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IS - 4283

COAL BENEFICATION
PROGRAM REVIEW

MASTER

AMES LABORATORY, DOE
IOWA STATE UNIVERSITY
AMES, IOWA

Submitted November 9, 1977

PREPARED FOR THE DEPARTMENT OF ENERGY
UNDER CONTRACT W-7405-eng-82

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Ames Laboratory, DOE
Iowa State University
Ames, Iowa 50011

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INTRODUCTION

This report is written to acquaint DOE personnel with the coal beneficiation program of the Ames Laboratory, Iowa State University. It appears that the involvement of the Laboratory in this area is not widely known, probably owing to the fact that much of the work performed here and the facilities developed here were initiated with state, rather than federal, support as part of the Iowa Coal Project. At this time, however, the Laboratory has committed itself to participation in a continuing and substantial fossil energy effort, much of which is integrated with and dependent upon a growing program in coal beneficiation. In order to sustain and encourage this program growth to help meet national energy needs, support and cooperation from the Department of Energy are essential.

BACKGROUND

The Iowa Coal Research Project was initially funded by the legislature at \$3,000,000 for a three-year period beginning July 1, 1974. Additional funding of \$500,000 was secured for the period July 1, 1977 through June 30, 1978. The project has been administered by the Energy and Mineral Resources Research Institute (EMRRI) at Iowa State University which is also the contractor for the Ames Laboratory. Primary goals of the mission centered on the feasibility of mining coal in an economically and environmentally acceptable manner and on demonstrating methods for improving the quality of Iowa coal by beneficiation to reduce the content of sulfur and other impurities in accordance with environmental standards. Supporting goals included characterization of Iowa coal, including character alteration by processing and development of new methods for chemical desulfurization and other chemical processing of coal.

The Ames coal beneficiation research facility was completed during the spring of 1976 and included a 60' x 120' x 32' steel frame building, crusher, prewasher, heavy media separation unit, concentration tables, dewatering devices, settling basins, and auxiliary equipment such as conveyors, hoppers, and weighing devices. The plant has a designed capacity of 70 tph but has a wide operating range of 20-85 tph. The hydrocyclone circuit will operate at 3-7 tph and will supply feedstock for the advanced fine coal beneficiation circuit which operates in the range of 100-300 lb/hour.

While much of the funding for this research facility has come from the State of Iowa, it is available for long term government research should it be needed.

PRESENT STATUS

A. Washability Studies

Washability studies are performed on samples selected from the mine seam before coal from that seam is processed through the preparation plant. The standard procedure developed by the Bureau of Mines is used whereby a repre-

sentative sample is collected and a series of float-sink tests is conducted in liquids that vary in specific gravity from 1.30 to 1.60. The procedure has been modified so that the individual samples are small enough that the particles of different density are not prevented from floating or sinking due to interference. The amount of coal that floats or sinks at the different specific gravities is used to produce a separation curve. Each float and sink portion is then analyzed for sulfur and ash. These data are used to predict what processes the coal can best be cleaned.

Washability studies on coals processed through the Ames facility show a wide variation in coal characteristics. For this reason coal preparation equipment is flexible enough to permit adjustments to be made easily when different coals are processed through the same beneficiation plant.

B. Physical Cleaning

Run of mine coal brought to the coal preparation plant is fed through a crusher and screened, and the coarse ($1\frac{1}{2}$ " x $3/8$ ") material is run through the heavy media plant. Medium sized ($3/8$ " x 48m) particles are cleaned physically on a concentration table. The mechanical beneficiation processes remove up to 90% of the pyritic sulfur as well as much of the other mineral matter.

C. Fine Coal Circuitry and Advanced Chemical Desulfurization

A fine coal circuit for beneficiating material sized 48m x 0 is being installed in the preparation plant. The circuit includes hydrocyclones, preconditioning equipment, froth flotation cells, oil agglomeration, pelletizer, disc filters, and a water clarifier. Plans call for the fine coal circuit to be on-line by February 1978.

The chemical beneficiation loop of the fine coal circuit was the result of extensive laboratory testing and evaluation procedures aimed at desulfurizing pulverized high sulfur coal. As a result of this testing, selective oil agglomeration, froth flotation, and a chemical extraction process were identified as the most promising methods for further development. It was further shown that the cleaning efficiency of both the selective oil agglomeration and froth flotation processes can be greatly improved by application of a newly developed chemical pretreatment step. The chemical extraction step is being tested on the laboratory scale and will be integrated into the circuit at a later date.

D. Coal Microstructure and Petrography

Microstructure data collected on raw coals should contribute substantially to the understanding of the nature of coal as it relates to various methods of beneficiation, desulfurization, and other processing. The coal microstructure is determined and characterized by combined scanning electron

microscope-energy dispersive analysis techniques. Microstructural information on a micrometer size scale is correlated with microchemical information developed by energy dispersive X-ray microanalysis to establish the presence and distribution of pyrite, the primary sulfur bearing phase in coal. Complementary analytical techniques such as scanning transmission electron microscopy, X-ray diffraction and reflectance and petrographic microscope studies are also employed. Studies of this kind evaluate particle size, shape, and distribution factors which bear on decisions on methods of removing additional sulfide particles.

E. Analytical and Structural Characterization

Proximate and ultimate analyses on raw and beneficiated coals are performed as described by ASTM and the U. S. Bureau of Mines. Other analyses have been performed on coal samples at the request of the groups studying the mechanical and physical beneficiation of coal. These include analyses for sodium, potassium, chloride, and fluoride as well as most metallic and metalloid elements which are found in coal at the trace level. Several analytical techniques, including spectrophotometry, X-ray fluorescence, atomic absorption, and instrumental neutron activation analysis are used. In addition, a new technique, inductively coupled plasma-atomic emission spectroscopy which has been developed at the Ames Laboratory, will be applied to the multi-element coal analysis.

In addition to the analytical characterization of raw and beneficiated coals, techniques are being perfected for determining structural characteristics. One program uses pulsed nuclear magnetic resonance for structural determinations, while another program addresses itself to identifying and quantifying organosulfur components in coal and coal derived fuels.

Sulfur determinations are performed in several ways. Supplementing the ASTM procedure is a determination of total sulfur by on-line irradiation with thermal neutrons followed by counting of the prompt gamma rays. This technique has proven to be rapid and highly accurate. Another technique in development uses X-ray diffraction to determine pyritic sulfur only.

FUTURE PLANS

Future plans call for increased activity in coal beneficiation at the Ames Laboratory, particularly in the development of new technologies and in the evaluation of new processes. It is anticipated that major areas of interest will include equipment and process development, fine coal beneficiation by HGMS, wet and dry separation techniques, and chemical desulfurization. A primary goal will be to prepare coal for its best use, whether that be direct utilization or a conversion process.

Much of the effort of the fossil energy program at the Ames Laboratory will

be centered on coal beneficiation in which the Laboratory expects to assume a major role. It is expected that the direction of future growth in this area will be toward advanced technological processes which are greatly needed to insure that coal may be burned in an economically and environmentally sound manner.

LEAD MISSION IN COAL BENEFICIATION

The Ames Laboratory, Iowa State University, is a multi-purpose laboratory under contract with the Department of Energy. In 1974 the Laboratory became involved, through the Energy and Mineral Resources Research Institute (EMRRI), with the Iowa Coal Project, a state-funded project initiated to develop the coal resource bank in the State of Iowa. Several projects were undertaken by the Laboratory, among them the design and fabrication of a coal beneficiation plant which went on-line in the spring of 1976. Since the completion of Phase I the preparation plant has cleaned approximately 50,000 tons of coal mined in Iowa and in other locations. The plant operates at a maximum capacity of 85 tph and has a rail siding, a crusher, heavy media separation equipment, concentration tables, and circuitry for moving the coal through the plant. A fine coal circuit which includes hydrocyclones, thickeners, chemical pretreatment, froth flotation, oil agglomeration, and pelletizing equipment is on hand and scheduled to be installed this winter.

The Ames Laboratory presently has DOE funding for fine coal beneficiation, advanced chemical desulfurization research, and a materials program related to coal conversion problems as well as funds for program development. The beneficiation facility has been made available by the state for long term federal research. Additional funds have been received as pass-through money for development of on-stream analyzing equipment for the clean and hot dirty gas streams at the Morgantown Energy Research Center. Other problems which are presently being addressed by Ames Laboratory scientists and engineers which relate to coal beneficiation include on-stream analyzers for total and pyritic sulfur and coal characterization, both analytical and structural. Work on several other related problems will be initiated as funding becomes available.

The Ames Laboratory has many special facilities including machine and sheet metal shops, electronic instrumentation services, and excellent analytical instrumentation. The Laboratory has unique capabilities in the design and fabrication of equipment and instrumentation. Much of the equipment in the coal preparation plant was designed and built within the Ames Laboratory. The Laboratory is also responsible for bringing to the scientific community such diverse instrumentation as the inductively coupled plasma-atomic emission spectrometer and the triple axis neutron diffractometer presently in use at the National Bureau of Standards. The Laboratory is widely recognized for developing and operating demonstration processes for producing pure uranium, thorium, yttrium, and calcium metals and for separating rare earths by ion exchange.

In essence, then, the Ames Laboratory is basically a research institution with special facilities and capabilities for solving problems. Personnel within the Laboratory represent a diversity of scientific and engineering disciplines and have, through the Laboratory's history, demonstrated a willingness to bring those diverse backgrounds together in order to focus on problems of concern. We feel that the Laboratory is presently in a unique position to serve in a lead laboratory role in coal beneficiation. We have an operating facility which can be put to use immediately and affords sufficient flexibility to serve the immediate and future needs of the DOE. The role we envision would include serving as the DOE test and research facility until other test facilities can be brought on-line. Should other test facilities be built elsewhere we would then plan to relinquish some of the testing operation aspects, if recommended by DOE, and retain the research aspect of the lead mission. We consider our long term commitment in coal beneficiation to be in the area of research into new and advanced technologies which seem to be sorely needed if we as a nation are to utilize our coal resources in an economically and environmentally sound manner. We feel that we are in an excellent position to contribute to the design and evaluation of new systems and desire to cooperate with other institutions who share similar goals.

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EMRRI/AMES LABORATORY COAL PREPARATION RESEARCH PLANT
WITH SUPPORT FACILITIES AT IOWA STATE UNIVERSITY, AMES, IOWA

I. Existing Facilities

Installed Costs

A. Coal Plant Processing Equipment

Heavy Media Separator
Concentration Tables
Separating Screen
Dewatering Screen
Sieve Bend
Conveying Equipment
Settling Basin with Flocculation Equipment
Crusher
Hopper and Vibratory Feeder
Electronic Weigh Scale
Process Control Systems

Total Equipment \$775 K

Washability and Sample Preparation Laboratory \$15 K

Building and Grounds with Complete Utilities
Support \$280 K

Total Existing Coal Processing Plant \$1,070 K

B. Existing Support Facilities

Rail Siding Serving ISU Power Plant and Is
Available to Serve Coal Preparation Facility

One Half Mile Trackage
Car Unloading Equipment with Hoppers
Crusher
Conveying Equipment

Approximate Total Value (\$750 K)

Major Equipment Holdings of the Ames Laboratory
for Elemental and Structural Analysis

Inductively Coupled Plasma-Atomic Emis-
sion Spectroscopy
Atomic Absorption
Emission Spectroscopy (conventional)
Microprobe--Energy and Wavelength Disper-
sive with X-ray Readout Channels
X-ray Excited Optical Luminescence
Vidicon Direct Reading Spectrometer
Laser Induced Optical Luminescence
Inductively Coupled Plasma--Mass Spectro-
meter System (in development)

B. Existing Support Facilities (continued)

Plasma Asher
 Flame Emissions
 ESCA (Electron Spectroscopy for Chemical Analysis)
 SIMS (Secondary Ion Mass Spectrometer)
 Auger System
 Energy Dispersive Vacuum X-ray Fluorescence
 Scanning Electron Microscope with Energy Dispersive X-ray Readout
 Conventional Mass Spectrometers
 Spark Source Mass Spectrometer
 GC Mass Spectrometer
 Laser Mass Spectrometer
 Neutral Particle Mass Spectrometer
 Infrared Spectrometers (Range 30-5000 Wave Numbers)
 X-ray Diffractometers (computer controlled)
 Neutron Diffractometers (2 and 3 axis, computer controlled)
 Picosecond Spectroscopy
 Tunable Dye Lasers
 Laser Induced Atomic Fluorescence
 Laser Raman Spectroscopy
 Low-Energy Electron Diffraction
 UV, Visible Spectrometers
¹H NMR (Nuclear Magnetic Resonance)
¹³C NMR
 Fourier Transform NMR
 Gas Chromatography Units
 Magnetic Susceptibility Unit
 Electron Microscopes
 Neutron Activation Analysis Equipment
 Including Several Multichannel Analyzers and Solid State Detectors
 Petrographic Laboratory

The considerable facilities of Iowa State University, including Fourier Transform IR, electrochemistry equipment, and other major items, are also available.

Approximate Total Cost	<u>\$2,500 K</u>
Total Existing Support Facilities (not including railroad)	<u>\$2,500 K</u>
Total Existing Coal Plant with Support Facilities	\$3,570 K

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II. Advanced Fine Coal Processing Equipment Under Construction

A. State of Iowa Funded

Hydrocyclones
Dewatering Cyclones
Disc Filters (coal and refuse)
Advanced Design Thickener
Pumps and Process Control Systems

Total System Cost

\$300 K

B. Federally Funded

Coal Slurry Preconditioning Circuit
Mixing Tanks with Heat/Cool Capacity
Ball Mill
Cyclone Classifiers
Chemical Injection
Froth Flotation Equipment
Oil Agglomeration Circuit
Pelletizer
Pumps and Process Control Systems

Total System Cost

\$175 K

Total Equipment Under Construction

\$475 K

Grand Total: Existing Coal Processing Plant with Support Facilities and Equipment Under Construction

\$4,045 K

III. Circuits to be Added at Coal Preparation Plant

- A. Heavy Media Cyclones in Parallel with Concentration Tables
- B. Advanced Fine Coal Beneficiation Equipment
- C. High Gradient Magnetic Separation Magnet
- D. Chemical Desulfurization Circuit

IV. Additional Facts

A. Processing Rates

Full-Scale Coal Preparation Research Plant - 20 to 85 TPH
State-Funded Fine Coal Hydrocyclone Circuit - 3 to 7 TPH
Federally-Funded Advanced Fine Coal Circuitry - 100 to 300 Lbs/Hr

B. Coals Processed to Date Through Plant

Seven 1000-ton Samples of Various Iowa Coals
Approximately 40,000 Tons of Coal from ISU Demonstration Mine #1

C. Emphasis of Research

Full-scale coal beneficiation-sulfur reduction using known processing techniques on high sulfur coals.

Advanced fine coal beneficiation-sulfur reduction using advanced mechanical, chemical, and magnetic separation techniques.

D. Plant Availability

Plant is available and suitable for full-scale washability evaluations on any U. S. coals.

Plant is available and suitable for installation of any new processing or control equipment technology being developed at Ames Laboratory or elsewhere.

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EMRRI/AMES LABORATORY
FOSSIL ENERGY PROGRAM
COAL BENEFICIATION DIVISION
FUNDED PERSONNEL

Name	Title and Project Job Description	Background
Ray W. Fisher	Program Director	Chemist/Mechanical Engineer
Richard A. Grieve	Head, Coal Preparation Engineering	Mechanical/Industrial Engineer
David H. Birlingmair	Design Engineer	Mechanical Engineer
Thomas D. Wheelock	Principal Scientist (Chemical Desulfurization)	Chemical Engineer
Richard Markuszewski	Post Doctoral Associate (Chemical Desulfurization)	Chemist (Analytical)
Dalsoo Shin	Post Doctoral Associate (Chemical Desulfurization)	Chemical Engineer
Thomas G. Squires	Post Doctoral Associate (Structural Characterization)	Chemist (Organic)
Margaret A. Wechter	Post Doctoral Associate (Nuclear Instrumentation)	Chemist (Analytical)
Tom E. Scott	Principal Scientist (Alloy Evaluation Project)	Metallurgist
Charles V. Owen	Assistant Scientist (Alloy Evaluation Project)	Metallurgist
Michael M. Crow	Program Development Coordinator	Political Science/ Environmental Science
James B. Gulliford	Ecologist	Ecologist
James P. Reilly	Assistant Scientist (Physical Coal Cleaning)	Mining/Geodetic Engineer
Robert Z. Bachman	Associate Chemist (Analytical Characterization)	Chemist (Analytical)
Edward L. DeKalb	Associate Chemist (Analytical Characterization)	Chemist (Analytical)
Richard N. Kniseley	Senior Chemist (Analytical Instrumentation)	Chemist (Analytical)
Shen-Ann Shei	Post Doctoral Associate (Alloy Evaluation)	Metallurgist

Name	Title and Project Job Description	Background
Stephen R. Beck	Assistant Chemical Engineer (Chemical Desulfurization)	Chemical Engineer
William J. Haas	Associate Physicist (Analytical Characterization)	Physicist
Raymond T. Greer	Professor of Materials Engineer- ing (Coal Microstructure)	Engineering Science

The personnel roster above reflects those individuals receiving at least 25% of their funding from Fossil Energy. An addition to these individuals we have 17 professionals working on related projects but not receiving Fossil Energy Program funds. These individuals are:

Lawrence E. Burkhardt	Senior Engineer (Process Control)	Chemical Engineer
George Burnet	Professor (Fly Ash Utilization)	Chemical Engineer
Donald L. Biggs	Petrographer	Geologist
Velmer A. Fassel	Analytical Program Director and Ames Laboratory Deputy	Chemist (Analytical)
Robert S. Hansen	Ames Laboratory Director	Chemist (Physical)
Tommy J. Barton	Associate Professor of Chemistry (Structural Characterization)	Chemist (Organic)
James E. Benson	Associate Chemist (Development of Pyrite Monitor)	Chemist (Physical)
Bernard C. Gerstein	Professor of Chemistry (NMR Structural Studies)	Chemist (Physical)
Russell L. Simpson	Assistant Engineer (Work on HGMS)	Electrical Engineer
Delwyn D. Bluhm	Head, Plant Engineering (Work on HGMS)	Electrical Engineer
Mark E. Grootveld	Assistant Engineer Design Engineer	Mechanical Engineer
Ruth B. Wildman	Assistant Dean (Environmental Assessment)	Plant Pathologist