"MULLED COAL" A BENEFICIATED COAL FORM
FOR USE AS A FUEL OR FUEL INTERMEDIATE

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Technical Progress Report No. 4
For the Period January 1, 1991 through March 31, 1991

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I. EXECUTIVE SUMMARY

Energy International has been awarded a contract by DOE-PETC (DE-AC22-90PC90167) to evaluate a new concept for utilization of the fine coal wet cake produced by many of the physical beneficiation processes now under development.

During the past quarter (January 1, 1991 to March 31, 1991), Energy International has evaluated additional mull formulations with varying reagent additives, mixing times, and particle sizes. The Environmental Review was completed and conceptual designs developed for the Mull Preparation and CWF Conversion Systems. The Phase I Review Meeting was held at PETC in February and agreement was reached for moving into Phase II.

Activities during the next quarter will concentrate on revising the Project Management Plan, restructuring the Project Team to implement the revised plan, Phase II feed coal procurement and design, and assembly of component systems. A patent application will be filed for the mulled coal process.

II. INTRODUCTION

The mulled coal project started with the selection of coal feedstocks on May 15, 1990. This report covers activities from January 1, 1991 through March 31, 1991.

Under the auspices of the Department of Energy and private industry, considerable progress has been made in:

- Preparation of coal-water fuels.
- Combustion of low-ash coal based fuel forms.
- Processes to provide deeply-cleaned coal.

These technology advances are compatible with the national objective to increase the environmentally responsive use of readily available and abundant coal resources, and to reduce oil and gas fuel consumption.

As these technical developments move toward commercial application, the needs for coordinated efforts and integrated requirements have become increasingly apparent. Systems are vitally needed to integrate energy delivery systems from the raw resource through processing to application and end use. Problems have been encountered in the preparation of conventional coal-water fuels that mutually satisfy the requirements for storage stability, handling, preparation, atomization, combustion, and economics. Experience has been slow in evolving generic technologies or products and coal-specific requirements and specifications continue to dominate the development. Thus, prospects for commercialization remain highly specific to the coal, the processor, and the end use.

Developments in advanced beneficiation of coal to meet stringent requirements for low ash and low sulfur can be anticipated to further complicate the problem areas. This is attributable to the beneficiated coal being produced in very fine particles with a high surface area, modified surface characteristics, reduced particle size distribution range, and high inherent moisture. Experience in the storage, handling, and transport of
highly beneficiated coal has been limited. This is understandable, as quantities of such product are only now becoming available in meaningful quantities.

Much of the existing work and experience with advanced coal-based fuels, such as CWF, has been built on the premise that the fuel should and will be capable of being compatible with use in the existing storage, handling, and transport equipment as a "one-to-one" replacement for conventional oil fuels. Expensive additives, additional processing, equipment, and system modifications will be needed to provide effective and versatile fuel delivery systems. This will be even more significant for fine particle highly beneficiated coal fuels.

The conventional approach for formulation of CWF would be to develop a single formulation that meets the requirements of the total fuel system. At best, the resultant material will have specifications that represent a compromise.

Energy International has been awarded a contract by DOE-PETC to evaluate a new concept for utilization of the fine coal wet cake produced by many of the physical beneficiation processes now under development.

Energy International is developing a technology that will create a staged formulation with the first coal form (Mulled Coal) that can be stored, transported, and pumped. Just prior to combustion, the Mulled Coal (MC) would be modified to provide the properties needed for proper atomization. This concept is an alternative to the expensive and energy intensive thermal drying processing of fine coal wet cakes. The material is suitable for both direct feed use in conventional and fluid bed combustors as well as on-site conversion to combustible slurries. By maintaining the coal form relatively close to the feed wet cake, only minor processing with low additive levels and low energy blending is needed at the point of production. Its conversion to slurry or other use-feed form is made near the time of use and thus the requirements for stability, climatic control, storage, transport, and handling are much less severe.

III. PROJECT DESCRIPTION

The U.S. Department of Energy (U.S. DOE) Pittsburgh Energy Technology Center (PETC) and Energy International Corporation have executed a contract to address the storage, transport and handling of beneficiated coals in the form of a modified wet cake ("mulled coal") to yield a coal water fuel having acceptable properties for atomization and combustion on industrial, commercial and/or residential scales.

The Mulled Coal project is divided into a series of tasks designed to produce formulations and system designs suitable to convert fine coal "wet cakes" into a material that can be stored, handled, and transported to a site where it can be utilized as a fuel in existing and developing combustion devices.

The work is divided into two phases.

Phase I has provided the technology base and established the technical feasibility of the concept of stabilizing the fine coal "wet cake"
in a form that can be readily stored and moved to the location where it can either be combusted "as is" or converted into a desired fuel form at the combustion site. The information developed during Phase I provided a basis for design and implementation of a demonstration program (Phase II).

Phase II will demonstrate the ability to utilize the Mulled Coal to improve the ability to store and move fine coal products as stable "wet cakes". Tasks in this Phase will first test components of the various systems required for storage, handling and combustion of the fine coals and subsequently demonstrate operations of an integrated system for storing, handling, and combustion of the highly beneficiated fine coal products.

Phase I - Once the feed coals were selected, basic technical information for formulations of Mulled Coal was developed through bench studies. Some basic assumptions for the process concept were:

1. Chemical formulations will have to consider end use as well as the chemistry involved with the beneficiation processes.

2. If any changes in particle sizes are required for the combustor, they should be made previous to formulation of the Mulled Coal.

3. Conversion from Mulled Coal to the final fuel form should require minimal equipment and relatively moderate conditions such as dilution, addition of small quantities of reagents, mild agitation, little heating. Grinding, high temperatures, corrosive or hazardous materials, etc. should be minimized.

Phase II - Phase II will comprise the design, acquisition, and testing of the system components required to simulate the storage, transportation and combustion of the "mulled coal" formulation(s). The "mulled coal" will be prepared in sufficient quantities to perform full system testing of the preferred formulations.

IV. PROJECT STATUS

1.0 Phase I - Technology Development

1.1 Feedstock Acquisition

No additional coal purchases were made during this quarter.

1.2 Feasibility Study

Task activities included Mulled Coal Formulation Studies and Plan Revisions for the Bench Scale Testing Task.

Mulled Coal Formulation Studies

Several coals other than the Upper Elkhorn #3 bituminous coal were evaluated to see if they could be converted to the granular product. The coals tested were:

1. Pittsburgh #8 coal (bituminous)
2. Illinois #6 coal (bituminous)
3. Powder River Basin coal (subbituminous)
4. Beulah-Zap (lignite)
5. Anthracite coal from eastern Pennsylvania

**Pittsburgh #8 Coal**

Three hundred grams of a Pittsburgh #8 coal (PGH-249-PCO-P) and approximately 300 grams of water were ground in the small ballmill to prepare micronized coal. After grinding, the coal was filtered and dried to give a coal with a mean volume of 13.3 microns. Three hundred grams of the dry coal was placed in the bench scale blender. To this was added 190 grams of water. After thorough blending of the water and coal, 9.0 grams of the mulling agent was added and mixing continued. A good granular mull was prepared.

To determine if a sprayable slurry can be prepared from the Pittsburgh #8 mill, 1% (based on the weight of dry coal) of a non-ionic surfactant was added to the blender and stirred for about 5 minutes. A good low viscosity slurry was formed. The viscosity was 398 cps at 110/sec. and 715 cps at 200/sec. with a power law factor of 1.493. These results are typical of the results obtained with the Upper Elkhorn #3 micronized coal.

**Illinois #6 Coal**

A 50/50 mixture of Illinois #6 coal (11% ash) and water were ground for one and one-half hours in the small ball mill. The mixture was filtered and the filter cake dried overnight at 110°C under a nitrogen purge. The mean volume diameter (Mv) was determined to be 13 microns. Three hundred grams of the dried coal was mixed with 190 grams of water. After thorough mixing of the coal and water, 9.0 grams of the mulling agent was added. After continued mixing a good granular product was obtained.

As before, 1% of a non-ionic surfactant was added to the mull and the mixing continued to determine if a sprayable slurry could be prepared. However, no slurry was formed. An additional 1% of the surfactant was added and still no slurry formed. The granular mull was turned into a lumpy mass. It may be that a different surfactant will be necessary to form a slurry or that the high ash content has a detrimental effect on slurry formation. Attempts to prepare a slurry were discontinued. At a later time other surfactants will be evaluated as slurry agents for the Illinois #6 mull.

**Powder River Basin Subbituminous Coal**

The mulling agent was added to a Powder River Basin coal containing 29% moisture to determine if a mull could be prepared using the inherent moisture in the coal. The agent was simply adsorbed into the coal and no mull was formed. A second experiment was run to determine if the addition of a surfactant directly to the coal could "free" the water and possibly form a slurry. Again no change could be detected. Finally, water was added to the coal and mixed in. Then 3% of the mulling agent was added. This time a mull did form and could be converted to a slurry containing 30% solids. Therefore, mulling of subbituminous coal does not appear to be a worthwhile endeavor.
Beulah-Zap Lignite Coal

Attempts to prepare mulls from a lignite coal gave essentially the same results as with the subbituminous coals. The mulling agent was adsorbed into the coal with no appearance of a granular mull being formed. No further work was carried out.

Anthracite Coal

A sample of minus 20 mesh anthracite coal from eastern Pennsylvania was ground with water for one hour and fifteen minutes in the small ball mill. The product was filtered and dried overnight at 110° C under a nitrogen purge. The product had a mean volume diameter (Mv) of 12.7 microns with 98% having less than 44 microns. Three hundred grams of the dried product was blended with 190 grams of water in the bench scale mixer. After thorough mixing 9 grams of the mulling agent was added. Continued blending did yield a granular appearing product but much different in texture than the mulls prepared from the Upper Elkhorn #3 bituminous coal. Addition of up to 2% non-ionic surfactant did not yield a slurry. As with the Illinois #6 coal a different surfactant might be necessary to prepare a slurry from the anthracite coal.

It appears, therefore, that the mulling technique for handling wet filter cakes will be primarily applicable for bituminous and possibly anthracite coals with little or no application for subbituminous or lignite coals.

1.3 Lab Scale Tests

Task administration activities consisted of subcontract preparation, consultant coordination, and submission of the following reports/deliverables.

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Much of the technical information provided in this quarterly was presented at a Project Review Meeting at PETC on February 14, 1991. At that time, agreement was reached for beginning Phase II activities on the provision that the revised Project Management Plan (PMP) covering the amended Phase II activities would be provided in a timely fashion. Considerable time has been spent on preparation of the revised PMP with submission to PETC anticipated in April.
Mulled Coal Preparation

In an effort to scale-up the preparation of the solid fuel form (granular) of mulled coal, various types of mixing equipment were investigated. It was decided to try to make the mull in a commercial 6 cu. ft. mortar mixer. A mixer was rented from a local equipment rental agency and brought to the laboratory. In the first experiment 25 lbs. of dry coal with a mean volume of 42 microns and an ash content of 1.36% was blended in the mixer with 13.5 lbs. of water to yield a synthetic 35% moisture filter cake. It took about 15 minutes to obtain a uniform mix. After the thorough mixing 340 grams of the mulling agent was added and the mixing continued. The mull began to form slowly and in about 15 minutes was completely transformed to a free flowing, dry appearing, granular product. The advantage of this type of mixer was the wiper blades affixed to the stirring blades which continually wiped the walls and ends of the mixing chamber. In the bench scale blender the container walls were cleaned manually with a rubber scraper.

A second run was made but the mixer was allowed to run an additional five minutes (total of 20 minutes) after the addition of the mulling agent. Again a very good appearing granular mull was produced.

A portion (300 grams) of mulled coal was placed in the bench scale blender. To this was added 1% (based on dry weight of coal) of a non-ionic surfactant. After a few minutes mixing the slurry began to form and after about five minutes the slurry formation was completed. The measured slurry viscosities were 394 centipoise at a shear rate of 100/sec. and 410 centipoise at 200/sec. The Oswald power law factor was 1.350.

A third experiment was carried out using a micronized coal prepared by grinding coal in the ball mill to a mean volume diameter of 13 microns. The ash content of this coal before grinding was 1.9%. Twenty-five pounds of the ground coal was blended with 13.5 pounds of water as before. After addition of the mulling agent, some balling of the material was observed. These balls were screened out. A 300 gram portion of this mull was made into slurry as before and its viscosity was 465 centipoise at 100/sec. and 870 centipoise at 200/sec. The Oswald power law factor was 1.665. Upon breaking up of the balls it was observed that the interior of the ball was wet and the outside coated with agent. This did not occur with the larger (42 Mv) particle size coal. It appears that some sort of inner blades (i.e., intensifier blades) are needed in the equipment to prevent this ball formation with the micronized coal.

A final run was made in the mortar mixer and the preparation videotaped. The coal used was the 42 micron mean volume diameter coal (UE3-244-DCC-H). The ingredients used and their quantities were the same as the first run. The mull was again converted to slurry and the viscosities measured. At 100/sec. shear rate the viscosity was 251 centipoise and at 200/sec. it was 358 centipoise with a power law factor of 1.431.

For coarse coals the mortar mixer seems satisfactory but for the micronized coal it seemed to cause balling of the wet coal. Other equipment will be surveyed and evaluated for scale-up of the mulled coal preparation.
Physical Testing

The angle of repose is the natural angle a solid material will form between the slope of the pile and the horizontal line of the base of the pile when piled on the ground. All solid material will form stable piles but the base will continue to expand without additional solid material being added until an almost flat-like pile is formed. Unfortunately, the literature does not identify such materials. However, the procedure we used was to place approximately 150 cc of the material in a graduated cylinder whose base had been cut off. With the cylinder resting on the bench top it is quickly pulled straight up allowing the material to flow out and form a pile. A Polaroid picture of the profile of the pile is taken. The angle of repose is then measured with a protractor either from the photo itself or from a Xerox copy of the photo.

The initial angle of repose experiments were carried out using untreated Upper Elkhorn #3 coal which was sieved into various particle size fractions. This was done to see if particle size would influence the angle of repose. The results are shown in Table I.

Table I

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<tr>
<th>Material</th>
<th>Angle of Repose</th>
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<tr>
<td>1/4&quot; x 10 mesh coal</td>
<td>20 degrees</td>
</tr>
<tr>
<td>10 x 30 mesh coal</td>
<td>28 degrees</td>
</tr>
<tr>
<td>30 x 100 mesh coal</td>
<td>24 degrees</td>
</tr>
<tr>
<td>100 x 200 mesh coal</td>
<td>23 degrees</td>
</tr>
<tr>
<td>200 x 0 mesh coal</td>
<td>could not measure</td>
</tr>
</tbody>
</table>

It can be seen that, with the exception of the very fine coal, particle size had little or no effect on the angle of repose.

The next experiments were carried out in the same manner using the granular mulls of different particle size distributions. All mulls were prepared from the Upper Elkhorn #3 coal at 30% moisture content and 3% mulling agent. The results of the measurements are given in Table II.
Table II
ANGLE OF REPOSE FOR MULLS PREPARED FROM UPPER ELKHORN #3 COAL OF DIFFERENT PARTICLE SIZE DISTRIBUTIONS

<table>
<thead>
<tr>
<th>Material</th>
<th>Angle of Repose, Degrees</th>
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<tr>
<td>Mv = 88 microns</td>
<td>26</td>
</tr>
<tr>
<td>Mv = 41 microns</td>
<td>25</td>
</tr>
<tr>
<td>Mv = 20 microns</td>
<td>23</td>
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*Mv is the mean volume diameter of the coal used to prepare the granular mulled coal as determined by Microtrac analysis.

The results of these tests show that, as before, particle size has little effect upon the angle of repose and that the granular mulled coal can be stacked like coal.

Environmental Review

The mull coal contract has been established to address the storage, transport, and handling of beneficiated coals in the form of a modified wet cake. The objective is to generate a coal water fuel (CWF) from the modified wet cake having acceptable properties for atomization and combustion on industrial, commercial and/or residential scales. Project activity has resulted in two distinctly different types of mulls; the "gal" and "granular" formulations. The granular mull has been selected as the preferred formulation due to its stability and lower cost additive package.

The "granular" mull version has the consistency of dry sand with a particle size several times larger than the feed coal. The mull formulation utilizes moisture content. The granular mull can also be converted to an atomizable CWF with conventional reagents in a low shear system. The handling characteristics do not appear sensitive to moisture levels, feed particle size or variations in coal processing at the lab scale testing level. Preliminary studies also indicate that the loss of moisture during storage has little effect on the handling characteristics.

The feedstock for a granular mull would be the fine coal wet cake produced by many of the beneficiated processes now under development. Environmental and safety considerations would have to take into account the base coal utilized with the respective additives, their storage and handling as well as the mull formulation storage, handling and transportation.

The typical wet coal filter cake used for the mull formulation has a moisture content of +/- 30%. This moisture content reduces the concern of explosion hazards which would occur when handling a dry, powdered coal. Converting the filter cake into the granular mull product requires an addi-
tive which is a liquid. The additive does not require hazardous warning labels under current OSHA regulations and would require the same handling considerations as a mineral oil.

The handling and transportation of the mull would be similar to the accepted commercial operation for sand or cement. Based on testing to date, the mull formulation is expected to remain stable during the transportation and handling phases precluding any concerns about additive separation. Any spillage of the product would be handled as a non-toxic material.

Bin storage of the mull is anticipated with minimal residence time. Upon preparation of the coal water fuel, a surfactant is added to the mull for water dispersion. This additive is designed to be added immediately upstream of the atomization point. The surfactant would be stored as a at the consumption site. Although not classified as a hazardous waste, caution is advised against overexposure to the product which may cause temporary eye or skin irritation. The surfactant can effectively be handled with rubber gloves and goggles and removed with soap and water cleanup.

Storage procedures should keep the surfactant under its melting point of 125°F for product effectiveness, not environmental or safety reasons. Flash point for the surfactant is greater than 500°F. Additional water is the only other additive anticipated to adjust the final fuel consistency.

Based upon Phase I activities, no environmental or safety concerns have been created with the granular mull formulation. The formulation, transportation, handling and storage of the granular mull is expected to be performed under established procedures for non-hazardous materials.

System Design

The systems should have the following characteristics:

Mull Preparation: A low shear mixer with capability to add liquid reagent.

Handling Systems: Screw type conveyor and/or pneumatic transport capable of handling 0.5-5 tons/minute for filling carriers and transferring to storage units. Smaller scale handlers 10-100 lbs/hour for transfer to the combustion unit.

Transport: Covered containers should be used to exclude fugitive dust and to control blowing designed to handle material such as wet sand, grain, etc. with a definable angle of repose and tendency to form friable masses on freezing.

Storage: Covered bins or silos with heights and widths to be determined. The bottoms should be slanted and fitted with screw feeders to remove the gravity fed product. Requirements for vibration and heating to be determined. Capacity will vary from 100-1000 tons at the plant and distribution centers, to 1-50 tons at the point of usage.
CONCEPTUAL FLOW SCHEME FOR CONVERTING WET FILTER CAKE TO MULLED COAL

Figure 1
The conceptual design for the Mulled Coal preparation, storage, and transportation system are shown in Figure 1.

Coal Water Fuels Preparation Circuit:

The specifications will vary depending on the combustion system. However, general specifications will include:

Particle size: Provided in feed coal
Solids Loading: 65-70% for Industrial Size
60-65% for Residential/Commercial
45-50% for Heat Engines

Viscosity: To be determined but will generally be required to have a power law factor <1.1 with viscosity <1000 cp @ 100 sec⁻¹.

Stability: CWF will be prepared at the point and time of usage with no storage. The system will be purged at shutdown.

A conceptual block flow of the CWF prep circuit is shown in Figure 2. The projected capacity will be 10-100 lbs/hr of mull. Provision for addition of CWF reagents and makeup water will be required as will in-line monitoring of critical CWF properties.

2.0 Phase II - System Demonstration

2.1 Component Development

Three mixing equipment manufacturers have been contacted in regard to making the granular "mulled coal" product and to converting it to a coal water slurry. Micronized feed coal plus the required reagents will be taken to each manufacturer and tested in their laboratory test facility to determine the best mixer configuration and operating parameters needed to make the required products. These tests will be performed during the month of May, and the equipment selection will be made as soon as all the test results have been evaluated.

Arrangements have been made to obtain the services of consulting engineers on transportation and materials handling systems to perform shear testing and flow analyses on our granulated mulled coal samples. They will determine the flowability of several mull compositions and make recommendations for designing storage bins, feed hoppers, and conveying equipment. The preparation of the mulled coal samples will commence as soon as sufficient feed coal is available and a laboratory size mixer has been obtained.

V. PLANNED ACTIVITIES

1. The revised Project Management Plan will be completed and submitted.

2. The project team will be restructure to implement the revised PMP.

3. Equipment requirements will be identified for large scale (6 ton) production of both the solid (granular) and liquid (slurry) fuel phases of "mulled" coal.
4. Feedstock coal will be procured for Phase II.

VI. SUMMARY

Preparation of the solid fuel form (granular) of "mulled" coal was increased from bench scale (+/- 1 lb. coal) to lab scale (+/- 25 lb. coal) operations. The mixing times and feed coal sizes were varied. The solid fuel form was converted to a slurry in each case. For coarse coals the mixing appeared satisfactory but the micronized coal resulted in lumping and balling.

Several coal types were also evaluated. It appears that the mulling technique for handling wet filter cakes will be applicable for bituminous and possibly anthracite coals but with little or no application for subbituminous or lignite coals. Further testing concluded that coal particle size had little effect on the angle of repose and that the solid form (granular) of mulled coal can be stacked like coal.

Preliminary environmental review of the "mulled coal" product indicates that no special handling or packaging precautions are required for safety or health purposes. Temperature considerations will be monitored for certain surfactant effectiveness.

Subcontractors and equipment manufacturers will be selected to conduct further testing to determine the best mixer configuration(s) and operating parameters for preparing both the solid and liquid fuel forms of mulled coal in commercial quantities.
September 11, 1991

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Subject: Contract No. DE-AC22-90PC90167
Quarterly Technical Progress Report

Dear Mr. Mayne:

Enclosed are two Final copies of the Technical Progress Report for the period of January 1, 1991 through March 31, 1991 as required by the subject contract.

Very truly yours,

Burl E. Davis
Project Manager

Enclosures (2)

cc: John R. Columbia (MS 921-165)
END

DATE
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