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A PROGRAM OF BASIC RESEARCH ON THE
UTILIZATION OF COAL-WATER MIXTURE FUELS:

QUARTERLY REPORT
FOR THE PERIOD ENDING
SEPTEMBER 30, 1981

to the

U.S. Department of Energy
Pittsburgh Energy Technology Center
Pittsburgh, PA 15213

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1. INTRODUCTION

This is the first quarterly report of Grant No. DE-FG22-81PC40285 from the U.S. Department of Energy, Pittsburgh Energy Technology Center (DOE/PETC) entitled, "A Program of Basic Research on the Utilization of Coal-Water Mixture Fuels." The objective of this research involves improving basic understanding of two areas of special importance to the successful use of coal-water slurries: mixture stability and optimization. This two-year research program was initiated September 8, 1981. The present report covers progress for the calendar quarter ending September 30, 1981, hence, reviews the start-up activities for the first three weeks of the project.

Section 2 of this report reviews activities in the study of mixture stability. Section 3 discusses studies on the atomization of coal-water slurries. Sections 2 and 3 each are organized according to specific tasks listed in the proposal Statement of Work. For each task, research accomplishments and plans for the next report period are discussed. Finally, Section 4 of the report covers other activities related to information exchange and utilization of project results.

2. STABILITY STUDIES

2.1 Scope of Tasks for the Report Period

The overall objective of this work is to improve basic understanding of coal-water slurry stability. Although the grant did not begin until September 8, 1981, during the previous three months we had made some preliminary measurements on Pittsburgh Seam #8 coal samples in order to clarify the requirements to be met in our first task of ordering equipment, choosing materials and defining the experimental conditions. This task, planned for the first month, is almost completed. Some equipment has been ordered, and decisions are being made on all other equipment. We have chosen Pittsburgh Seam #8 as our baseline coal and agreed to defer selection of other coals until we learn which properties are the chief determiners of slurry stability. The conditions for all initial measurements have been specified.

The second task, also scheduled to begin in the first month, is the measurement of properties of the powdered coal as received. Preliminary measurements on this material have included surface area, particle size distribution, wettability and density. In addition, the third task, measurement of coal-water slurry properties, has also been started with measurements of suspendability, viscosity, particle size distribution, density in water, and pH, although this work was not scheduled to begin before the second month.

2.2 Task 1: Materials Selection and Conditions

2.2.1 Research Accomplishments

Sampling and sieving equipment have been ordered. A demonstration and trial period for the Micromeritics Electrophoretic Mass Transport Analyzer has been arranged so that we can evaluate its usability for high slurry loadings.

The application of available viscometers to concentrated slurries has shown us that the existing viscometers in our laboratory are usable only for loadings below about 40% by weight. Therefore we have been

conferring with other investigators who have worked with coal-oil or coal-water slurries, and with instrument suppliers, in order to make the best decision for the purchase of a suitable rheometer.

At a meeting at CMU with the atomization group and Jim Ekmann of PETC, at which Dr. Ekmann gave us information on the availability of various coals, we concluded that we would all use Pittsburgh Seam #8 coal as our baseline, and that probably three more coals would be chosen from the group of seventeen coals selected for the Advanced Research and Technology Development Program of DOE. However, the choice of the other coals will be deferred until we can make a more informed decision based upon the properties of Pittsburgh Seam #8 which prove to have the most import for slurry stability. For example, grindability, particle shape, surface chemistry and/or impurities may be key factors.

For the first six months we will be studying suspensions of coal in distilled water. Most measurements will be made at 25°C, but we plan to observe the effects of storage of slurries at 60 to 70°C and 5°C on suspendability, and to discover whether room temperature properties have changed subsequent to such storage. We also plan to examine the temperature dependence of viscosity. The measurements of powder and suspension properties will be performed on the coal powder as received and on various sieved fractions, down to less than 5 µm. This should allow us to test some possible explanations of stability requirements. The recent publication of the Alfred University patent #4,282,006, for example, suggests that particle size distribution and surface charge are important in determining stability. We will supply characterization data as well as sieved fractions to the atomization group to facilitate correlation of our joint findings.

2.2.2 Plans for Next Report Period

Task 1 is nearing completion, except for the deferred selection of

future coals. We expect to order the remaining equipment within the next month.

2.3 Task 2: Coal Sample Properties

2.3.1 Research Accomplishments

During the past summer we obtained from PETC two small samples of Pittsburgh Seam #8 coal which appear to differ somewhat.

(a) Surface Area: A BET surface area measurement with the Quantasorb gave a surface area of approximately $1.6 \text{ m}^2/\text{gm}$.

(b) Particle Size Distribution: Portions of both samples have been suspended in 0.15 molar NaCl, and particle size distributions, on a volume basis, have been measured with a Coulter Counter. The results are listed in Table 1.

Table 1. Size Distribution of Powdered Coal, on a Volume Basis

	<u>Coal Sample #1</u>	<u>Coal Sample #2</u>
Maximum particle size	~ 140 μm	~ 140 μm
Cumulative % less than 75 μm	85%	90-95%
Median size, d_v	40 μm	27 μm
δ	2.3	2.1

We have measured particle size distributions on very dilute suspensions of Coal Sample #2 in water using an Andreasen Pipette, thus obtaining the Stokes diameter. Although initial results with this method are erratic we expect to refine our technique. It appears, nevertheless, that the Stokes diameter distribution is close to the volume distribution obtained with the Coulter Counter, as would be expected if particles are roughly spherical.

(c) Density: We have measured density in water, using a pycnometer, with results given in Figure 2.1. The extrapolated value of 1.3 gm/cc is in the range of densities reported for bituminous coal. This infinite dilution value will later be checked with helium density measurements upon the powder.

(d) Wettability: We have made crude measurements of the wettability of a layer of coal powder immobilized upon paraffin, and find that the coal powder is less wettable than the wax.

2.3.2 Plans for Next Report Period

The preliminary experiments carried out to date have made clear that the methods we have chosen for measuring properties of coal powder as received are applicable. In the next quarter we expect first to concentrate on checking the reliability of our sampling methods, and to get sizable sieved fractions of Pittsburgh Seam #8 coal. Then we will measure properties of the coal as received, and of sieved fractions. We will observe the shape of particles using either a Bausch and Lomb Comparator or a Quantamet Comparator. We expect to complete Task 2 in the next quarter, on schedule, for our baseline coal, Pittsburgh Seam #8. When other coals are chosen, the time and effort required for measurement of powder properties will be less.

2.4 Task 3: Preliminary Measurements on Coal in Water

2.4.1 Research Accomplishments

(a) Suspendability: Samples of 1 to 60% by weight of coal in water were mixed and transferred to graduated cylinders and left to settle undisturbed. There is a definite change in behavior at about 20% by weight. Below that loading, there is gradual clearing of the supernatant as first large and then small particles settle out. Above that loading, all the particles settle as a mass, leaving the supernatant liquid clear. The settled layer

Figure 2.1 Density of Pittsburgh Seam #8 coal in water at room temperature.

○ Sample #1

□ Sample #2

in all cases appears to contain about the same proportion of water to coal, corresponding roughly to simple cubic packing of the particles. If these settled suspensions are then vibrated, considerably more compaction occurs. Results are shown in Figure 2.2. For undisturbed samples, most of the settling occurs in the first two hours, and is complete within 24 hours.

The method of mixing slurries affects the total amount of powder which can be incorporated. With a shaker, the highest loading obtained was 60% by weight, while with an impeller stirrer, a loading of over 65% by weight was obtained.

(b) Particle size distributions: Small samples withdrawn from freshly suspended coal samples at various loadings were used for particle size distribution determinations in the Coulter Counter. The particle size distributions did not appear to change substantially for different loadings except that in the more dilute suspensions rapid settling caused large particles to be underrepresented in the samples taken for the Coulter Counter. The distributions also resembled those found by adding powder directly to the Coulter Counter sample beaker as listed in Table 2.1. Coulter Counter particle size distributions on supernatant liquid at half-hour and 24 hours of settling indicated that it is possible to measure sedimentation rates with the Coulter Counter.

(c) Viscosity: A Stormer Viscometer was used for measurements upon slurries of up to 40% by weight of coal. Above this loading results were not reproducible. An attempt to measure viscosity of a 58% by weight coal slurry in the Sieglaff-McKelvey Capillary Rheometer was unsuccessful owing to extreme dilatancy of the sample.

(d) Density: The density in water of several slurries was measured. For low loadings, coal powder was placed in the pycnometer and water added. For high loadings, the settled layer of a suspension was loaded into the pycnometer and the weight of coal added was determined by evaporating off

Figure 2.2 Settled volumes of Pittsburgh Seam #8 coal at 25°C
(1) $\circ$ Samples mixed by shaking and left undisturbed for 24 hrs.
(2) $\bullet$ Samples from (1), subjected to subsequent vibration.
(3) $\square$ Sample mixed by stirring and left undisturbed for 24 hrs.

the water at room temperature. There appears to be a gradual increase in density with loading, and then a constant density at higher loadings, with the break occurring in the same region as the change in settling properties noted above.

(e) pH: The pH of the supernatant solution after 24 hours of settling was measured for several slurries with loadings of 20 to 30%, and was found to be 3.5. When a 40% by weight suspension was made up in NaOH to give a final pH of 10, a change in the settling behavior was noted. Instead of the mass settling with a clear supernatant solution seen previously for higher loadings, the gradual settling characteristic of loadings of less than 20% in water was seen. The final volume of the settled layer was the same as for the pure water case, but the rate of settling was slower.

2.4.2 Plans for Next Report Period

Task 3 is well under way. Microelectrophoresis experiments will be started, and measurements of adsorbed contaminants will be considered in the next quarter.

3. ATOMIZATION STUDIES

3.1 Scope of Tasks for the Report Period

On September 8, 1981, when the proposal "A Program of Basic Research on the Utilization of Coal-Water Mixture Fuels" was granted, Task 1 of the sub-program "Atomization Studies of Coal-Water Mixtures" was initiated. In this task the design and construction of the atomization test facility is proposed and the first step to identify design parameters have been taken. A literature review was started and summaries of a few select articles are included in Section 3.3.

3.2 Task 1: Design and Construction of Experimental Set-up

3.2.1 Research Accomplishments

The principal investigator employed a Ph.D. student (Ms. Jayashree Padmanaban) to work full-time on the project. An undergraduate work-study student was hired for drafting and minor design work.

The design and construction of the atomizer-test chamber was started. Design parameters such as operating pressure, slurry, mass flow rate, viscosity and other material properties of the slurry have been identified for dimensioning the pump and piping system. The first detailed chamber design was discussed and revised in order that certain details might be reworked. Information regarding instrumentation for the slurry loop has been collected. Discussions with the workshop foreman have been conducted in order that the location for set-up might be identified, and that water, power and pressurized air supply at this location could be examined. The experimental site was approved, and material for the frame of the facility was ordered.

3.2.2 Plans for Next Report Period

The employment of a second Ph.D. student is projected for the end of the next report period (December, 1981).

During the next report period the design phase of the test facility will be completed, and most of the construction will be accomplished. The design and realization of the slurry-loop control system will be initiated after decisions have been made regarding which instrumentation is necessary to characterize the loop operation. The control-strategy will be discussed, and, depending upon the requirements, micro-processor systems will be selected for their feasibility.

3.3 Task 2: Development and Testing of Basic Atomizer Types

3.3.1 Research Accomplishments

This task was initiated by collecting information about industrial atomizers.

3.3.2 Plans for Next Report Period

Designs of industrial atomizers will be reviewed, and potential design tests will be identified. Design of atomizers stressing certain physical effects will be initiated.

3.4 References and Summaries of Selected Papers

1. Reitz, R.D., Bracco, F.V., "Ultra-High-Speed Filming of Atomizing Jets," Physics of Fluids 22 (6), June 1979.
2. Rizkalla, A.A., Lefebvre, A.H., "The Influence of Air and Liquid Properties on Air Blast Atomization," Fluid Mechanics of Combustion, ASME, Montreal, Quebec, Canada (1974).

3. Petela, R., and Zajdel, A., "Atomization of Coal-Liquid Mixtures," Fuel, Vol. 59, July 1980.
4. Carmi, S., and Ghassemzadeh, M.R., "Atomization of Coal-Oil Mixtures," presented at Second International Symposium on Coal-Oil Mixture Combustion, Danvers, Massachusetts, November 27-29, 1979.

Summaries of Selected Papers

(1) Ultra High Speed Filming of Atomizing Jets

R.D. Reitz and F.V. Bracco, Department of Mechanical Engineering,
Princeton University, Princeton, New Jersey

AIM: To study the forces controlling the jet break up process of a liquid jet.

EXPERIMENT: A rectangular spray chamber (25x45 cm) was used with nozzle interchangeably mounted on 2 holders. Three 10 cm diameter quartz windows allowed to view the spray in the chamber. Framing cameras with high-framing rates were used for the resolution of break-up transients due to high injection velocity and small diameter. Nine different nozzles having fixed exit-diameter ~0.34 mm were tested. Nozzle designs included nozzles with length to diameter ratio's ranging from 1/2 to 50, and allowed the effect of changes in nozzle inlet geometry and material construction to be studied. Four different mixtures of water and glycerol were tested. Injection velocity (high) $\sim 10^4$ cm/sec. Liquid pressure - 33 to 140 atm. Chamber gas pressure (nitrogen) - 1 to 20 atm. Test environment - room temperature.

Two test methods were used: 1. transient, 2. steady state.

Transient method: the break-up details were recorded at the onset of injection, by high speed framing camera. Here, very little change (in the appearance of the jet) from one frame to the next, was observed. Changes become evident if distant frames are compared. The emerging jet rapidly

approaches its steady state configuration.

Steady state method: Details were recorded about one second after the initial emergence of the spray into the chamber. Here the jet cross-section was found to increase in the direction of motion of the jet.

RESULTS: The spray angle σ was found to increase as the chamber gas density and not increased gas pressure. Within the tested range, liquid cavitation and boundary layer velocity profile relaxation phenomena could explain the observed experimental trends adequately.

(2) The Influence of Air and Liquid Properties on Air Blast Atomization

A.A. Rizkalla and A.H. Lefebvre, School of Mechanical Engineering,
Cranfield Institute of Technology, Cranfield, Bedford, England

AIM: To study the effects of air and liquid properties on atomization.

EXPERIMENT: Atomizer used - air blast atomizer. Liquids used - water and kerosene. The atomizer is specially designed - the liquid is first spread into a thin sheet and then exposed on both sides to high velocity air. The high relative velocity is achieved by injecting fuel at low velocity into a high velocity air stream. Variation in air temperature was achieved (to study effect of air density on mean diameter of droplet) using kerosene-fired fire heater located upstream of the test section. Temperature varied between 23 and 151°C. Air mass flow was measured using an orifice plate fitted with pressure tapings. Drop sizes were measured using the light-scattering technique suggested by Dobbins, Crocco and Glassman - based on forward scattering of a parallel beam of monochromatic light passed through a spray. The main advantage is that it does not affect the spray pattern or secondary atomization.

RESULTS: The effect of air temperature, air velocity, flow rate of liquid, and liquid/air ratio on the mean diameter of the droplet and graphs were drawn.

(3) Atomization of Coal-Liquid Mixtures

R. Paleta and A. Zajdel, Gliurce, Poland

AIM: To study the atomization process of a mixture of heavy set and solid combustible particles.

EXPERIMENT: Liquid used - benzoic acid at 453k. Solid used - coal dust.

Type of atomizer used - swirling type with outlet nozzle diameter d_0 of 1.05 mm. Atomized by means of compressed air, pressure of mixture - constant or varied within 0.142 - 0.804 MPa before it entered atomizater. Dust gram fraction, g - 3.25 - 28.7%; dust grain coarseness - 0 - 0.385 mm. A run was complete when the finest particle in the chamber had sedimented. Out of 140 atomizations, only 74 successful runs were considered for analysis.

CONCLUSION: Operating difficulties faced due to nozzle blockage by large grains in the mixture. Maximum grain dust fraction g_m , depends on dust size. Increase in dust size relates to a decrease in the values of grain dust fraction. Larger dust contents, g relates to larger droplet diameter of the mixture, whereas increased pressure of the mixture relates to smaller droplet diameters. Larger dust content in the mixture (at constant pressure and temperature) relate to increased atomizer output and greater outflow velocity.

(4) Atomization of Coal-Mixtures

S. Carmi and M.R. Ghassemzadeh, Department of Mechanical Engineering,
Wayne State University

AIM: To study the atomization characteristics of coal-oil mixtures, mainly concentrating on the viscous effects which resist spray formation, thus leading

to an increase in the mean particle diameter.

EXPERIMENT: Oil used - #6 oil; coal - pulverized 200 mesh coal.

Mixtures - 30 - 50% coal by weight. Atomizer used - air blast atomizer.

Spray channel - 2'x2'x10', made in part by plexiglass (2 feet) and followed by galvanized sheet metal (8 feet). Plexiglass portion for setting camera-flash unit. Storage tank - 55 gallon drum. Flow control unit - Tri-Rotor Model 20D positive displacement pump. Nozzle discharge pressure - 5 psi; air pressure - 40-50 psi; feeding fuel tank - kept at 140°F - 180°F.

Photographic equipment - light scattering methods. Pentax 35mm SLR camera filled with bellows and extension tube resulting in a lens extension of 280 mm focal length and out light source was EG&G model 549 microflash. Flash duration 1 μ sec.

RESULTS: Viscosity has an adverse effect on spray quality. Increase in coal concentration in the mixture will cause an equivalent increase in viscosity, which influences the forces resisting the break up of the fuel jet into small droplets, leading to spray formation.

Increase in viscosity caused increase in the mean drop size, resulting in an adverse effect on spray quality.

4. OTHER RELATED ACTIVITIES

(1) Representatives from Babcock and Wilcox visited CMU on September 8, 1981. They were interested in arranging sub-contract work on the stability of coal/water slurries, in connection with their proposal to DOE to fund work in coal/water slurry technology. It was clear that the work they proposed is already covered under our present DOE/PETC contract. It was agreed that subject to DOE approval a line of communication would be established between CMU and Babcock and Wilcox, if they are successful in their bid for funds.

(2) Professor R. Rowell of the University of Massachusetts delivered a lecture at CMU on Thursday, October 1, 1981 entitled "Stability and Rheology of Coal/Water Slurry Fuels." From this and extended discussions with Professor Rowell we established that no other university groups are working on the fundamental aspects of the stability of coal/water slurries.

(3) Contact was made with Babcock and Wilcox Research Laboratories in Alliance, Ohio, where a groups of researchers are involved in setting up a large-scale test facility for atomization of slurry fuels. It was agreed that a direct information link would be set up between the two projects. In consultation with DOE/PETC, it was announced that exchange of results from this project is not restricted.

(4) Contact was made with the Department of Chemical Engineering at the University of Pittsburgh. Information on instrumentation was gathered from an experimental set-up used for studying, "Hydrodynamics of Coal-Water Slurries in the Presence of Gas."