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

DOE/PC/90167--T4

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PHASE I FEASIBILITY STUDIES

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1.0 EXECUTIVE SUMMARY

Energy International Corporation was awarded a contract by the Department of Energy, Pittsburgh Energy Technology Center (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 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, and other storage, transport, and handling requirements are much less severe.

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 project is divided into two phases:

Phase I will provide the technology base and establish 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 should provide a basis for design and implementation of the 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 a stable "wet cake". Tasks in this phase will first test components of the various systems required for storage, handling and combustion of the fine coals and subsequent demonstration operations of an integrated system for storing, handling, and combustion of the highly beneficiated fine coal products.

The Phase I studies of the Mulled Coal Contract resulted in the production of two distinct types of mulls, the "gel" form and the "granular" form.

The "gel" mull is of the type anticipated in the original proposal, i.e., a highly viscous liquid with consistency ranging from that of bread dough to a heavy oil. Conversion to coal water fuel (CWF) can be achieved by shearing the mull at high shear rates ($>200/\text{sec}$) without using additional chemicals or by high shear mixing with water.

The "granular" form of the mull was discovered when a small amount of a specific additive was added to a wet filter cake resulting in a granular product. The granular mull can be converted to CWF utilizing conventional CWF reagents in a low shear system. This report presents the results of Phase I. Phase II will demonstrate the Mullied Coal technology as it relates to the granular product.

2.0 INTRODUCTION

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

- a. preparation of coal-water fuels
- b. combustion of low-ash coal based fuel forms
- c. processes to provide deeply-cleaned coal

These technology advances are compatible with national objectives 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 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 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 specification that represent a compromise.

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, and other storage, transport, and handling requirements are much less severe.

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 project is divided into two phases:

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

This report summarizes the results of the Phase I feasibility studies.

3.0 COAL SELECTION

Four feedstocks were selected for testing and use in the mulled coal process. Three of the feedstocks selected were based on the Upper Elkhorn #3 coal and were products from different advanced physical fine coal cleaning methods, namely:

1. Microbubble column flotation.
2. Heavy media cyclone cleaning.
3. Oil agglomeration.

The fourth coal was a western subbituminous coal (Spring Creek) processed by heavy media cyclone and hot water drying. Samples of the subbituminous coal were obtained both with and without an acid leach before the hot water drying. A total of five feedstocks were available to the project. A summary of the feedstocks, their ash values and source are shown in Table I.

TABLE I
SELECTED COAL FEEDSTOCKS

<u>Coal</u>	<u>Cleaning Method</u>	<u>% Ash</u>	<u>Source</u>
Upper Elkhorn #3	Heavy Media	1.6	CQ, Inc.
Upper Elkhorn #3	Microbubble Column Flotation	1.2	VPI*
Upper Elkhorn #3	Oil Agglomeration	1.4	Otisca
Spring Creek	Heavy Media/Hot Water Dried	1.6	UNDEERC**
Spring Creek	Heavy Media/Acid Leach/Hot Water Dried	1.2	UNDEERC**

* Virginia Polytechnical Institute

** University of North Dakota Energy and Environmental Research Center

The heavy media cleaned Upper Elkhorn #3 coal from CQ, Inc. ground to a mean volume diameter of 10-12 microns was chosen as the reference coal for the screening and bench-scale studies. Other sizes were used in some expanded studies.

4.0 ADDITIVE SELECTION

4.1 Mull Additives

The process concept was the development of a stabilized wet cake with improved handling characteristics that could be readily converted into a atomizable coal water fuel at the point of use. The approach was to select an additive package from those already used in CWF formulations to produce highly viscous liquid or gel that could then be diluted with water in a high shear mixer to produce a suitable CWF.

As a result of literature searches and discussions with manufacturers, additives for mulled coal preparation were selected and evaluated. The different types of additives included:

Dispersants

Nonionics

Nonyl-polyethylene glycol ethers (Tergitol NP series, Union Carbide Corporation).

Polyethylene glycol ethers of primary and secondary alcohols (Tergitol 15-5-X series, Union Carbide Corporation.)

Hydroxy terminated block copolymers of propylene oxide and ethylene oxide (Pluronic, BASE Corporation).

Anionics

Polynaphthalene sulfonates (Morristown Chemicals Group).

Lignin sulfonates (Lignisols, Reed Lignin, Inc.)

Stabilizers

Xanthan Gum (Pfizer Chemical Corporation).

Quar Gum (Henkel Corporation).

Methyl Cellulose (Dow Chemical Company).

Hydroxypropyl Methyl Cellulose (Dow Chemical Company).

An alternative formulation utilizing a non-polar organic material was also chosen for evaluation. Rather than forming a highly viscous liquid, it was felt that an agglomerating mechanism could be attractive whereby the wet cake could be more readily handled.

4.2 Beaker Scale Experiments

The initial screening process consisted of addition of the reagents at concentrations ranging from 0.5 to as high as 5.0% (based on the weight of dry coal) to filter cakes of known moisture contents in a beaker and hand mixing with a spatula for anywhere from 5 to 10 minutes depending upon the ease of mixing, etc. The resulting "mull" was then tested for flowability by determining how it would flow off the spatula due to gravity alone. It was felt that the mull should exhibit some flowability in order to be pumpable or moveable. The results of such screening are given in Table II.

5.0 PREPARATION OF EXPERIMENTAL MULLS

5.1 Preparation of Standard Wet Filter Cake

The moisture content of the filter has a profound effect upon the rheology of the resulting mull. The "natural" water content of the filter cake in turn is controlled by the particle size and particle size distribution of the coal in the filter cake. This relationship is shown in Figure 1, which shows the relationship of the coal's mean volume diameter (Mv), as determined by Micro-Trac analysis, to the "natural" water content of the filter cake.

Therefore, to reduce the variability of the filter cake moisture content it was decided to attempt to prepare a standard filter cake by grinding a 50% coal-water mixture in the ball mill for a predetermined length of time and then filtering with vacuum filtration. After the majority of the water was removed the air was pulled through the filter cake for approximately five minutes and the filter cake was then removed and the water

Table II

Beaker Scale Screening Results of Selected Additives
For Preparation of Milled Coal

Sample I.D.	Filter Cake		Additive	%	Comments
	% Moisture	Mv, microns*			
40-1-T	50	8.5	Tergitol NP-10	1.0	filter cake turned to flowable slurry
40-1-G	50	8.5	Xanthan gum	1.0**	good lower viscosity mull produced
40-1-O	50	8.5	NPA-1	5.0	turned cake into free flowing powder
20-1-T	48	11.6	Tergitol NP-10	1.0	slightly lower viscosity than 40-1-T
	48	11.6	Tergitol NP-10	2.0	still too viscous
	53	11.6	Tergitol NP-10	2.0	seems like optimum viscosity, low solids
20-1-G	48	11.6	Xanthan gum	1.0	too viscous for use
	53	11.6	Xanthan gum	1.0	improved viscosity, more pourable mull
20-1-O	48	11.6	NPA-1	5.0	gives free flowing powder upon mixing
10-1-T	42	18.2	Tergitol NP-10	1.0	more viscous than 20-1-T
	47	18.2	Tergitol NP-10	1.0	soft mull will not flow from spatula
	52	18.2	Tergitol NP-10	1.0	less viscous, will drip from spatula
	52	18.2	Tergitol NP-10	2.0	no apparent change
	57	18.2	Tergitol NP-10	2.0	becomes foamy, appears less viscous
10-2-T	47	18.2	Tergitol NP-10	1.0	same foamy appearance as 10-1-T
	52	18.2	Tergitol NP-10	1.0	appears less viscous but still foamy
10-1-G	42	18.2	Xanthan gum	1.0**	very stiff paste
	47	18.2	Xanthan gum	1.0**	very good consistency, low solids
10-1-O	42	18.2	NPA-1	5.0	gives free flowing powder upon mixing

The following formulations were mixed using a mortar and pestle

n-40-1-T	50	8.5	Tergitol NP-10	1.0	produced flowable mull, good consistency
n-40-1-T-GS	50	8.5	Tergitol NP-10 Guartec SJM	1.0 0.15	less viscous than with Tergitol alone
n-40-1-O	50	8.5	NPA-1	5.0	form. free flowing agglomerates
n-10-1-T	42	18.2	Tergitol NP-10	1.0	formed very stiff paste
n-10-1-T+7%	49	18.2			flowable mull after 7% water addition
n-10-1-N	42	18.2	Napthalene Sulfonate	1.2	a flowable mull was produced
n-10-1-N-G	42	18.2	Napthalene Sulfonate	1.2	Turned into a more viscous mull with addition of gum
			Xanthan Gum	1.2**	

* mean volume diameter

** percent of a 15% active ingredient solution

content determined. This method produced the filter cakes used for the initial screening of some of the reagents shown in Table II. Even though this method produced a more uniform wet filter cake, there was still too much variability of moisture content. Two to three percent difference in moisture content often resulted in large changes in rheological properties (flowability, etc.).

It was then decided to take the dry ultraclean coal and add water to obtain the desired moisture content. The coal was wet ground in the ball mill to the desired particle size and then filtered and air dried to the equilibrium moisture content (usually 2%). This product was then used to prepare "synthetic" wet filter cakes by addition of a known amount of water for the reagent screening. This method was used to prepare wet filter cakes for bench-scale testing.

5.2 Bench-Scale Experiments

Most reagents were screened using a 30% moisture "standard" filter cake. The Micro-Trac analysis showing the particle size distribution of a typical filter cake used for the reagent screening is given in Figure 2. The preparation of the filter cakes and the blending of the reagents was accomplished using an ordinary kitchen blender (Kitchen Aid) with a doughmixer blade. The feasibility of the mull coal concept can be demonstrated with the results shown in Tables II and III. Continued refinement of the formulations should give the desired stable mull. However, the results of the addition of the non-polar additive (NPA) to the wet cake to produce a flowable granular product was unexpected and opened up a whole new dimension to the problem of stabilization for the purpose of storage, transportation, and utilization of wet filter cakes. Therefore, studies to characterize, and develop the granular product were accelerated.

Either method of stabilization of the wet cake can produce a product that can be turned into a low cost atomizable slurry fuel. However, the granular form has the potential to be used directly as a boiler fuel and bypass any further grinding. Therefore, the granular mull was chosen as the preferred form.

6.0 PREPARATION OF SLURRIES FROM MULLED COAL

Initially, dilute solutions of various surfactants were added to the granular product and indeed slurries could be made. The surfactants that seemed to work fastest and make a less viscous slurry had the highest HLB (hydrophobic lithophobic balance) number (e.g., the most water soluble products). A series of experiments were carried out in which granular products were produced from filter cakes of different moisture contents and different particle size distributions. To each of these stabilized filter cakes was added 1% of the neat (solid) surfactant and the mixture stirred. All formed liquid slurries. The viscosity of each slurry was measured using the Haake Rotovisco viscometer. The results are presented in Figure 3. It can be seen that the most profound effect upon the viscosity of the resulting slurries was the initial water content of the filter cake rather than particle size distribution. At 40% moisture the particle size, at least in the range tested, appeared to have no effect.

Figure 2
Particle Size Distribution of
Feedstock used for Mulled Coal Development

up to 20 chars	: ch-top %pass %-ch	: summary data
	: 704.00 100.0 0.0	: dv: 0.0186
	: 497.80 100.0 0.0	: %10: 5.10
MM9	: 352.00 100.0 0.0	: %50: 24.12
MICROTRAC DATA: DATA BASE UPDATE	: 248.90 100.0 0.8	: %90: 70.73
measurem't/present'n#: 479 - 1:	: 176.00 99.2 1.2	: mv: 32.62
7997 0.69-704. SRA res: NORMAL	: 124.45 98.1 2.6	: cs: 0.477
chan select bounds: 0.69- 704.00	: 88.00 95.4 8.1	:
progression: geom/sqrt2 # chans:20	: 62.23 87.3 12.9	: sd: 25.03
sample date/time: 12/07/90 15:59	: 44.00 74.4 13.2	: ma: 12.59
sample ident: UE3-304-DCC-H	: 31.11 61.2 14.6	LMM
sample ID : MULLED COAL FEED	: 22.00 46.6 13.0	: parameters
lot code:	: 15.56 33.6 9.3	: name value
account #:	printer: OFF: 11.00 24.3 7.5	:smpl amt 0.000
run time: 10 sec. run #: 100/ 3	: 7.78 16.8 5.7	:disprsnt
sample preparation code: 0	: 5.50 11.0 4.3	:disp amt 0.000
notes:	: 3.89 6.7 3.3	:disp med
	: 2.75 3.4 2.8	:dmed amt 0.000
	: 1.94 0.5 0.5	:agitat'n
	: 1.38 0.0 0.0	:agit tim 0
	: 0.97 0.0 0.0	:circul'n
	:	:circ tim 0
MM9	:	:param #1 0.000
Esc-exit	F2-commands :	:param #2 0.000
		:param #3 0.000

**UE3-304-DCC-H
MULLED COAL FEED
Record Number: 479**

Table III

TABLE 3

MULLED COAL FORMULATION DEVELOPMENT

Prep. Date	% Moisture*	Reagent	Funnel Flow Test			Stability Check		
			% Added*	W/O Gum	W/Gum***	8/22/90	8/23/90	8/27/90
8/13/90	30%	Pluronic F68	0.50	Hi Flow	Lo Flow	settled out will remix		settled out hard remix
8/14/90	30%	Pluronic F68	1.00	Hi Flow	Med. Flow	settled out will remix		settled out hard remix
8/14/90	30%	Pluronic F68	1.50	Hi Flow	Med. Flow	no soft pack good		no change good
8/14/90	30%	Pluronic F68	2.00	Hi Flow	Med. Flow		soft pack easy remix	no change
8/14/90	40%	Pluronic F68	0.50	Hi Flow	Hi Flow		soft pack easy remix	no change
8/14/90	25%	Pluronic F68	0.50	no test, too much foam			foam like no soft pack	no change
8/15/90	25%	Pluronic F68	1.00	no test, too much foam			foam like no soft pack	no change
8/15/90	25%	Pluronic F68	1.50	no test, too much foam			foam like no soft pack	no change
8/15/90	30%	Napthalene Sulfonate**	0.50	no test, solid like material			no change	no change
8/15/90	30%	Napthalene Sulfonate**	1.00	no test, solid like material			no change	no change
8/15/90	30%	Napthalene Sulfonate**	1.50	no test, solid like material			no change	no change
8/16/90	30%	Napthalene Sulfonate**	2.00	no test, solid like material			no change	no change
8/16/90	30% (micronized coal)	Pluronic F-68	1.00	Hi Flow	Hi Flow		soft pack	soft pack hard to mix

* percent water and reagent addition based on 300 grams of dry coal

** addition based on active ingredient from a 40% ammonium polynapthalene sulfonate solution

*** ~0.03% xanthan gum

Table III (cont'd)

TABLE 3 (cont.)

MULLED COAL FORMULATION DEVELOPMENT

Prep. Date	% Moisture*	Reagent	% Added	Funnel Flow Test		Stability Check	
				W/O Gum	W/Gum***	8/22/90	8/23/90
8/17/90	30%	Pluronic F-84	0.50	no test, too much foam		8/22/90	8/27/90
8/19/90	30%	Pluronic 84	1.00	no test, too much foam			still foam no soft pack
9/20/90	30%	Pluronic 84	1.50	no test, too much foam			still foam no soft pack
9/20/90	30%	Pluronic 84	2.00	no test, too much foam			still foam no soft pack
8/21/90	30%	Tergitol T 15-5-40	0.50	no test, too much foam			still foam no soft pack
8/21/90	30%	Tergitol T 15-5-40	1.00	no test, too much foam			still foam no soft pack
8/21/90	30%	Tergitol T 15-5-40	1.50	no test, too much foam			still foam no soft pack
8/21/90	30 to 35%	Tergitol T 15-5-40	2.00	no test, too much foam some ooze at 35% moisture			slurry like no soft pack
8/21/90	35%	Tergitol T 15-5-40	1.00	Free Flow	HI Flow		slurry like no soft pack
8/21/90	35%	Pluronic 84	1.00	Free Flow	HI Flow		slurry like no soft pack
8/22/90	30%	Lignosol TS****	4.00	no test, too much foam			foam no soft pack
8/22/90	33%	Lignosol TS****	4.00	ooze	no test		heavy slurry
8/23/90	24 to 32%	Pluronic F-68	2.00	foam, no test	Lo Flow		hard pack

* percent water and reagent addition based on 300 grams of dry coal

** addition based on active ingredient from a 40% ammonium polynaphthalene sulfonate solution

*** ~ 0.03% xanthan gum **** 50% active solution

Table III (cont'd)

TABLE 3 (cont.)

MULLED COAL FORMULATION DEVELOPMENT

Prep. Date	% Moisture*	Reagent	Funnel Flow Test		Stability Check	
			% Added* W/O Gum	W/Gum***		
8/23/90	24 to 32%	Pluronic F-68	0.50 Hi Flow	Hi Flow	8/23/90	8/27/90
8/23/90	33#	Pluronic F-68	1.00 Hi Flow	Hi Flow		9/04/90
8/24/90	33%	Tergitol T 15-5-40	0.50 Med. Flow	ooze		hard pack
8/24/90	33%	Tergitol T 15-5-40	1.00 Med. Flow	ooze		hard pack
8/29/90	33%	Pluronic F-68	0.50 Hi Flow	ooze		soft pack easy mix
8/29/90	33%	Pluronic 84	1.00 Lo flow	no test		soft pack easy mix
8/29/90	33%	Tergitol T 15-5-40	1.00 Hi Flow	Hi Flow		soft pack hard mix
8/30/90	33%	Naphthalene Sulfonate**	0.20 ooze	no test		no soft pack
8/31/90	33%	Naphthalene Sulfonate**	0.40 Hi Flow	ooze		hard pack
8/31/90	33%	Lignosol TS****	2.00 Hi Flow	slow flow		no change

* percent water and reagent addition based on 300 grams of dry coal

** addition based on active ingredient from a 40% ammonium polynaphthalene sulfonate solution

*** ~ 0.03% xanthan gum **** 50% active solution

Figure 3
Effect of Particle Size and Moisture Content
on Slurry Viscosities

Figure 4
Effect of Surfactant Concentration
on Slurry Viscosities

Figure 5
Effect of Surfactant Concentration
on Slurry Viscosities

The effect of surfactant concentration is shown in Figures 4 and 5. Figure 4 shows the effect of filter cake moisture content and surfactant concentration. The moisture content has the greater effect on viscosity than surfactant concentration in the ranges evaluated. The slight increase in viscosity observed at 2% surfactant concentration at all three moisture levels has been observed by others in slurry studies and has been attributed to stearic hindrance between particles due to multiple layers of surfactant on the particle surfaces. The results of these experiments show the feasibility of viscosity control by a variety of preparation methods and techniques.

Mull formulations containing gums can be thinned under high shear. This can be seen in Figure 6, the graph from the Haake Viscometer in which the mull was sheared for four minutes at the maximum shear rate (229/sec.) and then the down curve taken. The drastic change in viscosity (i.e., the drop in shear stress values with shear rate) is apparent. The close agreement of the second set of curves indicate that the reduction due to shearing was completed (e.g., there is little or no hysteresis in the second set of curves).

7.0 SCALE-UP OF MULLED COAL PREPARATION

In an effort to scale up the preparation of the mulled coal, it was decided to try to make the mull in a commercial mortar mixer. 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 ballmill 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

Figure 6
Effect of Shearing on the Viscosity
of "Gelled" Milled Coal

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. 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 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.

8.0 PROPERTIES OF GRANULAR MULLED COAL

8.1 Bulk Densities

The bulk densities of the granular mulled coal products were measured by pouring the granular product through a powder funnel into a tarred 100 ml graduated cylinder and then weighing and measuring the volume of product. Attempts were made to allow the powder to settle with as little compaction as possible. The values reported are the average of five separate determinations. Figure 7 shows the measured bulk densities of the various size fractions of the Upper Elkhorn #3 coal as a function of the fraction topsize. It can be seen that above a size range of 30 x 100 mesh the bulk densities do not change very much.

Figures 8, 9, and 10 show the measured bulk densities of 30% moisture filter cakes having different mean volume diameters (Mv's) as a function of the NPA-1 additive concentration. While no explanation is offered at this time for the various shapes of the resulting curves it has been consistent in all measurements that the maximum bulk densities are obtained at about the 1% additive level regardless of the mean volume diameter. Once compaction tests are carried out these numbers will be useful in determining the best storage conditions such as bin heights, shapes, etc.

8.2 Drying Studies

The formulation work showed that the wet filter cakes would rapidly lose water after treatment with the NPA-1 additive. However, the resulting material would retain its free flowing, dustless properties. It was decided to study the drying properties of these mulls with the thought of developing a process that would be better (i.e., faster, cheaper, etc.) than the current thermal drying process. The work carried out thus far is summarized in Figures 11, 12, 13 and 14. The apparatus used to make the measurements is shown schematically in Figure 15.

Figures 11 through 13 give the drying curves as a function of filter cake particle size at three different drying temperatures while Figure 14 gives the drying curves for a micronized coal at air temperatures ranging from room temperature to 64 degrees C. Particle size had little effect upon drying times, i.e., the time to remove 30% moisture and that, with the excep-

Figure 7
Bulk Densities of Coal Fractions

Figure 8
Effect of Additive Concentration on the Bulk Densities
of 30% Moisture Filter Cakes

Figure 9
Effect of Additive Concentration on the Bulk Densities
of 30% Moisture Filter Cakes

Figure 10
Effect of Additive Concentration on the
Bulk Density of 30% Moisture Filter Cake

Figure 11

Drying of 30% Moisture Stabilized Wet Filter Cake

Figure 12

Drying of 30% Moisture Stabilized Wet Filter Cake

Figure 13
Drying of 30% Moisture Stabilized Wet Filter Cake
Air Flow Rate = 33.9 scfh
Air Temperature = 64 deg. C

Figure 14

Drying of 30% Moisture Stabilized Wet Filter Cake

Figure 15

MULLED COAL DRYING APPARATUS

tion of room temperature, most of the moisture was removed in three hours at the air flow rate used (33.9 scfh) regardless of temperature. This suggests that there could be dramatic drying of the mull during pneumatic transfer of the product and that, in all likelihood, water would have to be added at the burner front to prepare sprayable slurries.

8.3 Angle of Repose

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 in some cases 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 IV.

TABLE IV

ANGLE OF REPOSE MEASUREMENTS FOR THE UPPER
ELKHORN #3 COAL AS A FUNCTION OF PARTICLE SIZE

<u>Material</u>	<u>Angle of Repose</u>
1/4" x 10 mesh coal	20 degrees
10 x 30 mesh coal	28 degrees
30 x 100 mesh coal	24 degrees
100 x 200 mesh coal	23 degrees
200 x 0 mesh coal	could not measure

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 V.

TABLE V

ANGLE OF REPOSE FOR MULLS PREPARED FROM
UPPER ELKHORN #3 COAL OF DIFFERENT
PARTICLE SIZE DISTRIBUTIONS

<u>Material</u>	<u>Angle of Repose, °</u>
Mv = 88 microns*	26
Mv = 41 microns	25
Mv = 20 microns	23

* 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 particle size has little effect upon the angle of repose and that the granular mulled can be stacked like coal.

9.0 ENVIRONMENTAL ANALYSIS

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 "gel" 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 a proprietary additive for stabilization while maintaining the original 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 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 additive that does not require hazard 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 polyglycol 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 waxy solid 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 126 degrees F for product effectiveness, not environmental or safety reasons. Flash point for the surfactant is greater than 500 degrees 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.

10.0 CONCEPTUAL SYSTEMS DESIGN

10.1 Systems Design for Mullied Coal

The systems should have the following characteristics:

Mull Preparation: A low shear mixer with capabilities 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.

The conceptual design for the mulled coal preparation storage and transportation system are shown in Figure 16.

10.2 Coal Water Fuels Preparation Circuit Design

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 diagram of the CWF preparation circuit is shown in Figure 17. The projected capacity will be 10-100 lbs/hour of mull.

11.0 CONCLUSIONS AND RECOMMENDATIONS

The feasibility studies of the Mulled Coal Contract (Phase I) resulted in the production of two distinct types of mulls, the "gel" form and the "granular" form.

The "gel" mull is of the type anticipated in the original proposal, i.e., a highly viscous liquid with consistency ranging from that of bread dough to a heavy oil. The formulations are similar to those for coal-water fuels (CWF). Conversion to CWF can be achieved by shearing the mull at high shear rates ($>200/\text{sec}$) without using additional chemicals or by high shear mixing with water.

The "granular" form of the mull was discovered when a small amount of a specific additive was added to a wet filter cake resulting in a granular product. The material has the consistency of dry powder with a particle size several times larger than the feed coal while still retaining the moisture content of the original filter cake. The formulation utilizes a reagent readily available at costs 10% that of the gel type. The granular mull can be converted to CWF utilizing conventional CWF reagents in a low shear system. The preparation of the granular mulls seem insensitive to moisture levels, feed particle size, or variations in coal processing. Initial studies suggest that loss of moisture during storage has little effect on the handling characteristics.

Figure 16

CONCEPTUAL FLOW SCHEME FOR CONVERTING WET FILTER CAKE TO MULLED COAL

Figure 17

CONCEPTUAL FLOW SCHEME FOR CONVERTING MULLED COAL TO LIQUID SLURRY FUEL

Energy International Corp.

The granular form of the mulled coal is the formulation of choice because of the:

1. Consistency of handling characteristics over a range of moisture levels, coal particle sizes, and coals.
2. Lower reagent costs.
3. Potentially simpler storage, handling and transportation systems.
4. Easy conversion to coal-water fuels.

The designation of the granular mull as the preferred form results in the following:

1. Subsequent work will be on granular formulations.
2. Preliminary results suggest that the original problems anticipated during transport will be considerably simplified. This is especially true for generation of hard pack due to the low frequency vibrations and viscosity changes due to temperature cycling.
3. Storage, handling, and transport now becomes more of a solids handling issue with the likelihood of identifying existing systems that can serve as prototypes.
4. Stability issues are now related more to surface phenomena than to bulk properties. Preliminary results indicate that the bulk properties are relatively constant over a wide range of feedstocks. However, issues such as oxidation and alteration of the coal surfaces are more critical for the higher surface areas of solids relative to the plan for gels.
5. CWF preparation may become more complex due to the pronounced transition from the solid to liquid properties with an anticipated requirement to add both reagents and makeup water to achieve necessary requirements for viscosity and atomization.

Phase I activities are defined such that these changes can be readily implemented with minor impact on the Work Plan. However, these changes will have a greater impact on Phase II of this program where the demonstration systems will be designed, constructed and operated.

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