlife values, and for waste disposal; (5) historic and cultural protection permits; (6) mining; (7) health and safety; (8) transportation and communications; and (9) socioeconomics Including Industrial siting. Permits and approvals required from federal agencies, state agencies in six states, and selected county agencies are considered.

Flow diagrams are being developed that illustrate the approval process for the various permit categories and subcategories. Emphasis is on the Interaction between the developer and the various regulatory agencies and general public. The Colorado Joint Review process is already being addressed.

Examples of permitting requirements are being developed for a hypothetical 50,000 barrel-per-day (bpd) surface oil shale plant in Rio Blanco County, CO, and a 5,000 bpd demonstration plant in eastern Montgomery County, KY.

Finally, major Impediments to oil shale development are being Identified.
ACCOMPLISHMENTS

The plan for the oil shale plant siting methodology identifies the available resources in terms of location and quantity of oil shale, water, typography and transportation, and human resources. Restrictions on development are then addressed, e.g., land ownership, land use, water rights, environment, socioeconomics, culture, etc. Descriptions of technologies for development of oil shale resources are included. The impacts of oil shale development on the environment, socioeconomic structure, water availability, and loss of resource are discussed. The plan includes an extensive bibliography of published and unpublished information and also identifies data gaps and research needs.

FUTURE PLANS AND PUBLICATIONS

The guide for permits and approvals relating to oil shale plant siting, construction, operation, and closure is scheduled to be completed in August 1988.

THIRD ANNUAL OIL SHALE,
TAR SAND AND MILD GASIFICATION
CONTRACTORS REVIEW MEETING

JULY 19-21, 1988

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Annual Oil Shale, Tar Sand, and Mild Gasification Contractors Review Meeting

July 19-21, 1988
Morgantown, West Virginia

Sessions Attended

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What sessions were you most interested in attending? (Rank hi order 1 to 6 -1 being most interest in attending)

- ______ Oil Shale Properties and Behavior
- ______ Mild Gasification
- ______ Oil Shale Surface Processes R*D
- ______ Tar Sand
- ______ Mining, Material Handling and In-Situ Processes
- ______ Environmental R&D

Length of Meeting:

- ______ Too Long
- ______ Too Short
- ______ Appropriate

Meeting Format:

- 20-Minute Presentations and 5-Minute Question and Answer
- ______ Too Long
- ______ Too Short
- ______ Appropriate

Concurrent Sessions

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<tr>
<td>Average</td>
</tr>
<tr>
<td>Poor</td>
</tr>
</tbody>
</table>
Quality of Speakers:

________ Excellent

________ Average

________ Poor

Quality of Facility (Lakeview Sheraton):

________ Excellent

________ Average

________ Poor

Quality of Conference Staff:

________ Excellent

________ Average

________ Poor

Why did you attend?

________ Contractor - made presentation.

________ Interested in topic.

________ Networking - find a job and meet colleagues.

________ METC employee - made presentation.

Quality of media used during presentations (slides, tapes, posters):

________ Excellent

________ Average

________ Poor

Will you attend next year’s meeting?

________ Yes

________ No

________ Not sure, depends on topics/speakers.

Suggestions for Improving next year’s conference: