4.2 Beneficiation of Low-Rank Coals

BENEFICIATION OF LOW-RANK COALS

Annual Technical Project Report for the
Period July 1, 1989 through June 30, 1990

Including

the Quarterly Technical Progress Report
for the Period April through June 1990

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BENEFICIATION OF LOW-RANK COALS

1.0 GOALS AND OBJECTIVES

The overall objectives of the Low-Rank Coal Beneficiation project were to study methods of reducing the inorganic content and increasing the heating value of low-rank coal (LRC) to produce high-quality dry coal products and/or coal/water fuels (CWF). The University of North Dakota Energy & Environmental Research Center (EERC) has demonstrated that high-energy content dry or slurry fuels can be produced from lignite and subbituminous coal using the hot-water drying process (HWD) and that low-ash, coal-based fuel can be produced using a combination of physical and chemical cleaning processes. These processes were integrated to achieve a greater than 400 lb/hr pilot-scale throughput for producing clean, energy-dense coal and/or CWF from low cost, highly reactive LRC feedstocks. These pilot-scale capabilities continue to supply fuels to DOE-sponsored Combustion, Gasification, and Heat Engine programs running concurrently with the Low-Rank Coal Beneficiation Program.

Past year LRC beneficiation project research was conducted at the bench-scale level using the EERC's cold-charge autoclave and laboratory coal processing equipment. Sample characterization was performed by UNDEERC's coal analysis and water analysis laboratories. The Fuels & Process Chemistry Group provided significant support for the oil agglomeration process on lignites and subbituminous coals.

The specific objectives for the EERC LRC Beneficiation project for the past year were to:

1. Evaluate oil agglomeration with acid leaching as a combined process.
2. Investigate colloidal coal-cleaning (CCC) based on previous efforts.
3. Explore pressurized hot-water drying (HWD) with direct air injection (partial oxidation) heating.

Major emphasis was placed on the oil agglomeration task. The CCC technique investigations were not attempted due to the volume and depth of the oil agglomeration testing. The partial oxidation hot-water drying (PO-HWD) was completed and coordinated with similar testing for the State Electric Commission of Victoria, Australia, which also funded work on partial oxidation processing.

Near-term objectives for the period April through June 1990 were to:

1. Design, perform, and evaluate the extended oil agglomeration test matrix to determine the optimal acid strength, oil strength, and oil mix time conditions for beneficiating a North Dakota lignite.

2. Complete PO-HWD characterization on a North Dakota lignite concurrent with identical PO-HWD testing for another client.

2.0 INTRODUCTION

This report describes progress towards goals and objectives established previously (1), as well as those listed in section 1.0.

3.0 BENEFICIATION OF LOW-RANK COALS

3.1 Oil Agglomeration

3.1.1 Background

The preparation of clean, stable coal combustion and/or conversion (i.e. liquefaction, gasification) feedstocks is a priority for the future. Additionally, the feedstock preparation processes must achieve the irreversible removal of moisture, particularly in LRCs, an increase in Btu content, and the production of products not prone to spontaneous combustion. Acid cleaning of finely ground coal followed by oil agglomeration accomplishes these improvements using common chemicals and equipment, and brief, uncomplicated processing conditions.

Oil agglomeration of bituminous coals has been successfully carried out for many years. However, agglomeration of LRCs has achieved limited success, until recently, due to the different chemical nature of LRCs. The EERC oil agglomeration process for beneficiating LRCs has been highly successful in producing uniform, low-ash, low-moisture, oil-bound coal particles of varying sizes.

Preliminary work to remove mineral matter and agglomerate coal fines on the laboratory scale began with Illinois #6 bituminous coal. Although the ash content of as-mined bituminous coals may be quite high, most of the ash results from detrital mineral deposits. This detritus is commonly removed by washing at the mine, leaving only a fraction of the original mineral content to be removed by coal-cleaning processes. Removal of most of the remaining mineral material can be done with pH adjustment during agglomeration. However, with contaminants such as pyritic sulfur, which is best removed at a pH between 7-11, the coal becomes more hydrophilic and agglomeration is less effective (2). To achieve maximum ash removal and optimum agglomerate size, and to reduce agglomeration time, multiple pH adjustments are required. Bituminous coals are characterized by relatively few oxygen functionalities, few ion-exchangeable cations, low moisture contents, and limited pore structure. These characteristics make the coal surface quite compatible with nonpolar hydrocarbon oils, thus quite amenable to oil agglomeration. In the EERC's preliminary experiments with the Illinois #6 coal, the effects of coal particle size, various oil-coal ratios, mixing speeds and times were defined (3).

The successful results from the tests with the bituminous coal were applied to hot-water-dried (HWD) Indian Head lignite. The chemical surface of the HWD coal was apparently similar to that of the bituminous coal and behaved similarly under agglomeration conditions. The raw lignite, however, behaved much differently than the bituminous or HWD lignite coals using the previously successful agglomerating conditions, due to the differences in surface functional groups and moisture content.

The differences in surface chemistry between bituminous coal and LRCs suggest that the compatibility of the coal and binding oil must be better understood. Most oils used for agglomeration are nonpolar or only slightly polar and, as a result, are readily adsorbed on the surface of coal particles that have a minimum of exposed polar groups. The subbituminous and lignitic coals contain large amounts of surface oxygen, making their surfaces more oleophobic than those of the bituminous coals. Ion-exchangeable cations which are not compatible with the nonpolar or slightly polar oils are found on many of the oxygen functionalities. The LRC structure is also rich in moisture-filled pores which further contribute to phobicity toward nonpolar oils. Since the theory of agglomeration assumes the mineral material is considerably more hydrophilic and oleophobic than the organic coal matrix, the mineral material will dissolve or form a suspension in an aqueous medium. The moisture in the pores can be at least partially removed through displacement by water-soluble polar organic liquids. When the desired mineral and moisture removal conditions have been met, the oleophilic organic matter will form aggregates and separate from the aqueous phase.

In successful agglomeration, the mineral content of the coal will be reduced significantly as the coal forms aggregates of organic-rich material, and the detrital and chemically deposited minerals are suspended in the aqueous phase. The degree to which a coal can be beneficiated by agglomeration is limited by several factors. The first factor is the particle size. The smaller the particle size, the more liquid-solid surface contact and, consequently, the better the chances of the carbonaceous material liberating its associated minerals, thus lowering the ash content. Although fine grinding enhances inorganics removal, it creates product-handling problems and increases the area for undesirable surface reactions with oxygen. Effective agglomeration immediately following ash reduction helps to solve these problems.

The second factor to be considered is the composition of the oil used as a binder. Light agglomerating oils (density < 0.90 g/cc) have been shown to reduce ash contents in bituminous coals to within 10 to 20 percent of those obtained with the Stoddard solvent (similar to Certigrav washability specific gravity fluids) (2). These oils, however, do not sufficiently wet the surface of low-rank coals and are not useful as binding oils for these coals. Heavier oils such as coke oven tars, pitches, and petroleum crudes can agglomerate LRCs, but these oils are more difficult to recover for reuse. The third factor is the pH of the coal/water mixture during agglomeration. An optimum pH provides cation exchange on the organic moieties as well as dissolution of some inorganics.

The size of the agglomerates produced is a function of, but not limited to, mixing speed, mixing time, mixer design, binding oil composition, and oil-to-coal ratio. Physical impact forces, resulting from contact with surfaces of small areas such as stir-blade edges, reduce the agglomerate size as mixing speed and mixing time increase (4). To minimize these effects, a blunt stir bar, providing maximum contact area between agglomerates and bar, is recommended. Increasing the ratio of binding oil to coal tends to increase agglomerate size; however, binding oil recovery becomes an economically important aspect of this method of beneficiation. Recovering 40 to 50% of the binding oil by thermal treatment maintains the calorific value of the product, decreases the moisture content, increases mechanical stability, and results in reduction of self-ignition hazards (5).

Successful EERC beneficiation studies on Indian Head lignite using oil agglomeration served as the basis for the agglomeration technique (6,7). In those studies, -200 mesh and micronized (100% -325 mesh) Indian Head lignite was acid leached, treated with surfactant, and then agglomerated with a phenolic stream from the Dakota Gasification Plant (formerly Great Plains Gasification Plant). Operating conditions included ambient temperature and pressure, and blender stirring rates of <3000 rpm. The tar-oil stream from the same plant was also used successfully as the binding oil without additional surfactant. Recoveries as high as 95% coal on a moisture-oil-free basis and agglomerate ash levels as low as 0.7% were achieved. Typically, recoveries were greater than 80% and ash levels ranged from 1-2%. The successful operating conditions were applied to Beulah lignite in an agglomeration test matrix designed to obtain data for eventual process scale-up.

3.1.2 Experimental Procedure

The EERC agglomeration procedure is divided into two steps: acid cleaning followed by oil agglomeration. The acid cleaning step has undergone continual evolution from the preliminary scoping tests up to performance of the agglomeration test matrixes. The initial tests used a sonication bath to enhance the separation of mineral matter that is not ion-exchangeable. Sonication was replaced by stirring, primarily because sonication was not considered scaleable to pilot, development, or production size. Stirring was provided by a simple malt mixer with a T-bar stirrer. The malt mixer was eventually replaced, prior to matrix testing, with a high RPM, high-shear, dispersion type mixer. The high-shear mixer provides intense agitation for acid leaching and is scaleable to larger sizes.

The leaching step consists of mixing approximately 52 grams of coal with 102 milliliters of a dilute acid solution. The coal/acid solution is agitated for a determined length of time using the high-shear mixer. The high-shear mixer is capable of shaft speeds up to 20,000 rpm. After the cleaning step, a surfactant can be added to lower the solution surface tension. The sample is mixed for 4 minutes in order to assure surfactant adherence. The sample is then diluted with 102 ml of distilled water to increase the sample volume. The solution is mixed for an additional 2 minutes.

The agglomeration step consists of adding 50 milliliters of binding oil to the solution of coal, water, dilute acid, and surfactant. The sample is mixed using a low speed malt mixer with T-bar for a determined length of time, during which agglomeration occurs. The agglomerates are collected on a 30-mesh screen. The fines that wash through the screen are separated from the cation-laden acid water by Buchner filtration. The fines and the agglomerates are air-dried overnight prior to analyses.

3.1.3 Data Interpretation for Screening and Matrix Tests

Data on agglomerate yields, oil uptake by the agglomerating coal, and agglomerate ash and moisture were obtained for all screening experiments. Agglomerate yields were determined after air-drying the washed agglomerates. Unagglomerated fines were collected on filter paper and weighed after air-drying. The coal recovery was determined as the ratio of agglomerate weight/coal feed weight on a fixed carbon basis, assuming that the oil weight measured by TGA was the binding oil's. Ash reduction was based on the ash content of the agglomerate compared with the ash content of the original coal on a fixed carbon basis. The data for calculating yield and ash reduction were obtained by TGA from a modification of proximate analysis as follows:

> 40-50 milligrams of coal feed or agglomerated material was placed on the sample pan of a DuPont 951 Thermogravimetric Analysis (TGA) module interfaced with a DuPont 1090 Thermoanalyzer. The sample was heated under flowing argon at 20°C/minute to ~110°C and held at that temperature for 5 minutes. The temperature was then increased to ~250°C at 100°C/min and held for 15 minutes, followed by heating to ~900°C at the same rate where it was held for the duration of the analysis. At ~32.5 minutes into the run, the argon flow was stopped, and air flow was started. The analysis was terminated at ~55 minutes, and the data was plotted and analyzed for moisture, oil, volatiles, fixed carbon (combustible residue), and ash. Examples of coal and agglomerate proximates by TGA are shown in Figure 1.

Ash and moisture data on selected larger (~1 gram) samples (and other samples not analyzed by TGA) were also obtained, for comparison, by a method modeled after ASTM method D271-58.

3.1.4 Analyses of Screening and Matrix Test Coals

Table 1 shows the proximate analyses of the coals used in the screening and matrix tests, as determined by TGA. The proximate, ultimate, and heating value analysis, as determined by ASTM methods, for Indian Head and Beulah lignites and Kemmerer subbituminous coal are contained in Table 2. Beulah was investigated because it was chosen as the coal to be studied in the test matrix. Kemmerer was tested for possible oil agglomeration because the coal had been investigated in previous LRC cleaning processes (1,8). Prior to the development of the test matrixes, Indian Head was investigated for oil agglomeration capabilities (3). The results from the oil agglomeration of Indian Head are presented mainly for background and comparison with matrix test results.

3.1.5 Washability Results for the Matrix Test Coal

Prior to oil agglomeration testing, washability (heavy liquid separation) testing was performed on samples of -30 mesh x 0 and -325 mesh x 0 screened fractions of Beulah lignite. The washability was done on an as-received sample and an air-dried sample of each sieve fraction to determine if there was any benefit in air drying the coal, as suggested by other researchers (9).

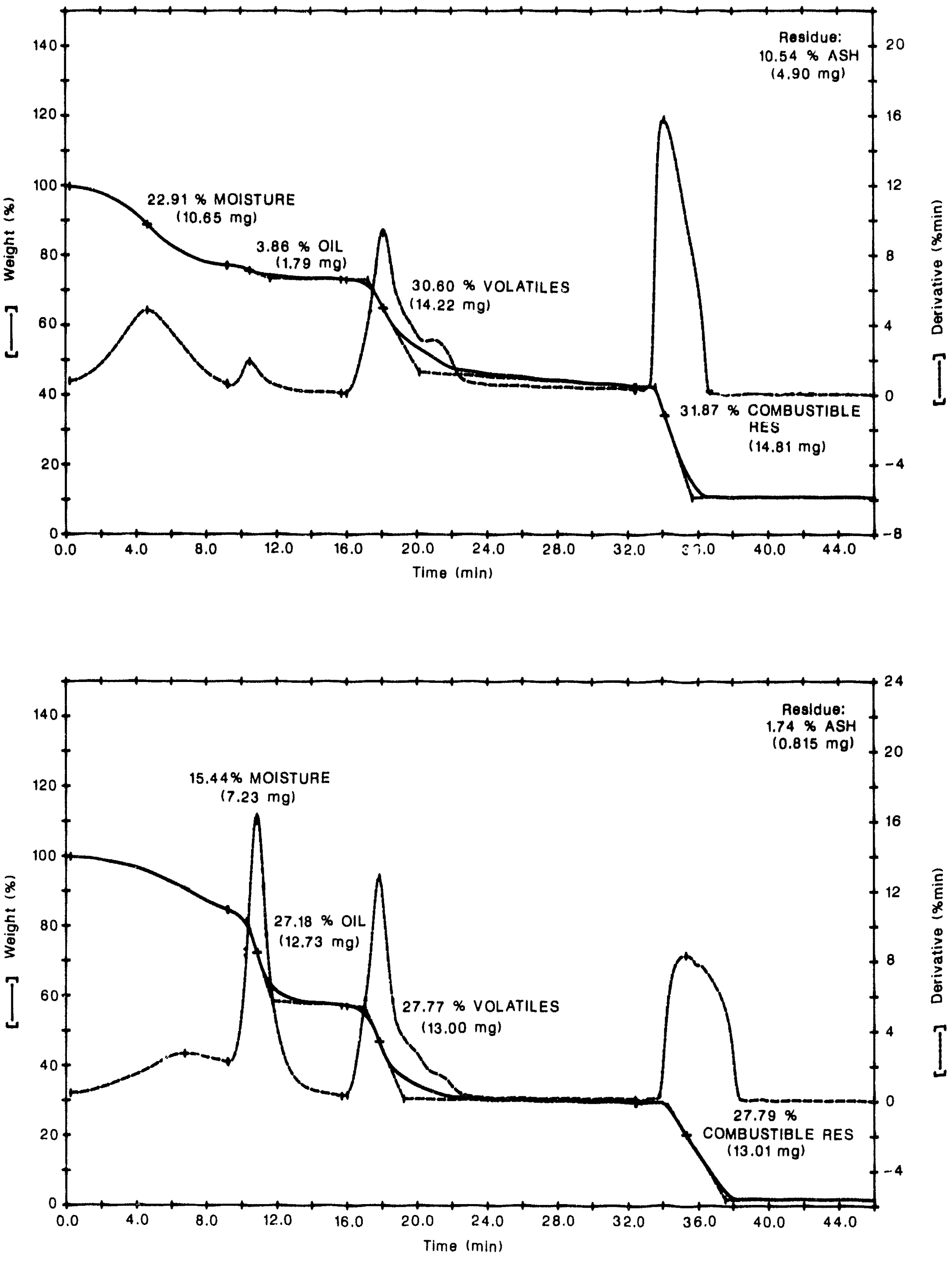

Figure 1. TGA determined proximate analyses for -30 mesh x 0 Beulah lignite agglomerate products.

TABLE 1

PROXIMATE TGA ANALYSIS OF LOW-RANK COALS USED IN THE OIL AGGLOMERATION SCREENING AND MATRIX TESTS

Analysis, wt%	Indian Head		Beulah	Kemmerer
Moisture	27.4	17.0	29.9	10.7
Volatile Matter (mf)	45.2	41.7	43.2	41.7
Fixed Carbon (mf)	46.6	43.1	47.0	53.8
Ash (mf)	8.2	14.9	9.8	4.4
Particle Size, Mesh	-200 x 0	-325 x 0*	-30 x 0	-200 x 0

* Fines not retained on 325-mesh screen.

TABLE 2

PROXIMATE, ULTIMATE, AND HEATING VALUE ANALYSES OF LOW-RANK COALS USED IN THE OIL AGGLOMERATION SCREENING AND MATRIX TESTS

	Indian Head	Beulah	Kemmerer
Proximate Analysis[a] (mf, wt%)			
Vol. Matter	45.2	43.2	41.9
Fixed Carbon	46.7	47.0	53.8
Ash	8.2	9.9	4.3
Ultimate Analysis[a] (maf, wt%)			
Carbon	68.2	70.0	76.7
Hydrogen	5.0	4.8	5.5
Nitrogen	0.9	0.9	1.4
Sulfur	0.9	1.1	0.2
Oxygen[b]	24.9	23.2	16.2
Heating Value[a] (mf, Btu/lb)	10,320	10,550	13,220

[a] ASTM determined.
[b] by difference.

These samples were selected according to the following criteria: the -30 mesh x 0 coal was the starting material used to obtain all the other fractions for the oil agglomeration study, and the -325 mesh x 0 fraction had been used to determine the fixed constants, such as acid strength, oil type, oil volume, etc., for the oil agglomeration test matrix. Additionally, the -325 mesh x 0 fraction of the total coal sample had the highest ash concentration. It was initially assumed that if this higher ash fraction was successfully cleaned and agglomerated, the other lower ash, larger size fractions will also be successfully cleaned.

The washability curves for the -30 mesh x 0 and -325 mesh x 0 fractions are presented in Figures 2 and 3. The specific gravity scale on the right-hand side of the graph corresponds to the points indicating the results for the various washability tests. For example, 50% of the air-dried -30 mesh x 0 Beulah lignite can be recovered at a specific gravity of 1.4. From the figures, the only differences in yield for the two coal samples are at the lower specific gravities. At higher than 1.4 specific gravity, the yields are practically identical. Figure 2 indicates air-drying the -30 mesh x 0 fraction does not benefit the ash separation compared to the as-received sample. However, Figure 3 indicates air-drying of the -325 mesh x 0 Beulah lignite liberates slightly more ash than the as-received sample. No significant difference was noticed between the -325 x 0 and -30 x 0 samples, with only a slight variation in specific gravities for the air-dried and as-received samples.

3.1.6 Comparison of Acid Cleaning-Oil Agglomeration to Washability-Acid Cleaning for the Matrix Test Coal

Acid cleaning-oil agglomeration and washability-acid cleaning were evaluated by comparing the clean coal ash content and clean coal yield achieved with each process. The results are presented in Table 3. The first process, acid cleaning-oil agglomeration, was performed according to the procedure described in section 3.1.2. Acid cleaning, during the second process, was performed on the combined float fractions produced from washability testing (physical cleaning) at 1.3, 1.4, and 1.5 specific gravity.

Acid cleaning-oil agglomeration produced a higher yield than washability-acid cleaning, but the ash contents were slightly higher. However, optimization was not performed on the acid cleaning-oil agglomeration process.

3.1.7 Coal Specific Agglomeration

Oil agglomeration was performed on Indian Head and Beulah lignites and Kemmerer subbituminous coal. The particle size for the coals was all combustion grind (80% -200 mesh). The binding oil used was crude phenol at a volume of 50 milliliters. The amount of surfactant, Triton X-100, used was 3.1 milliliters. A hydrochloric solution of 6.2 wt% was used for the acid cleaning part of the agglomeration. The volume of dilute acid used was 102 milliliters. During the acid cleaning, all samples were sonicated for a

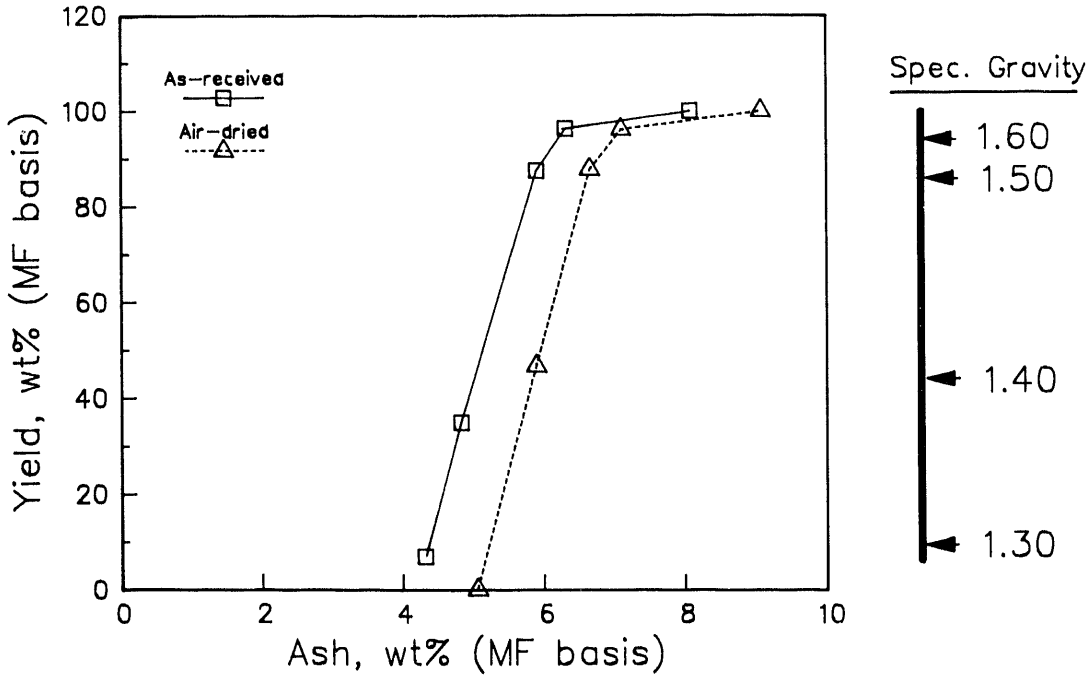

Figure 2. Washability performance of -30 mesh x 0 Beulah lignite.

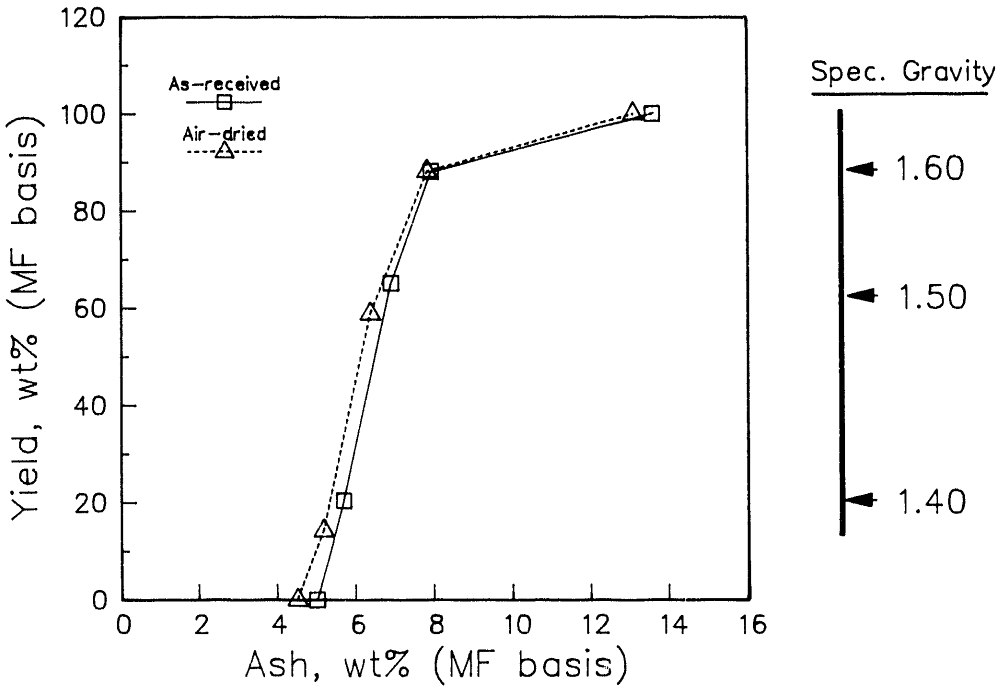

Figure 3. Washability performance of -325 mesh x 0 Beulah lignite.

TABLE 3

ACID CLEANING-OIL AGGLOMERATION VS. WASHABILITY-ACID CLEANING OF BEULAH LIGNITE

	Acid Cleaning-Oil Agglomeration		Washability-Acid Cleaning	
Sample	Ash, wt%	Yield, wt%	Ash, wt%	Yield wt%
-30 x 0	2.27 (3.68)*	89.24	1.61	83.11
-325 x 0	3.56 (4.86)*	91.41	2.18	60.90

* TGA determined ash contents are in Parentheses.

period of 30 minutes. During surfactant and binding oil addition, the samples were mixed 4 minutes for each addition. Table 4 is a summary of oil agglomeration testing on the three coals.

The results in the table indicate oil agglomeration as being beneficial for the coal samples. The agglomerate yields were based on the fixed carbon content of the agglomerates compared to the starting material fixed carbon content. The agglomerate yields in Table 5 were greater than 80% for all three coals and greater than 90% for two of the coals, indicating the agglomeration process was very successful. The selectivity of the process for simultaneously achieving high yields and low product ash contents was determined using the ash efficiency index (AEI). The AEI was calculated by the following formula:

$$\text{AEI} = \text{Agglomerate Yield(wt\%)} \times (\text{Refuse Ash(wt\%)}/\text{Agglomerate Ash(wt\%)}) \qquad [1]$$

Efficiency indices greater than 1500 indicate that the agglomeration process performs well with these coals. The ash reductions were somewhat lower for Beulah and Kemmerer than for Indian Head, which was expected because the process was optimized for Indian Head and not for the other two coals.

3.1.8 Fuel Product Quality

3.1.8.1 TGA Results

Analyses of the product agglomerates and reject fines was conducted by TGA to determine fuel quality. The procedure was described in section 3.1.4. The moisture content of the agglomerates was determined by ramping from ambient temperature to 110°C. The resulting weight loss consisted of water and a small, as-yet unidentified azeotrope, meaning that the actual water contents were somewhat lower than the reported values. The binding oil, which made up 15-25 wt% of the agglomerates, was removed by distillation over the temperature range of 110-250°C. Volatiles, fixed carbon (combustible residue), and ash in the agglomerates were assumed to be contributed by the

TABLE 4

SUMMARY OF OIL AGGLOMERATION PERFORMANCE DATA FOR INDIAN HEAD AND BEULAH LIGNITES AND KEMMERER SUBBITUMINOUS COAL

	Indian Head	Beulah	Kemmerer
Raw Coal Ash, wt%	7.86	9.07	4.41
Agglomerate Yield, wt%	84.80	96.80	97.60
ASTM Ash, wt%*	----	2.27	1.82
TGA Ash, wt%**	1.40	3.68	2.31
Refuse Ash, wt%	32.12	61.98	48.00
Efficiency Index Ash	1940	1630	2020
% Ash Reduction***	82.17	59.40	47.55

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* ASTM ash data for agglomerates is moisture free.

** TGA ash data for agglomerates is moisture-binding oil free.

*** Based on TGA ash determinations.

TABLE 5

ELEMENTAL CONTENTS IN ASH OF AS-RECEIVED AND ACID-LEACHED BEULAH LIGNITE AS DETERMINED BY X-RAY FLUORESCENCE ANALYSIS

	Coal	Coal	Coal(HCl)*	Coal(HNO_3)*
Mesh Size	-30 x 0	-325 x 0	-325 x 0	-325 x 0
Total Ash, wt%, mf	9.86	12.90	7.80	7.75
	% ELEMENTAL			
Silicon	9.28	11.21	24.77	23.68
Aluminum	5.11	5.23	9.67	9.18
Iron	6.14	6.36	8.54	9.58
Titanium	0.76	0.92	1.73	1.70
Phosphorus	0.52	0.55	0.30	0.31
Calcium	15.08	12.75	0.96	1.15
Magnesium	3.20	2.64	0.55	0.54
Sodium	5.86	4.26	0.00	0.00
Potassium	0.20	0.42	0.84	0.76
Sulfur	8.80	10.00	0.92	1.08
Oxygen (Difference)	42.29	44.81	44.30	43.38
Wt% Ash as Oxides	97.24	99.16	92.58	91.36

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* Acid used in leaching coal prior to forming agglomerates.

coal. Since the weight of fixed carbon was least likely to change between coal and agglomerate and is a direct measurement by TGA, the coal recovery (yield) was calculated on a fixed-carbon-in versus fixed-carbon-out basis. Ash reduction, which ranged from 50 to 80 wt%, was calculated on a coal basis. Please see Figure 1 and Table 4 for fuel quality data.

3.1.8.2 X-Ray Fluorescence Analysis (XRFA) Results

Removal of inorganics from Beulah lignite by acid leaching is indicated by the XRFA results shown in Table 5. The elemental composition of the ash from the coal varied between the -30 x 0 and the -325 x 0 screenings. The fraction having smaller particle sizes contained more ash and was enriched in silicon and sulfur, but contained less calcium, magnesium, and sodium on a normalized % element basis. Following acid leaching of the -325 x 0 coal, silicon, aluminum, iron, titanium, and potassium were found to be enriched in the ash, while the remaining element concentrations, most notably those of calcium, magnesium, sodium, and sulfur, were significantly reduced. No acid-specific effect was noted when cleaning the coal with HCl or HNO_3 of equal strength. Ash reduction appeared to be independent of the acid used. XRFA data in Table 5 indicates that the acids similarly affected the removal of inorganic elements in the coal.

3.1.9 Acid Selection for Leaching

Acid leaching of soluble or ion exchangeable mineral material was used as a cleaning step prior to agglomeration of the coal particles. In preliminary tests, 6.2 wt% hydrochloric acid solution was used as the reagent acid solution. Mineral matter content was successfully decreased as indicated in Table 5 using this acid. However, analysis showed a residual chloride in the agglomerate of 0.3 wt% when the agglomerates were prepared by this procedure. This level of chloride was unacceptable. Tests with a 6.2 wt% nitric acid solution resulted in mineral matter levels comparable to those produced by the HCl, and therefore HNO_3 was substituted for HCl. Analysis to determine nitrate residue in the agglomerates has not been completed, but is expected to show only small amounts of additional nitrogen in the agglomerates. This is based on an analysis of nitrogen content in the agglomerates that showed an increase of less than 0.1 wt% nitrogen. This was thought to be negligible in comparison to the hydrochloric acid; therefore, it was decided to use nitric acid for the matrix testing.

3.1.10 Karl Fischer Moisture Analysis Method

Gravimetric determinations of moisture (by ASTM method D271 and TGA proximate analysis) in the agglomerates and fines proved unsatisfactory due to the concurrent loss of low-boiling organics on heating the samples to 105°C. These low-boiling organics originate from the binding oil in the agglomerates and make up a large fraction of what had previously been classified as weight loss due to the removal of moisture. Alternatively, to provide a more accurate measure of moisture, the water contents of the agglomerates were determined by Karl Fischer analyses.

3.1.11 Oil Agglomeration Test Matrix

The test matrix developed for the oil agglomeration of Beulah lignite was based on a 5-factor central composite design (10). The factors (independent variables) selected were particle-size fraction, acid contact time, acid leaching mixing speed, oil mixing time, and agglomerating mixing speed. Table 6 contains the factors and the levels for each factor. Responses (dependent variables) were agglomerate ash, moisture and oxygen content, mechanical properties, and agglomerate size. Based on optimal conditions from the first matrix, a large amount of agglomerates were to be made, and two additional factors were to be determined by one-at-a-time experiments. These factors were drying time and temperature for drying the agglomerates. For example, five identical samples will be put in a 66°C oven, and one sample will be removed for moisture determination at 1, 6.75, 12.5, 18.25, and 24 hours.

The matrix for the first five factors was composed of 32 tests in two separate blocks. The 5-level central composite design was chosen because it allows evaluation of curvature in the responses after performing only the factorial (19 tests) or first block of the design (11). If there is curvature in each of the responses, it is possible to determine the optimal operating conditions. The remaining 13 tests (second block) are used to evaluate responses at the star points and center points in the design. The run order of the experiments was randomized to eliminate bias. The run order and test factors (independent variables) for the matrix design are presented in Table 7. The times for acid and oil mixing are in minutes, and the speeds for these factors are in rpm's.

3.1.11.1 Performance Value

The performance value in Table 8 is an arbitrary number based on the agglomerate yield and the description of the agglomerates. The scale ranges from 0 to 5, with 0 referring to the poorest agglomerate production. The descriptions for the agglomerates ranged from distinct individual ones to clumped agglomerates. The clumped agglomerates could be bridged distinct smaller agglomerates, or they could be a large clump of nondiscernible agglomerates.

3.1.11.2 TGA Results

Analyses of the product agglomerates and the reject fines were performed by TGA to determine fuel quality. The procedure has been described previously (12). The ash contents of agglomerates prepared according to the test matrix are shown in Table 8.

3.1.11.3 Karl Fischer Moisture Determinations

Table 9 shows the Karl Fischer moisture in the agglomerates and fines prepared in accordance with the experimental matrix. The moisture levels in the agglomerates ranged from 7.07 to 0.75 wt% with a average of 1.58 wt%. However, 27 of the 32 agglomerate samples had <2 wt% moisture. The fines moisture level ranged from 14.89 to 0.90 wt% for 25 samples, with a 3.33 wt% average. It should be noted that the samples with 0.00 wt% moisture mean that not enough sample was produced in order to determine the moisture content.

TABLE 6

OIL AGGLOMERATION TEST MATRIX FACTORS AND LEVELS FOR EACH FACTOR

I. Central Composite Design Factors

Factor 1: Particle Size(X_1)	
- 30 x 60 mesh	(-2)
- 60 x 100 mesh	(-1)
-100 x 200 mesh	(0)
-200 x 325 mesh	(1)
-325 mesh x 0	(2)

Factor 2: Acid Contact Time(X_2)	
10.0 minutes	(-2)
22.5 minutes	(-1)
35.0 minutes	(0)
47.5 minutes	(1)
60.0 minutes	(2)

Factor 3: Cleaning Mixing Speed(X_3)	
4,000 rpm	(-2)
6,000 rpm	(-1)
9,000 rpm	(0)
13,500 rpm	(1)
20,250 rpm	(2)

Factor 4: Oil Mixing Time(X_4)	
2.0 minutes	(-2)
6.5 minutes	(-1)
11.0 minutes	(0)
15.5 minutes	(1)
20.0 minutes	(2)

Factor 5: Agglomerating Mixing Speed(X_5)	
100 rpm	(-2)
200 rpm	(-1)
400 rpm	(0)
800 rpm	(1)
1600 rpm	(2)

II. One-at-a-time Factors

Factor 6: Drying Temperature(X_6)	Factor 7: Drying Time(X_7)
25.0°C	1.00 hours
45.5°C	6.75 hours
66.0°C	12.50 hours
86.5°C	18.25 hours
107.0°C	24.00 hours

TABLE 7

INDEPENDENT VARIABLES FROM THE OIL AGGLOMERATION TEST MATRIX OF BEULAH LIGNITE

Matrix Test No.	Particle Size Feed Coal, mesh	Acid Mix Time	Acid Mix Speed	Oil Mix Time	Oil Mix Speed
1	-325 x 0	35.0	9,000	11.0	400
2	-100 x 200	35.0	9,000	20.0	400
3	-200 x 325	47.5	6,000	15.5	200
4	-100 x 200	35.0	9,000	11.0	400
5	-200 x 325	22.5	13,500	6.5	800
6	- 60 x 100	22.5	13,500	15.5	800
7	- 60 x 100	47.5	13,500	15.5	200
8	-200 x 325	47.5	6,000	6.5	800
9	-100 x 200	35.0	9,000	11.0	400
10	-200 x 325	22.5	13,500	15.5	200
11	-100 x 200	35.0	9,000	11.0	400
12	- 60 x 100	22.5	6,000	6.5	800
13	-100 x 200	35.0	9,000	2.0	400
14	-100 x 200	35.0	9,000	11.0	400
15	-100 x 200	35.0	9,000	11.0	400
16	- 30 x 60	35.0	9,000	11.0	400
17	- 60 x 100	47.5	6,000	6.5	200
18	-100 x 200	10.0	9,000	11.0	400
19	-200 x 325	22.5	6,000	6.5	200
20	-100 x 200	35.0	9,000	11.0	1,600
21	-100 x 200	35.0	4,000	11.0	400
22	- 60 x 100	22.5	13,500	6.5	200
23	-100 x 200	60.0	9,000	11.0	400
24	-200 x 325	47.5	13,500	15.5	800
25	- 60 x 100	47.5	6,000	15.5	800
26	-100 x 200	35.0	20,250	11.0	400
27	- 60 x 100	22.5	6,000	15.5	200
28	-100 x 200	35.0	9,000	11.0	400
29	-100 x 200	35.0	9,000	11.0	100
30	-200 x 325	47.5	13,500	6.5	200
31	-200 x 325	22.5	6,000	15.5	800
32	- 60 x 100	47.5	13,500	6.5	800

TABLE 8

DEPENDENT VARIABLES FROM THE OIL AGGLOMERATION TEST MATRIX OF BEULAH LIGNITE

Matrix Test No.	Agglomerate Ash (mf, wt%)	Agglomerate Yield (mf, wt%)	Performance Value
1	3.88	2.10	0
2	1.66	98.38	5
3	2.64	15.71	1
4	1.79	91.60	4
5	1.89	4.24	0
6	2.35	100.00	5
7	1.24	92.25	4
8	2.20	48.92	3
9	2.23	98.53	4
10	2.53	7.53	0
11	1.33	99.48	4
12	1.21	82.65	2
13	1.57	97.70	3
14	1.71	98.62	3
15	2.80	98.49	3
16	0.97	100.00	2
17	1.37	96.67	2
18	1.85	98.14	4
19	2.12	22.94	1
20	1.23	88.22	4
21	1.85	94.41	5
22	1.12	100.00	2
23	1.45	91.30	3
24	3.37	0.70	0
25	1.76	90.89	3
26	1.77	97.05	4
27	1.40	92.57	3
28	2.33	78.17	3
29	3.02	29.95	1
30	2.27	3.41	0
31	2.62	69.37	3
32	0.92	100.00	4

TABLE 9

BEULAH LIGNITE AGGLOMERATE AND FINES KARL FISCHER MOISTURE CONTENT

Matrix Test No.	Particle Size Feed Coal, mesh	Agglomerate H_2O, wt%	Fines H_2O, wt%
1	-325 x 0	1.65	7.33
2	-100 x 200	7.07	1.27
3	-200 x 325	0.90	14.89
4	-100 x 200	3.86	1.29
5	-200 x 325	0.89	7.72
6	- 60 x 100	0.75	0.00
7	- 60 x 100	0.86	0.90
8	-200 x 325	0.93	2.53
9	-100 x 200	0.90	1.12
10	-200 x 325	1.10	8.97
11	-100 x 200	0.80	1.03
12	- 60 x 100	1.05	0.90
13	-100 x 200	1.16	0.00
14	-100 x 200	1.13	0.00
15	-100 x 200	1.21	0.00
16	- 30 x 60	4.94	0.00
17	- 60 x 100	1.14	0.90
18	-100 x 200	1.34	1.34
19	-200 x 325	1.28	5.87
20	-100 x 200	2.02	0.90
21	-100 x 200	1.14	0.92
22	- 60 x 100	2.65	0.00
23	-100 x 200	1.09	1.02
24	-200 x 325	0.96	10.14
25	- 60 x 100	1.37	0.96
26	-100 x 200	0.87	0.00
27	- 60 x 100	1.11	1.01
28	-100 x 200	1.19	1.05
29	-100 x 200	1.40	3.16
30	-200 x 325	1.31	0.00
31	-200 x 325	1.33	1.35
32	- 60 x 100	1.16	0.00

These results indicate that large difference exists between the TGA and ASTM moisture analysis compared to the Karl Fischer determination.

3.1.11.4 Cleaning Efficiency Results

The ash efficiency index (AEI) was calculated using equation 1 presented in section 3.1.7. Figure 4 is a graphical representation of the feed coal particle size versus the AEI. It should be noted that some of the higher index values are not indicative of the type of agglomerates produced.

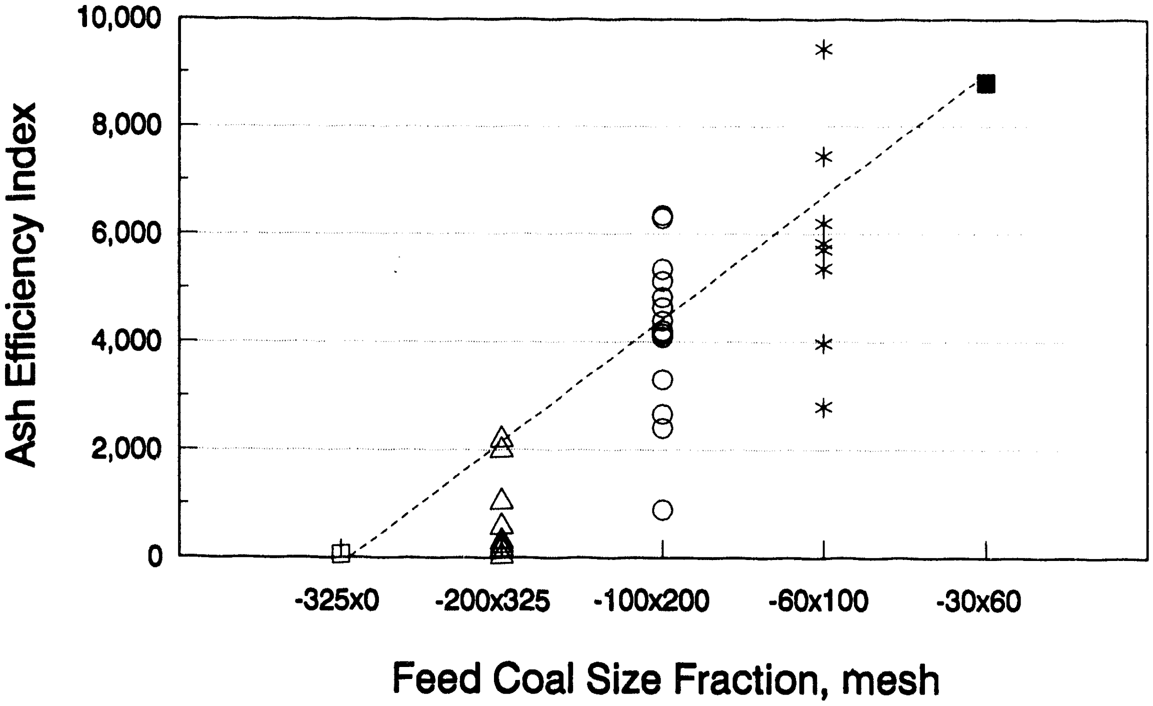

Figure 4. Ash efficiency index vs. coal particle size.

3.1.11.5 Statistical Analyses

The factors (independent variables) that were studied using the matrix design were feed coal particle size (X_1), acid mix time (X_2), acid mix speed (X_3), oil mix time (X_4), and oil mix speed (X_5). The responses (dependent variables) investigated were agglomerate ash content (Y_1), agglomerate yield (Y_2), recoverable oil--the oil retained in the agglomerates (Y_3), performance value--the general agglomerate quality (Y_4), ash efficiency index (Y_5), and ash reduction (Y_6). The numbers in parentheses are the identifier (variable name) for each respective independent or dependent parameter.

The data from the test matrix are given in Table 10. It should be noted that the X's were coded to be between -2 and +2 in order to facilitate the regression analysis performed. The actual values of the X's for each coded value (-2, -1, 0, +1, +2) are given in Table 11.

TABLE 10

CODED INDEPENDENT VARIABLES AND RESPONSES

Matrix Test No.	Y_1	Y_2	Y_2*	Y_3	Y_4	Y_5	Y_6
12	1.21	82.65	82.65	57.94	2	5820	82.3
19	2.12	22.94	22.94	42.34	1	1030	79.5
17	1.37	96.67	96.67	47.64	2	5700	80.0
8	2.20	48.92	48.92	46.78	3	1990	78.7
22	1.12	100.00	99.80	44.10	2	7440	83.7
5	1.89	4.24	4.24	37.56	0	222	81.7
32	0.92	100.00	99.80	39.52	4	9450	86.6
30	2.27	3.41	3.41	27.34	0	148	78.0
27	1.40	92.57	92.57	39.04	3	5370	79.6
31	2.62	69.37	69.37	38.82	3	2180	74.6
25	1.76	90.89	90.89	47.52	3	3980	74.3
3	2.64	15.71	15.71	58.90	1	570	74.4
6	2.35	100.00	99.80	39.74	5	2800	65.7
10	2.53	7.53	7.53	59.98	0	292	75.5
7	1.24	92.25	92.25	36.58	4	6190	81.9
24	3.37	0.70	0.70	17.53	0	20	67.4
16	0.97	100.00	99.80	31.54	2	8810	85.6
1	3.88	2.10	2.10	43.86	0	53	70.6
18	1.85	98.14	98.14	44.62	4	4210	78.8
23	1.45	91.30	91.30	40.90	3	5350	83.4
21	1.85	94.41	94.41	42.86	5	4090	78.8
26	1.77	97.05	97.05	40.98	4	4400	79.7
13	1.57	97.70	97.70	42.16	3	5130	82.0
2	1.66	98.38	98.38	44.12	5	4830	81.0
29	3.02	29.95	29.95	34.14	1	888	65.5
20	1.23	88.22	88.22	43.26	4	6290	85.9
33	1.59	94.28	94.28	44.50	4	5080	81.8
4	1.79	91.60	91.60	51.24	4	4150	79.5
9	2.23	98.53	98.53	49.44	4	3300	74.5
11	1.33	99.48	99.48	49.56	4	6340	84.8
14	1.71	98.62	98.62	45.34	3	4650	80.4
15	2.80	98.49	98.49	45.64	3	2400	68.0
28	2.33	78.17	78.17	39.88	3	2660	73.4

Y_1 Agglomerate Ash
Y_2 Yield
Y_3 Free Oil
Y_4 Performance Value
Y_5 Efficiency Index
Y_6 Ash Reduction
* Normalized values of Y_2

TABLE 11

CODING USED FOR INDEPENDENT VARIABLES

	Values of Coded Factors					
X_i = Coded Factor i	-2	-1	0	1	2	Units
i=1) Coal Mesh Size	-30x60	60x100	100x200	200x325	-325	mesh
i=2) Acid Mix Time	10	22.5	35	47.5	60	min
i=3) Acid Mix Speed	4	6	9	13.5	20.25	1000 rpm
i=4) Oil Mix Time	2	6.5	11	15.5	20	min
i=5) Oil Mix Speed	0.1	0.2	0.4	0.8	1.6	1000 rpm

The matrix of runs performed was a central composite design for five independent variables, and it required 32 runs (3). The 33 runs actually performed included the matrix and one repeat run (Run 33). This matrix allowed the fitting of a full quadratic equation to describe the impact of the factors (X's) on each respective response. The quadratic equation for each response can be represented as:

$$
\begin{aligned}
F_i = {} & b_o + b_1X_1 + b_2X_2 + b_3X_3 + b_4X_4 + b_5X_5 \\
& + b_6X_1^2 + b_7X_2^2 + b_8X_3^2 + b_9X_4^2 + b_{10}X_5^2 \\
& + b_{11}X_1X_2 + b_{12}X_1X_3 + b_{13}X_1X_4 + b_{14}X_1X_5 + b_{15}X_2X_3 \\
& + b_{16}X_2X_4 + b_{17}X_2X_5 + b_{18}X_3X_4 + b_{19}X_3X_5 + b_{20}X_4X_5
\end{aligned}
$$

In fitting this equation to the responses, any terms that were not statistically significant (at the 5% significance level) were deleted, so that, in all cases, a much smaller equation was finally obtained. The reduced equations are given in Table 12. Two summary statistics are also given in Table 3: a) the overall significance of the equation (which was always very high) and b) the amount of variation in the data explained by linear regression (called R^2). The R^2 varied from 0.26 for the percent ash reduction (Y_6), indicating a large amount of scatter around the equation, to a high of 0.87 for the yield (Y_2), indicating a good level of agreement between the data and the model.

The regression equations presented in Table 12 were used to draw conclusions about optimum operating conditions. To facilitate this data interpretation, the equation models were graphed for the predicted values, along with the actual data, shown in Figures 5-9. All the responses are depicted, except for Y_4, the performance value, which had lack of fit.

TABLE 12

STATISTICAL REGRESSION SUMMARY

Response	Response Quadratic Equation	Overall Significance	R^2
Agglomerate Ash	$Y_1 = + 1.94 + 0.59X_1 + 21X_4$	99.99%	0.60
Yield*	$Y_2^T = - 3.1 + 2.8X_1 + 0.8X_1^2 - 0.5X_5 + 0.7X_5^2 + 1.4X_1X_3 + 0.7X_2X_4$	99.99%	0.87
Free Oil	$Y_3 = + 42.9 - 1.9X_2 - 3.3X_3 + 3.0X_1X_4 - 4.1X_1X_5 - 5.2X_2X_3 - 4.5X_4X_5$	99.99%	0.67
Performance Value#	$Y_4 = + 3.53 - 0.88X_1 - 0.8X_1^2 + 0.38X_4 + 0.60X_5 - 0.34X_5^2 - 0.80X_1X_3 - 0.44X_2X_4$	99.99%	0.84
Efficiency Index	$Y_5 = + 3810 - 2410X_1$	99.99%	0.67
Ash Reduction	$Y_6 = + 78.15 - 2.27X_1 - 2.46X_4$	98.64%	0.26

* $Y_2 = 100\%/[1 + \exp(Y_2^T)]$
\# Equation shows lack of fit at 4% significance level

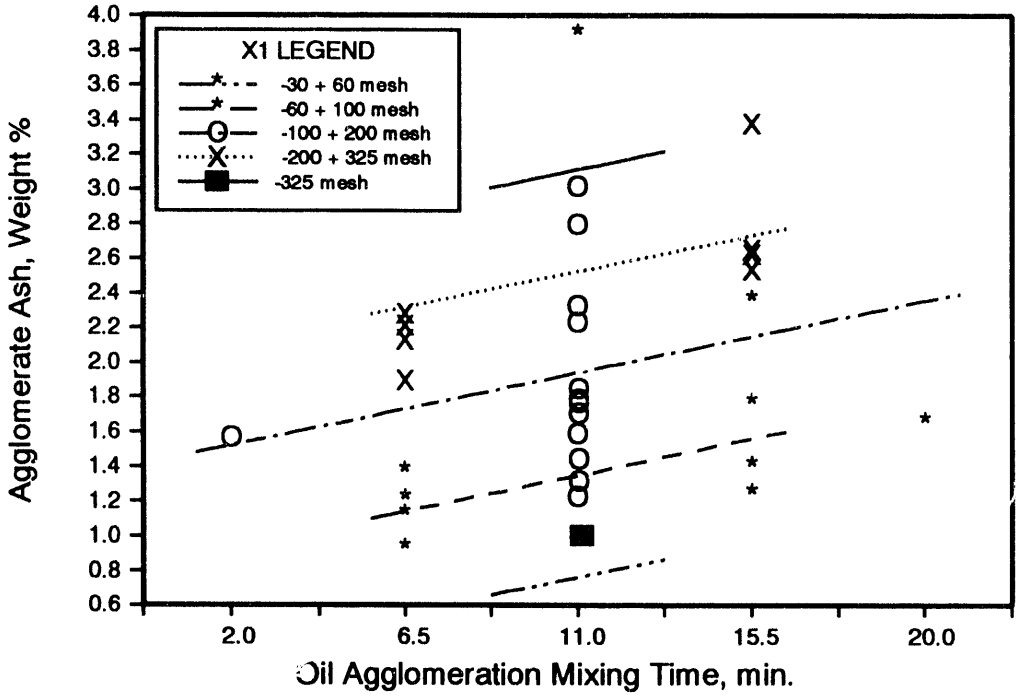

Figure 5. Ash content vs. oil mix time and particle-size fraction.

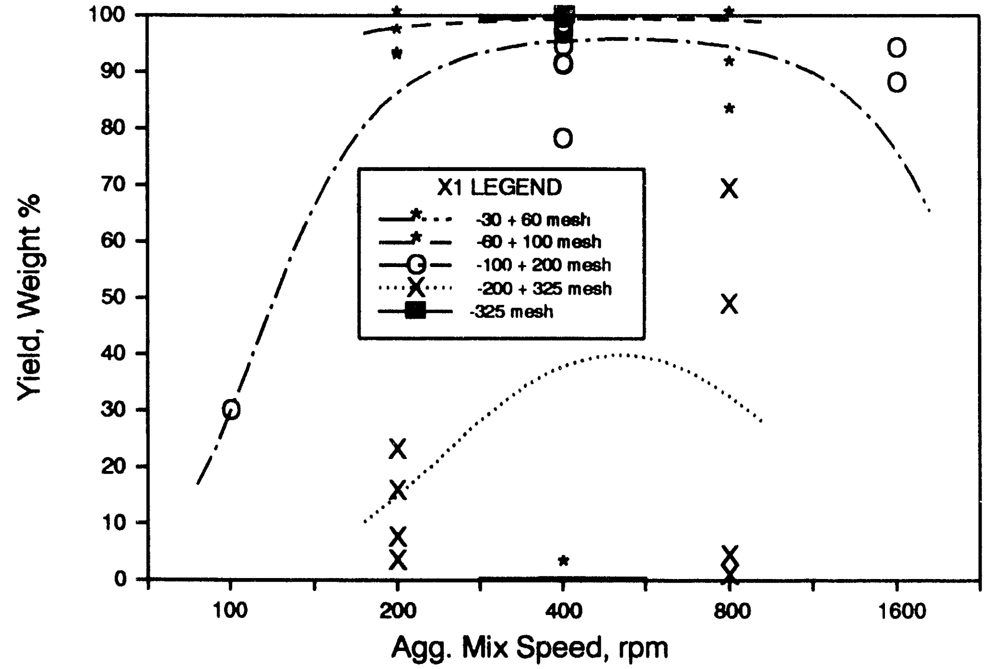

Figure 6. Agglomerate yield vs. agglomerate mix speed and particle-size fraction.

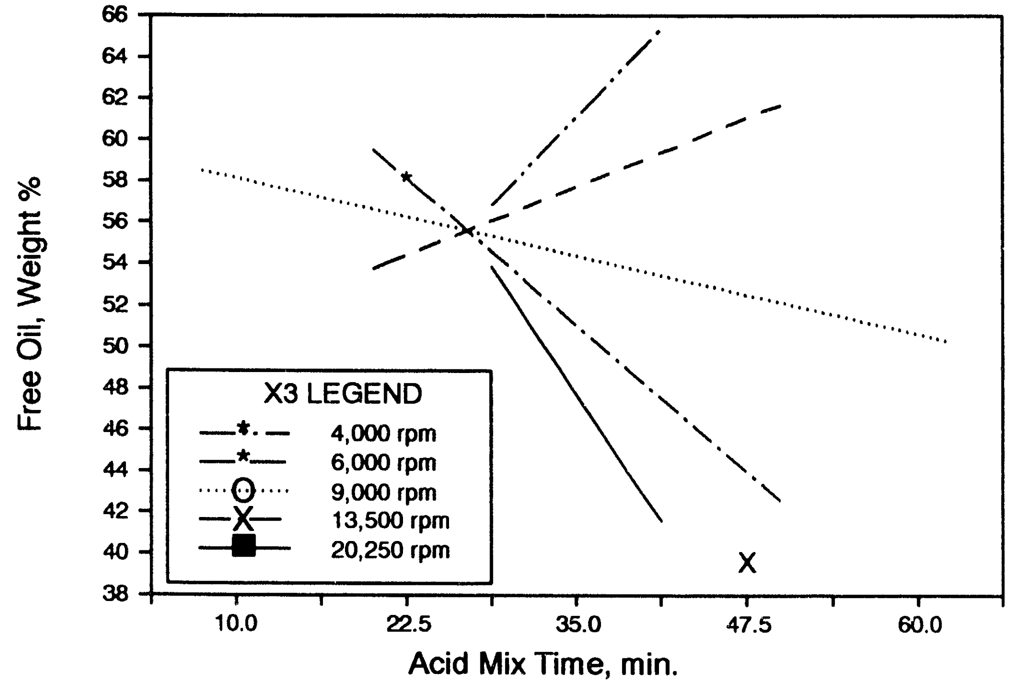

Figure 7. Free recoverable oil vs. acid mix time and acid mix speed.

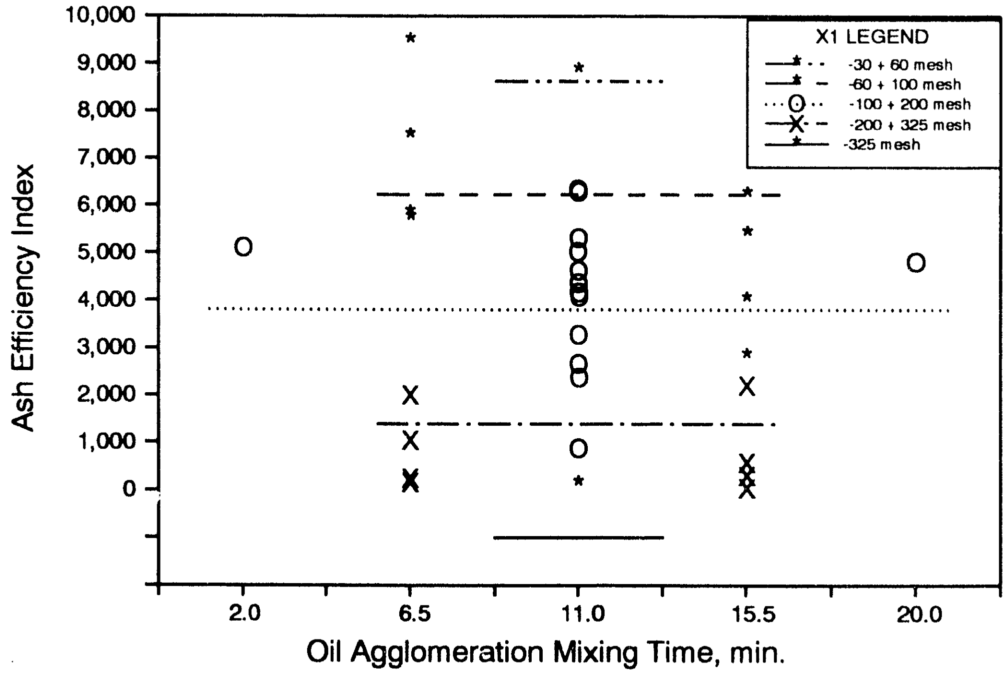

Figure 8. Ash efficiency index vs. oil mix time and particle-size fraction.

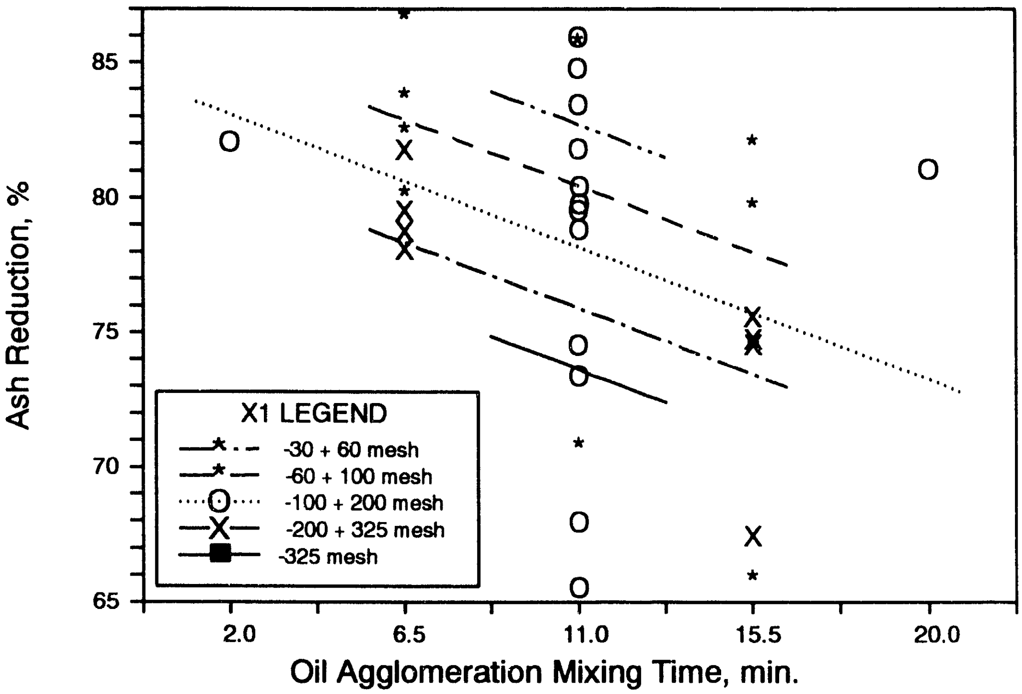

Figure 9. Ash reduction vs. oil mix time and particle-size fraction.

All responses were best at the lowest level of X_1 (-30 x 60 mesh). In addition, a low level of X_4 (oil mix time = 2 minutes) was best for Y_1 (agglomerate ash content) and Y_6 (ash reduction), while it did not adversely affect the other responses. With these two factors set, the best values for X_2, X_3, and X_5 are found via examination of responses Y_2, Y_3, and Y_4. The high level for X_3 (acid mix speed) was best based mainly on its effect on improved yields (Y_2), but it also accounted for higher free recoverable oil (Y_3) and performance values (Y_4). Mid to high values of X_5 (oil mix speed) were found to be best for all responses with too high or too low an oil mix speed causing lower agglomerate yields, etc. The only factor giving mixed results was X_2 (acid mix time), which gave the best yields and performance values at high mix times, but the most recoverable free oil at low mix times. The impact on the recoverable oil was the greatest; therefore, low acid mix times were judged the best condition according to the statistical results.

3.1.12 Expanded Test Matrix

The initial oil agglomeration matrix studies investigated the mechanics of the process. It was determined that another matrix should be performed to assess more factors important to the oil agglomeration process. The factors investigated for this matrix dealt mainly with the chemistry of the process using a single type oil.

The test matrix developed for these studies used a 3-factor Box-Behnken design (11). The three factors (independent variables) were acid strength (X_1), oil concentration (X_2), and oil mixing time (X_3). Table 13 shows the actual levels tested for the matrix. The responses (dependent variables) investigated were agglomerate ash content (Y_1), agglomerate yield (Y_2), ash reduction (Y_3), ash efficiency index (Y_4), recoverable oil (Y_5), and performance value (Y_6). The numbers in parentheses are the identifier (variable name) for each respective independent or dependent parameter. Table 14 shows the actual data from the 15 experiments used for statistical modeling.

The recoverable oil is a calculated value determined from TGA analysis. It is the fraction of the oil that can be easily recovered by distilling the agglomerates at 110°C. Oil recovery from the agglomerates may be possible at higher temperatures to recover more oil without affecting the agglomerate structural integrity. Other studies have shown successful oil recovery to 180°C, achieving a final oil content in the agglomerate of less than 5 wt% (13). The 110°C number is conveniently determined by TGA and Karl Fischer moisture data without performing an actual oil recovery test.

This matrix allowed the fitting of a full quadratic equation for each response as a function of the independent variables. The quadratic equation for each response can be represented as:

$$F_i = b_o + b_1X_1 + b_2X_2 + b_3X_3 + b_4X_1^2 + b_5X_2^2 + b_6X_3^2$$
$$+ b_7X_1X_2 + b_8X_1X_3 + b_9X_2X_3$$

TABLE 13

CODING USED FOR INDEPENDENT VARIABLES

	Values of Coded Factors			
X_i = Coded Factor i	-1	0	1	Units
i=1) Acid Strength	0.75	1.50	3.00	wt%
i=2) Oil Concentration	30	40	50	ml
i=3) Oil Mixing Time	2.5	5.0	10.0	minutes

TABLE 14

CODED INDEPENDENT VARIABLES AND RESPONSES

RUN	X_1	X_2	X_3	Y_1	Y_2	Y_3	Y_4	Y_5	Y_6
1	-1	-1	0	4.46	11.23	46.8	240	6.24	1
2	1	-1	0	2.71	10.67	67.6	383	11.22	1
3	-1	1	0	3.44	91.95	58.9	2580	33.64	3
4	1	1	0	2.22	96.11	73.4	4230	11.04	5
5	-1	0	-1	3.12	95.75	62.8	2980	26.88	4
6	1	0	-1	3.68	97.51	56.0	2550	11.96	3
7	-1	0	1	3.86	98.95	53.9	2460	8.36	3
8	1	0	1	1.74	81.51	79.2	4600	7.32	4
9	0	-1	-1	3.84	30.80	54.1	770	8.44	2
10	0	1	-1	2.84	91.97	66.0	3140	9.84	4
11	0	-1	1	3.25	22.36	61.2	666	7.32	2
12	0	1	1	2.61	100.00	68.8	3730	3.28	5
13	0	0	0	3.32	95.58	60.3	2780	13.92	4
14	0	0	0	3.80	100.00	54.5	2530	33.12	4
15	0	0	0	3.14	98.00	62.5	3020	11.48	4

X_1 Acid Strength
X_2 Oil Concentration
X_3 Oil Mixing Time

Y_1 Agglomerate Ash
Y_2 Yield
Y_3 Ash Reduction
Y_4 Efficiency Index
Y_5 Recoverable Oil
Y_6 Performance Value

This equation was fit for any terms statistically significant at the 5% significance level or greater. This resulted in the response equations shown in Table 15. Two summary statistics are also shown: a) the overall significance of the equation, and b) the amount of variation in the data explained by linear regression, R^2.

The regression equations presented in Table 15 were the statistical models for the experimental data and were used to draw conclusions about optimum agglomeration operating conditions. To facilitate the data interpretation, the equation models were graphed for the predicted values, along with the actual data, as shown in Figures 10-13. The graphical representations were presented in an effort to show the most significant relationships between the three factors and the responses.

All the responses were best at the highest oil concentration level of X_3 (50 ml). This was not unexpected since the 50 ml oil concentration proved to be near optimum in past screening experiments, but no statistical measurement of the significance of using less oil was made at the time of these earlier tests. In addition, increased acid strength and agglomeration mixing time resulted in lower ash content. This is well-illustrated in Figure 10 as ash content, as a function of all three factors. Ash reduction produced a very similar model to ash content as the same terms for all three factors appeared, as shown in Table 15 for the ash content and ash reduction.

For the model equation developed for the agglomerate yield, only one factor was significant, the oil concentration. Figure 11 shows the line for the model equation generated. The low R^2 value of 0.73 indicates a large amount of scatter around the data, which can be seen in Figure 11. It was a significant finding that the acid concentration was not significant to the yield. Therefore, merely the product quality of the agglomerate will be affected by lower acid concentration and not the formation of the agglomerates. The recoverable oil response had no factors appearing statistically significant for the matrix data and this range of variables.

The efficiency index, which is a parameter combining ash reduction with yield, had the highest number of significant terms (13). Large values of all factors were generally desired for a high efficiency index, as shown in Figure 12. At the highest level of acid strength (3 wt%) and all levels of mixing time, the efficiency index peaks at the highest level of oil concentration (50 ml). At the lower levels of acid strength (0.75 and 1.5 wt%), the efficiency index peaks at oil concentrations of 45 to 47 ml.

The performance value, which is a qualitative assessment of the appearance and integrity of the agglomerates, followed nearly the same trend as the ash efficiency index. The lines for the model equations and the actual data are shown in Figure 13. The degree of fit of the actual data around the model equations was quite high, as indicted by the high R^2 value of 0.95 for both the efficiency index and the performance value. At the highest level of acid strength (3 wt%) and all levels of mixing time, the performance value was starting to peak at the highest level of oil concentration (50 ml). Increasing the oil concentration would not increase the performance value because this dependent variable is a qualitative number with a maximum value of 5. At the lower levels of acid strength (0.75 and 1.5 wt%), the performance value peaks at oil concentrations of 44 to 47 ml.

TABLE 15

STATISTICAL REGRESSION SUMMARY

Response	Response Quadratic Equation	Overall Significance	R^2
Agglomerate Ash	$Y_1 = +3.20 - 0.57X_1 - 0.39X_2 - 0.25X_3 - 0.67X_1X_3$	99.99%	0.88
Yield*	$Y_2^T = -3.71 - 2.57X_2 + 2.71X_2^2$	99.99%	0.73
Ash Reduction	$Y_3 = +61.8 + 6.7X_1 + 4.7X_2 + 3.0X_3 + 8.0X_1X_3$	99.99%	0.88
Efficiency Index	$Y_4 = +2990 + 440X_1 + 1450X_2 + 250X_3 - 1020X_2^2 + 380X_1X_2 + 640X_1X_3$	99.99%	0.95
Performance Value	$Y_6 = +4.08 + 0.25X_1 + 1.38X_2 - 0.63X_1^2 - 0.88X_2^2 + 0.50X_1X_2 + 0.50X_1X_3$	99.99%	0.95

* $Y_2 = 100\%/[1 + \exp(Y_2^T)]$

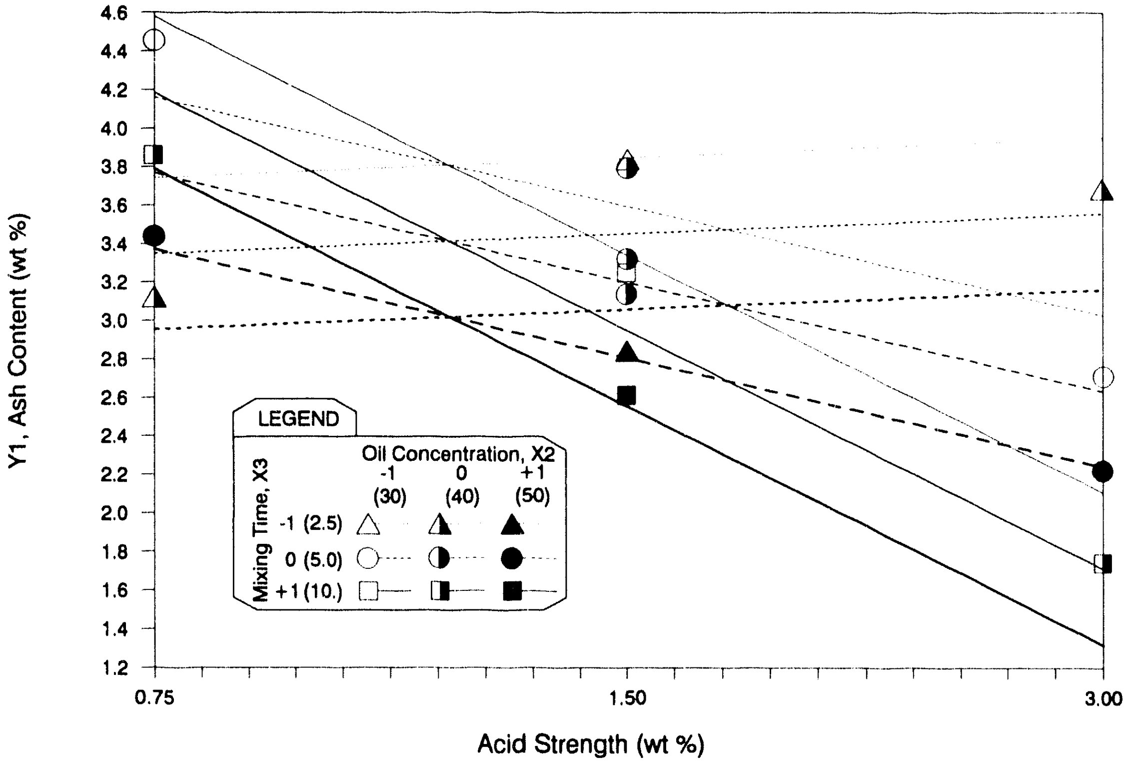

Figure 10. Agglomerate ash content vs. acid strength and oil mix time.

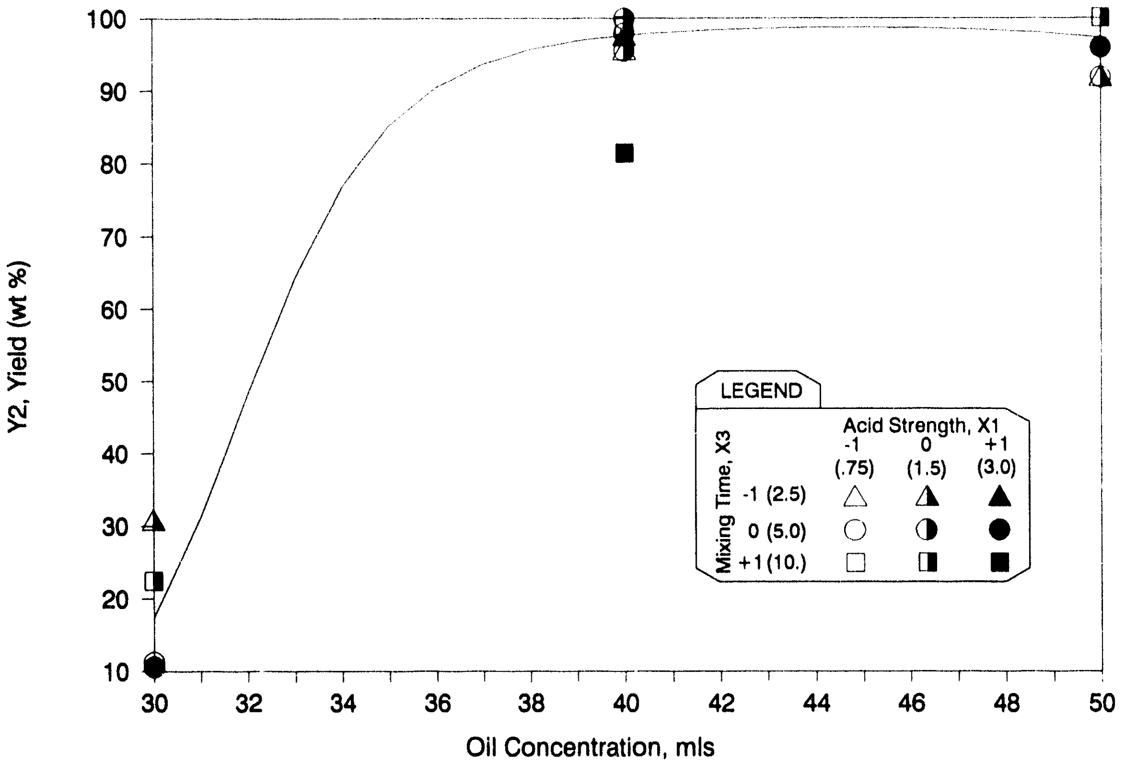

Figure 11. Agglomerate yield vs. oil concentration.

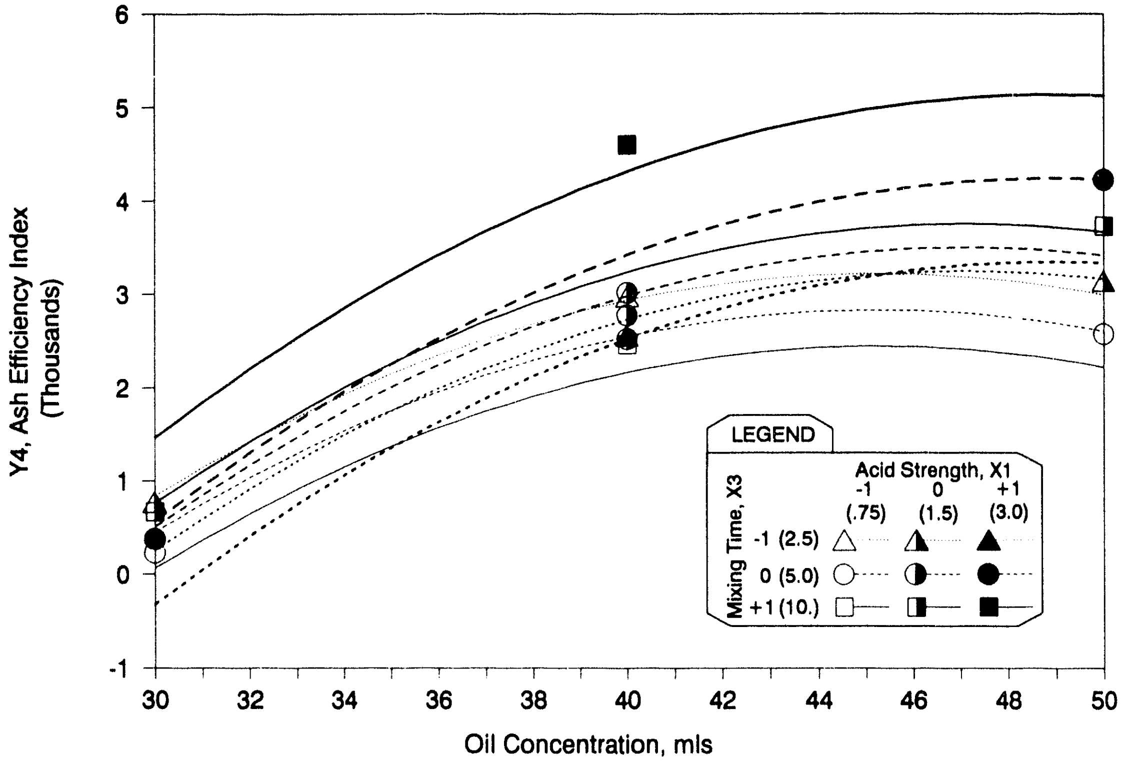

Figure 12. Ash efficiency index vs. acid strength, oil concentration, and oil mix time.

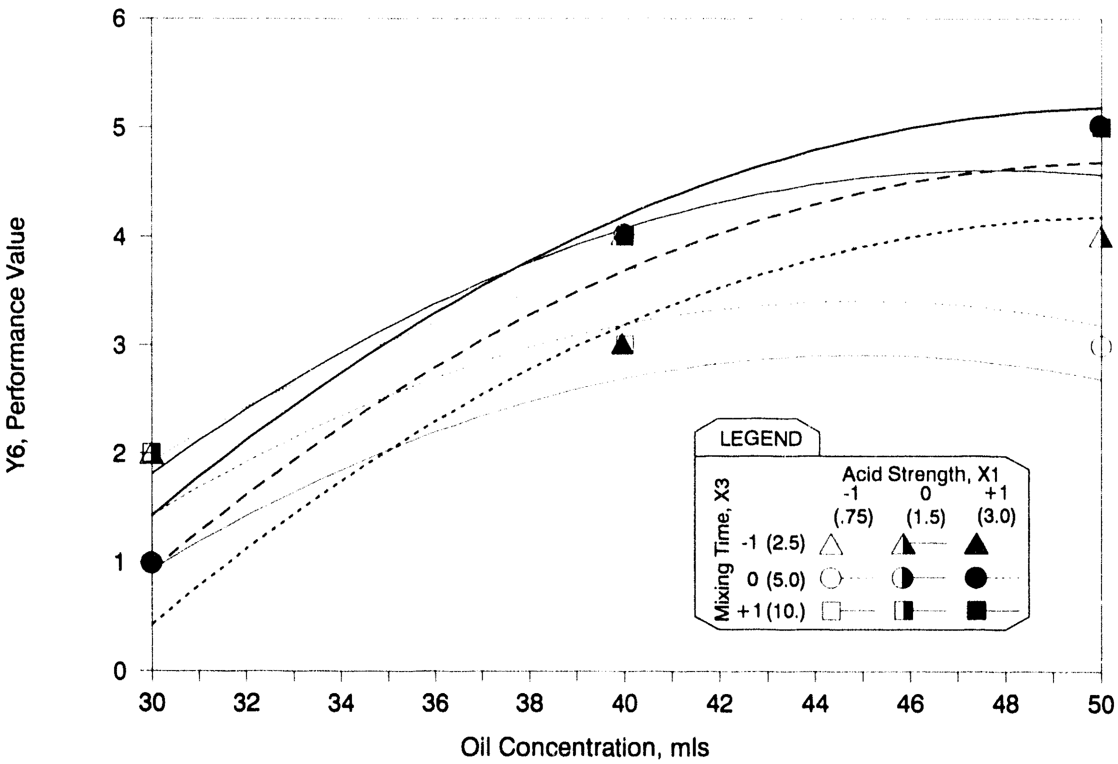

Figure 13. Performance value vs. acid strength and oil concentration.

3.2 Partial Oxidation Hot-Water Drying

3.2.1 Objectives

The purpose of the testing was to determine if partial oxidation during hot-water drying would a) enhance the cleanability of the coal, b) improve the rheology of CWFs made from LRC, and c) improve the economics of hot-water drying by direct particle heating.

3.2.2 Experimental Procedure

Partial oxidation hot-water drying was performed in the EERC 7.6-liter externally heated, stirred autoclave. The autoclave was equipped with a dip tube in order to sparge oxygen through the agitated slurry. A metal frit was fastened to the bottom of the dip tube to facilitate oxygen dispersion within the slurry.

A 3.8-liter autoclave, filled from a cylinder tank, was used as an accumulator for oxygen charging. The volume of the accumulator was accurately determined by measuring the mass of water needed to fill the accumulator. The pound-moles of oxygen in the accumulator, as a function of pressure and temperature, were determined from Van der Waal's equation (14).

Approximately 2000 to 4000 grams of a 50% Beulah/50% water slurry was used in each test. After charging the slurry, the autoclave was evacuated of residual air, and the heaters were turned on. Heat-up to 200°C took approximately 1.5 hours, whereupon the contents of the autoclave were allowed to stabilize at 200°C.

Prior to charging oxygen to the 7.6-liter reactor, the pressure and temperature in the accumulator were recorded. The oxygen was then metered from the accumulator into the 7.6-liter autoclave until the desired slurry temperature was achieved. The final oxygen pressure and temperature in the accumulator were then recorded.

After completing the test, the autoclave and contents were allowed to cool overnight. The product gas was metered and vented, and the product slurry was recovered and weighed. Product gas sampling was performed for two tests. The gas was not analyzed, however, to determine composition. The process water was separated from the solids by filtering. The filter cake was washed with deionized water to help remove any cations loosely adhered to the coal surface.

The filter cake from each test was diluted with deionized water to produce slurry for rheological testing. Apparent viscosity was determined for the slurry at three different solid contents. Washability testing was performed on the raw and hot-water dried coals.

3.2.3 Results

Combustion of the slurry was instantaneous upon addition of the oxygen, and the temperature rise was rapid. Temperature control, however, in the batch autoclave system was limited by an unsuitable oxygen charge system. The maximum charge pressure for the accumulator was 2000 psig, as set by the pressure transducer. As a consequence, the tests usually required multiple charges of oxygen to approach the desired temperature. The temperature of the slurry in the 7.6-liter autoclave usually decreased before the accumulator could be recharged with oxygen. The temperature drop was rapid because the autoclave was absorbing heat from the slurry. Additionally, as the steam pressure in the 7.6-liter autoclave increased and as combustion gases were produced, subsequent charges delivered less oxygen, producing a progressively lower temperature increase. This shortcoming was somewhat alleviated by decreasing the mass of slurry in the last PO-HWD test.

The conditions for the three PO-HWD tests and one HWD test are presented in Table 16. The oxygen requirements, in pound-moles of oxygen charged per gram of slurry per Celsius degree temperature rise, are presented in Table 17. The oxygen utilization, as a percentage of the original oxygen charge, was not determined because product gas analysis was not performed. The solids recovery per test is presented in Table 17.

TABLE 16

CONDITIONS FOR HWD AND PO-HWD TESTS

Test #	Test Type	Temperature (°C)	Slurry Charge (grams)	Oxygen (lb-moles)
1	HWD	303	3984	NA
2	PO-HWD	239	3988	0.0195
3	PO-HWD	282	3989	0.0190
4	PO-HWD	303	1991	0.0227

HWD Hot-Water Dried
PO Partial Oxidation
NA Not Applicable

TABLE 17

OXYGEN REQUIREMENTS AND SOLIDS RECOVERY

Test #	Temperature Rise (dC°)	Oxygen (lb-mole/g-dC°)	Solids Recovery (wt%)
1	NA	NA	90.8
2	36	1.36 x 10-7	93.4
3	75	6.35 x 10-8	91.6
4	96	1.19 x 10-7	80.5

NA Not Applicable

Preliminary calculations indicated that the lb-moles of O_2 necessary to raise one gram of slurry one degree Celsius via partial oxidation varies by only about 2% over the range of 300°C to 320°C. Similarly, over the range of 282°C to 303°C, the oxygen requirements should be nearly equal. However, the measured oxygen requirements varied by nearly 100% over the range of 282°C to 303°C. The large discrepancy may indicate overcharging of oxygen in order to achieve temperature.

The solids recovery for the HWD test at 303°C was approximately 91 wt%, while the solids recovery for the PO-HWD test at 303°C is approximately 81 wt%. The loss of solids in the HWD test is attributed to decarboxylation and mild pyrolysis reactions. The additional loss of solids in the PO-HWD test is presumably due to combustion reactions.

The large difference in solids recovery between the 282°C and 303°C PO-HWD tests also seems to substantiate the overcharging of oxygen in order to achieve 303°C.

The apparent viscosity at 100 sec^{-1} versus solids content is presented in Figure 14 for slurries produced from the raw, HWD and PO-HWD Beulah coal. The slurry with the highest solids content was achieved with the 303°C HWD coal. Partial oxidation hot-water drying at 303°C, however, did not produce a slurry with comparable solids content and viscosity. The variation may be due to insufficient residence time at temperature and consumption of combustible material during PO-HWD. The PO-HWD did, however, improve the slurry solids content above that of a raw Beulah slurry.

3.2.4 Washability Testing

Certigrav gravity fractionation, or washability, was performed on PO-HWD and normal HWD Beulah lignite in order to determine if partial oxidation enhances the cleanability of this coal. In addition, the raw coal was subjected to fractionation. All samples were air-dried prior to the separations.

Figure 15 is representative of specific gravity versus yield for the products. The graph indicates that at the lowest specific gravity, a yield of approximately 70 wt% can be obtained with the normal HWD product. With the PO-HWD products, the yields at 1.4 specific gravity (sp. gr.) are all less than 40 wt%. The yields for the products don't become similar until 1.6 sp. gr. is attained.

Figure 16 indicates what happens to the ash reduction during fractionation. The 300°C PO-HWD product has the highest ash reduction at 1.4 sp. gr. This is due to the pores on the coal not being coated with tars, which form during normal HWD, that inhibit the separation of the ash-forming components. This is probably why all the PO-HWD products and the raw coal show a better ash separation all the way to 1.5 sp. gr. As the graph indicates, at 1.4 sp. gr. the product HWD at 300°C doesn't indicate as large of an ash reduction, due to the formation of tars during HWD.

Another factor used to determine the washability performance of a coal was the heating value of the product at each fraction. Figure 17 is a graph of the specific gravity of the products versus the moisture-free heating value for all samples. The graph shows that the heating values increase slightly as the specific gravity decreases. The graph also indicates a substantial difference in the heating value for the HWD product compared to the PO-HWD products. The PO-HWD products have a heating value very similar to the raw coal, whereas the normal HWD product has a heating value approximately 1000 Btu/lb higher. Energy recovery was determined for all the fractions, and these results indicated a graph identical to that depicted in Figure 15.

To a certain extent it appears that partial oxidation can enhance the cleanability of the coal in that at lower specific gravities more ash liberation occurs during partial oxidation than during normal hot-water drying. During the hot-water drying process the tars that are formed coat the

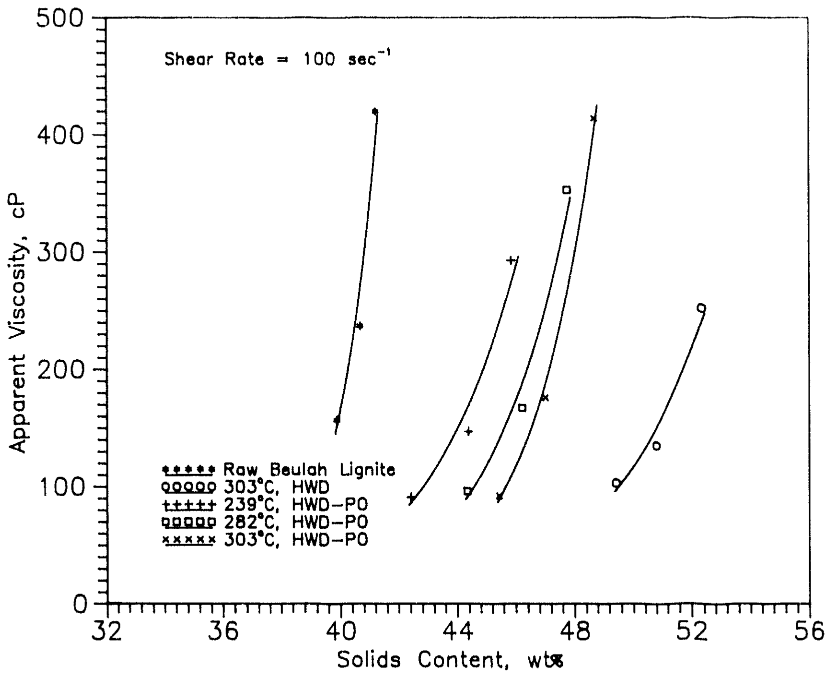

Figure 14. Apparent viscosity vs. solids content for raw, HWD, and PO-HWD Beulah lignite.

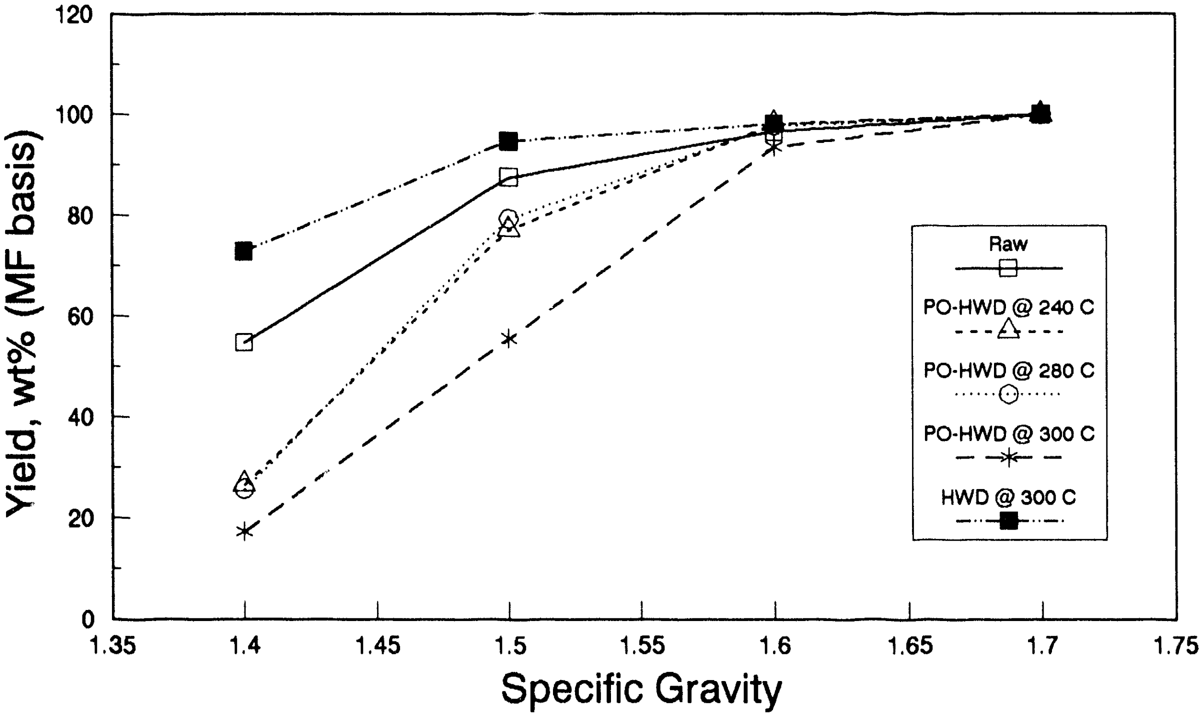

Figure 15. Specific gravity vs. yield for raw, HWD, and PO-HWD washability products from Beulah lignite.

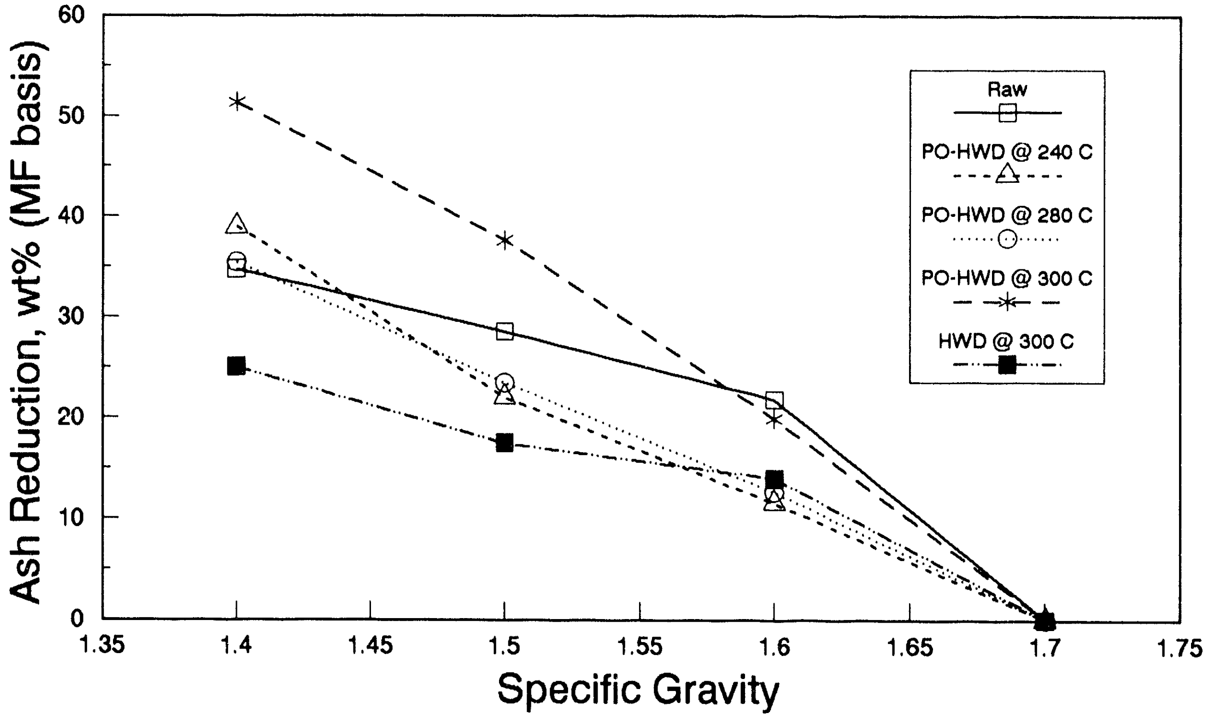

Figure 16. Specific gravity vs. ash reduction for raw, HWD, and PO-HWD washability products from Beulah lignite.

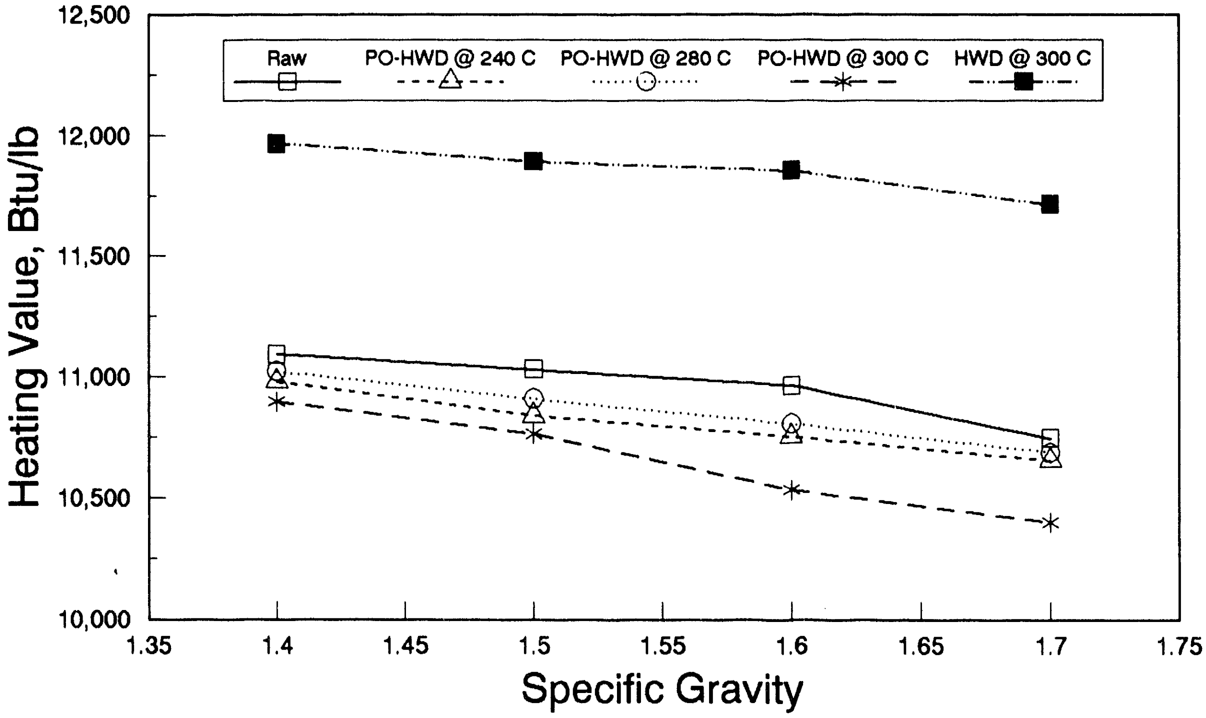

Figure 17. Specific gravity vs. heating value for raw, HWD, and PO-HWD washability products from Beulah lignite.

surface inhibiting ash separation, whereas during partial oxidation it appears that the tars are not formed and the ash is more easily liberated. Product yield and heating values are much lower at the lower specific gravities with partial oxidation products than with HWD products due to decarboxylation that occurs in the HWD process.

3.2.5 Economic Benefits

It was stated in a previous report that an electrical cost savings would be realized by supplying a portion of the thermal requirements of HWD by direct (internal) heating (15). However, for a commercial-scale HWD plant located at the mine mouth the electrical cost savings may be small or nonexistent. This is because the thermal requirements for heating Dowtherm™ fluid would probably be more economically achieved using raw coal or reject coal from the wash plant as a combustion fuel in a fired Dowtherm™ heater. The CWF produced at the plant could also be used as fuel for heat generation.

A simplified configuration for HWD slurry preheat is shown in Figure 18. The plant would use feed slurry/product slurry heat exchange to maximize system thermal efficiency and to reduce the thermal requirements on the Dowtherm™ preheat system. The majority (75 to 85%) of the feed slurry preheat will occur during the indirect heat exchange with the product slurry. The remaining heat to bring the slurry up to reaction temperature would be supplied by the Dowtherm™ preheat system. After preheating, the slurry would be maintained at reaction temperature for the desired residence time in an electrically heated downflow pipe reactor. The thermal (electrical) requirements for this reactor would be very small compared to the overall system thermal requirements.

The configuration in which the Dowtherm™ preheat system is replaced by a partial oxidation reactor is shown in Figure 19. In this scenario, the slurry is internally heated by combustion of the coal in the slurry. The oxidant would probably be air or perhaps oxygen, depending upon the size of the plant. The economies of scale may be favorable for a large plant to produce pure oxygen for internal combustion using an air fractionating unit.

Fuel costs were approximated for a HWD plant producing 1 million ton per year of CWF. The plant would be located mine mouth at a Powder River Basin mine site. The heating value of the coal is assumed to be 8100 Btu per pound and the plant would operate for 7824 hours per year (16). The thermal requirements for heating a slurry of 37 wt% solids from 260 to 300°C would be approximately 2.0×10^{11} Btu per year. The electrical cost for a Dowtherm™ preheat system operating at an 85% electrical to thermal efficiency would be $\$4.8 \times 10^6$ per year at $0.07 per kWh. The fuel cost for a Dowtherm™ preheat system fueled by raw coal and operating at a 35% thermal efficiency would be approximately $\$2.1 \times 10^5$ per year. The fuel costs would even be less if the preheat system was fueled by reject coal from the mine wash plant. The fuel cost for the partial oxidation scenario was assumed to be the value of the coal in the slurry which was internally combusted to provide direct heat. The cost of the coal was determined to be $\$7.4 \times 10^4$ per year assuming negligible heat losses from the partial oxidation reactor. The calculations for the above cost approximation are presented in Appendix A.

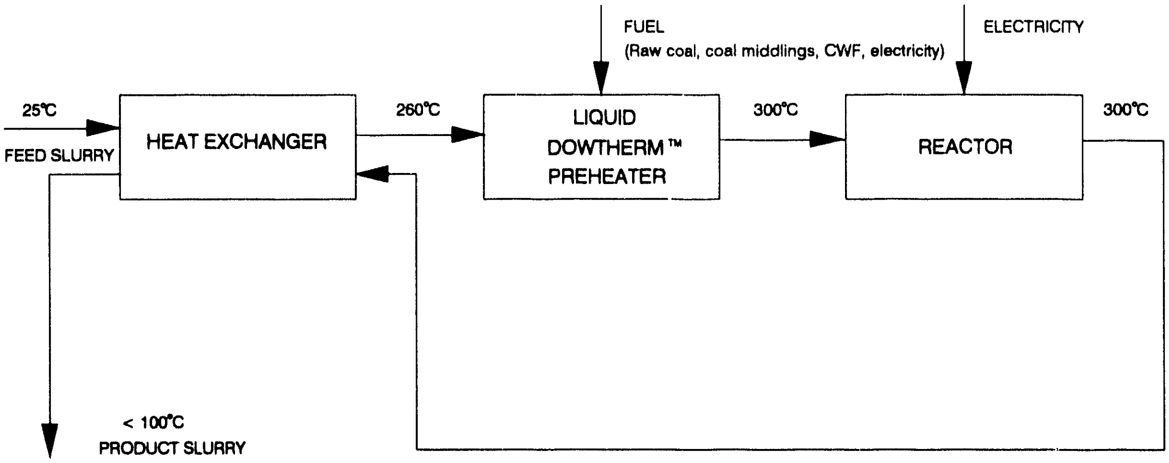

Figure 18. HWD scenarios for slurry preheat.

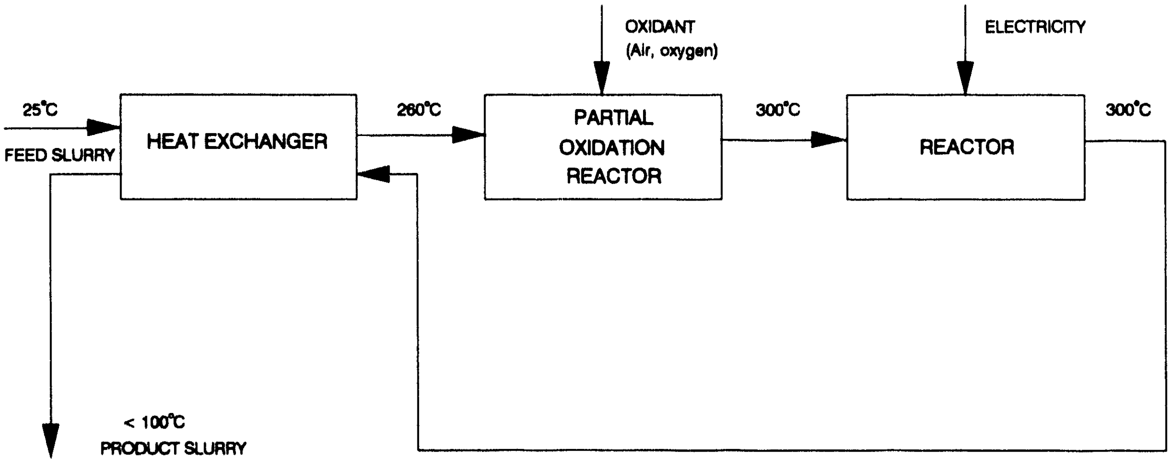

Figure 19. PO-HWD scenarios for slurry preheat.

Based on the above calculations of fuel costs, partial oxidation could replace the Dowtherm™ preheat system. However, the actual operating costs of each scenario can not be determined until all the pieces of equipment have been determine and sized. Additional Dowtherm™ preheat system operating costs would include electrical costs for pump and fan motors and chemical costs for makeup heat transfer fluid. Partial oxidation system operating costs would include electrical costs for compressor motors for supplying air, and, in the case of a large plant, operating costs for the air fractionation unit.

4.0 CONCLUSIONS

1. The use of a polar, coal-derived binding oil for oil agglomeration with LRCs produced excellent ash reductions and coal recoveries.

2. TGA analysis has proved to be a reliable method for determining proximate analyses compared to ASTM results, although the ash contents determined for agglomerates appear to be higher on a moisture-binding oil-free basis compared to the moisture-free basis for the ASTM method.

3. Washability studies on two size fractions of Beulah lignite, comparing air-dried versus as-received results, indicated no significant differences in performance.

4. Acid cleaning-oil agglomeration was similar in cleaning performance to washability-acid cleaning. The agglomeration process had higher recoveries but slightly higher ash contents, although the agglomeration process was not optimized.

5. Although the -325 mesh x 0 Beulah lignite fraction was higher in ash and smaller in size than the parent -30 mesh sample, it was agglomerated successfully with over 90% recovery and 72% ash reduction.

6. XRFA of ash elements in the agglomerated products showed that aluminosilicate compounds were concentrated by the removal of acid-soluble cations. This has the potential to significantly reduce the fouling tendency of LRCs during utilization in combustion or conversion systems.

7. Based on previous acid leaching studies with Beulah, the residual chloride was 0.3 wt% in the agglomerates, and consequently, it was necessary to change from hydrochloric acid to nitric acid for the agglomeration matrix.

8. Karl Fischer moisture determinations on agglomerates and fines have shown the water content to be significantly less than that suggested by ASTM results.

9. Agglomeration yields for the matrix indicate the process was successful using Beulah lignite, and the majority of the agglomerate yields were greater than 70 wt%.

10. The results indicate that finer particle sizes were less successful in agglomeration or ash reduction than were larger particle sizes. The lower ash reductions in the finer particle sizes were due to the fact that the parent coal was fractionated rather than pulverized to the smaller sizes. As a consequence, the fractions did not have identical initial ash contents.

11. Ash efficiency indexes of greater than 6,000 were attained for a few of the agglomerates and were representative of excellent agglomeration performance.

12. The autoclave testing has proved that heat for hot-water drying of Beulah coal slurry can be supplied internally by partial oxidation (combustion) of the coal.

13. Combustion of a 50/50 Beulah coal/water slurry is instantaneous at temperatures as low as 200°C.

14. The batch processing method used was not reliable for temperature control and oxygen charging for partial oxidation testing.

5.0 RECOMMENDATIONS

1. Efforts to determine oil recovery and recycle are needed to produce a low oil content agglomerate product and to test the agglomerating properties of recycled oil.

2. Further analyses to determine the agglomerate chloride or nitrate anion concentrations are needed to assess possible fuel utilization problems in liquefaction or combustion (i.e., chloride stress corrosion cracking or NO_x emissions).

3. Calorific value information on the agglomerates is needed for energy recovery data and to assess the contribution of oil and coal heating values.

4. Oil amounts and recovery have to be investigated further for Beulah lignite because some agglomerate products were a large clump instead of small agglomerates, indicating oil amounts were excessive.

5 Depending on the structural integrity of agglomerates after the bridging liquid phenol is recovered, binding agents to harden the product agglomerates may be needed.

6. Future partial oxidation work, to accurately determine oxygen utilization and temperature control, should be performed in a continuous unit such as the EERC 10 lb/hr CPU or 600 lb/hr PDU.

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APPENDIX A

FUEL COSTS FOR 1 MILLION TON PER YEAR CWF PLANT

Fuel Cost Criteria

Production:	1 million tons per year slurry fuel
Source Coal:	Powder River Basin
Rank	Subbituminous C
Higher Heating Value	8100 Btu/lb
Process Conditions:	
Slurry Feed Rate	4.11×10^5 lb/hr
Slurry Solids Content	37.0 wt%
Feed Slurry Properties:	
Average Specific Heat	0.85 Btu/lb-°F
Dowtherm™ Efficiency:	
Electrical Heater	1 Btu electrical/0.85 Btu thermal (85%)
Coal Fired Heater	1 Btu HHV/0.35 Btu thermal (35%)
Partial Oxidation Efficiency:	1 Btu HHV/1 Btu thermal (100%)

Calculations

Slurry Heat Requirements:

$$Q = 411000 \text{ lb/hr} \times 0.85 \text{ Btu/lb-°F} \times (300-260)\text{°C} \times 1.8 \text{ °F/°C} \times 7824 \text{ hr/yr}$$

$$Q = 2.0 \times 10^{11} \text{ Btu/yr (thermal)}$$

Fuel Cost for Electrically Heated Dowtherm™ System

$$\text{Cost} = 2.0 \times 10^{11} \text{ Btu/yr} \times 1/0.85 \times 2.93 \times 10^{-4} \text{ kWh/Btu} \times \$0.07/\text{kWh}$$

$$\text{Cost} = \$4.8 \times 10^{6}/\text{yr}$$

Fuel Cost for Coal Fired Dowtherm™ System

$$\text{Cost} = 2.0 \times 10^{11} \text{ Btu/yr} \times 1/0.35 \times 1 \text{ lb}/8100 \text{ Btu} \times \$6.0/2000 \text{ lb}$$

$$\text{Cost} = \$2.1 \times 10^{5}/\text{yr}$$

Fuel Cost for Partial Oxidation Reactor

$$\text{Cost} = 2.0 \times 10^{11} \text{ Btu/yr} \times 1/1 \times 1 \text{ lb}/8100 \text{ Btu} \times \$6.0/2000 \text{ lb}$$

$$\text{Cost} = \$7.4 \times 10^{4}/\text{yr}$$

4.3 Combustion Characterization of Low-Rank Coal Fuels

COMBUSTION CHARACTERIZATION OF LOW-RANK COAL FUELS

Annual Technical Progress Report
for the Period July 1, 1989, through June 30, 1990

Including

the Quarterly Technical Progress Report
for the Period April through June 1990

by

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September 1990

Work Performed Under Cooperative Agreement No. De-FC21-86MC10637

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COMBUSTION CHARACTERIZATION OF LOW-RANK COAL FUELS

1.0 INTRODUCTION

Coal research by the Pittsburgh Energy Technology Center (PETC) of the U.S. Department of Energy (DOE) is directed toward both increasing coal usage in existing markets and introducing new, premium-quality coal-based fuels to markets currently dependent on oil or natural gas. Accordingly, the work concentrates on coal cleaning, fuel formulation/handling, combustion, and flue gas cleanup. All of these activities are necessary technologies in the direct use of coal. The progress achieved over the past several years through PETC and related federal/private sector research activities has considerably strengthened these technologies, consequently enhancing the possibility of meeting DOE's coal research goals.

The industrial and utility sectors are major consumers of oil and gas. Many of these oil and gas systems could be converted to coal-fired systems, resulting in a significant reduction in the oil and gas needs of this country. As an example, oil- and gas-generating systems in the utility sector have a nameplate generating capacity of 156 gigawatts (GW). Based on remaining years of service, 91.6 GW have the potential for conversion to coal. This represents 4.8 quads of energy per year. There are similar opportunities to reduce the U.S. dependency on oil and gas in the industrial sector.

A number of critical factors need to be examined to determine the success of switching from oil or gas to coal. Significant differences exist in burner designs, furnace plan areas, heat release rates, tube spacing in convective passes and number/location of soot and wall blowers. Combustion characterization of replacement fuels is needed to assess the impact of the new fuel's properties on the existing design. This characterization should help assess any equipment modifications or derating necessary as a result of switching fuels.

2.0 GOALS AND OBJECTIVES

The purpose of this project is to expand the scientific and engineering data base on the combustion characteristics of advanced low-rank coal fuels for industrial and utility combustion systems currently using oil or gas as the primary fuel. To accomplish this overall objective, specific objectives are to use existing and new laboratory-, bench-, and pilot-scale techniques to characterize various dry and slurried coals and coal blends. Results of these enhanced characterization tests will be used to assess the effect of switching fuels on the ignitablitiy and stability of the flame, fouling and slagging, emissions, and carbon burnout as compared to the design fuel.

Work during the three-year period of April 1986 through June 1989 focused on the development of a small-scale combustion device suitable for the residential and commercial marketplace, followed by characterization of coals and coal/water slurries as replacement fuels for oil and gas. The direction of the project has shifted, with the new emphasis on the industrial and utility marketplace. Low-rank coals and slurries, alone or in blend, with potential to replace oil and gas will be characterized in the next three years (7/89-6/92).

The University of North Dakota Energy and Environmental Research Center (UNDEERC) has been involved in the characterization of coal for over 20 years. This work was initially developed to study fouling in the convective pass, and has expanded to look at slagging in the boiler and emissions. A number of analytical techniques and bench- and pilot-scale tests have been developed. These techniques will be used as a part of the proposed work. EERC does not currently have the capabilities to study flame stability and ignitability. A part of the three-year objectives will, therefore, be to add this capability. The capability for flame stability testing will be combined with existing techniques for studying slagging, fouling, and emissions; and new techniques being developed under other parallel DOE and EPRI programs to produce an "enhanced" combustion characterization program. This program will be used to characterize low-rank coals and slurries, alone or in blends, to determine their potential as replacements for oil and gas.

UNDEERC is a subcontractor to Combustion Engineering, Inc. for work under DOE Contract No. DE-AC22-89PC88654, which was awarded in March 1989. This contract is for the combustion characterization of beneficiated coal-based fuels; the primary focus will be on fuels derived from eastern bituminous coals. In the subject three-year Cooperative Agreement, the intention is to expand the data base on selected, advanced, low-rank coal fuels in a manner generally parallel to the contract noted above. To achieve this objective, UNDEERC will develop a cooperative program with the Coal Combustion R&D Division at PETC. UNDEERC will perform laboratory analyses and bench-scale tests (similar to the work performed for CE) that are practical and appropriate for the selected fuels. Atomization and combustion testing of these fuels will be performed at PETC.

It is not anticipated that the cooperative UNDEERC/PETC program will duplicate all aspects of the CE contract. The intention is to characterize the combustion properties of advanced LRC fuels (in both dry and slurry forms) by means broadly similar to those employed by CE and its subcontractors, within the constraints of the funding level of the Cooperative Agreement and the work priorities of the PETC staff and facilities.

Specific three-year objectives include:

1. Combustor Modifications and Development.

   Several modifications will be made to the EERC 550,000 Btu/hr test combustor to enhance its capabilities for characterizing fuels. These modifications include adding a data acquisition and control system, pressure transducers, thermocouple, control valves, and flow transmitters (this equipment is available from the residential-scale packed-bed combustion system). An adjustable swirl burner, similar to that used by PETC, will be purchased and installed. Modifications to the coal feed system are needed to allow accurate metering of the fuel, and to eliminate problems with uneven feeding.

Coal sizing equipment will be upgraded to allow more control over the size of the coal feed. An existing tubular ESP will be installed and additional viewports will be added to the combustor.

2. Characterization of Combustion Performance.

   The performance of various fuels, including coal blends, coal (both dry and slurred), and oil (for comparative purposes) will be characterized. These characterization tests will be designed to determine combustion performance, mineral matter behavior, and emissions.

   Aspects of combustion performance to be measured will include flame stability, ignitability, and carbon burnout. Mineral matter observations will include slagging on furnace walls, deposition on convective pass tubes, fly ash formulation, and mineral matter transformations. Gaseous emissions, including NO_x, SO_2, CO, and particulates, will be measured. Fly ash particle size and resistivity will be measured to help evaluate ESP performance.

3. Comparison to Commercial-Scale Demonstrations.

   Results from the enhanced characterization tests performed under this project will be compared to results (where available) of existing DOE and EPRI large-scale demonstration projects.

This work plan, originally scheduled for the three year period between 7/89 and 6/92, has been modified, due to changes in scope and the associated shifts in funding, to include only those areas to be completed during the first year. This final report outlines the accomplishments of the first year.

3.0 PROPOSED WORK FOR THE FIRST YEAR (7/89-6/90)

To achieve the specific goals for the first year, a five-task program was developed. The tasks include modifications to the 550,000 Btu/hr test furnace, system shakedown and baseline testing of a western subbituminous coal, and three characterization studies of various coals and coal blends.

3.1 Task A. Combustor Modifications

Several modifications will be made to the EERC 550,000-Btu/hr test combustor to enhance its capabilities for characterizing fuels. These modifications include adding a data acquisition and control system, pressure transducers, thermocouple, control valves, and flow transmitters (this equipment is available from the residential-scale packed-bed combustion system). An adjustable swirl burner similar to that used by PETC will be purchased and installed. Modifications to the coal feed system are needed to allow accurate metering of the fuel, and to eliminate problems with uneven feeding. Coal sizing equipment will be upgraded to allow more control over the size of the coal feed. An existing tubular ESP will also be installed using non-DOE funds.

3.2 Task B. Shakedown and Baseline Testing

Procedures for measuring flame stability and ignitability will be refined. Equipment that has been modified will be tested to ensure it operates properly. The data acquisition and control system and new instrumentation will also be tested.

A baseline test will be performed. The baseline coal will be a western subbituminous coal. Baseline testing will include flame stability testing and an extended ash fouling and slagging test. Funding for this task will be split 60%/40% Cooperative Agreement and non-Cooperative Agreement funding.

3.3 Task C. Characterization of a Lignite and Petroleum Coke Blend

This task is not part of the cooperative Agreement but will be performed in conjunction with this cooperative program.

A northern Great Plains lignite will be blended with petroleum coke in blends ranging from 60%:40% lignite:petroleum coke to 100% lignite. The purpose of the blending is mainly to improve the energy density of the lignite so that it can be competitively marketed and used in the industrial sector. Results will be applicable to utility customers. It is expected that the blending will also improve the fouling characteristics of the lignite. The characterization will include a detailed analytical screening, carbon loss and deposition studies on the drop-tube furnace, and an extended fouling and slagging test on the ash fouling furnace, including flame stability. All work will be performed at EERC.

3.4 Task D. Characterization of a Subbituminous Coal and Petroleum Coke Blend

This task is not part of the Cooperative Agreement but will be performed in conjunction with this cooperative program.

A high fouling western subbituminous coal will be blended with petroleum coke to a maximum blend ratio of 60:40 subbituminous coal:petroleum coke. The purpose of the blending in this case is primarily to reduce fouling, and will also increase the energy density while disposing of a by-product from an associated industry. The target of this blending exercise is the utility industry; however, results will be applicable to users in the industrial sector. The test protocol will be the same as outlined in Task C. All work will be performed at EERC.

3.5 Task E. Characterization of a Low-Sulfur Subbituminous Coal and a High-Sulfur Bituminous Coal Blend

This task assumes a carryover of $35,000 from the 1988-1989 Cooperative Agreement year.

A low-sulfur western subbituminous coal will be blended with a high-sulfur bituminous coal for this task. The target market would be utility customers in the Midwest. The main purpose of the blending is for sulfur control from the viewpoint of the bituminous coal. From the perspective of the subbituminous coal, the blending will increase the energy density and may improve the fouling tendencies.

EERC will perform a detailed analytical screening of the parent coals and various blends. The blend ratio will vary from 100%:0% to 0%:100% subbituminous:bituminous. Following the analytical characterization, drop-tube work to characterize carbon burnout and depositional characteristics will be performed by EERC. Combustion tests of this blend will be performed by the Coal Combustion R&D Division of PETC. EERC and PETC personnel will work together and combine the results into a detailed package characterizing the blending of the two test coals.

4.0 ACCOMPLISHMENTS

Work performed during the first year was focused primarily on Tasks A and B, described above. To accomplish the objectives of these tasks, a microprocessor-controlled weight-loss coal feeder and an IFRF-type adjustable secondary air swirl burner were purchased and installed on the ash fouling test combustor at EERC. A mechanical-aerodynamic coal sizer was also purchased and was installed in EERC's coal preparation facility. Other equipment added to the test facility were a tubular electrostatic precipitator (ESP), two high volume cyclones, and a data acquisition/control system (including pressure transducers and orifice meters). These items were available from past EERC projects. To accommodate the new equipment, the primary air line, secondary air line, and heat exchangers were repiped. A high velocity thermocouple (HVT) and a baseline heat flux probe were also designed and constructed by EERC engineers and technicians to provide enhanced characterization of coals, slurries, and blends tested in EERC's test furnace.

4.1 Task A. Combustor Modifications

Modifications to the AFU and auxiliary systems were broad in scope and covered all aspects of the combustion system from the mill to the particulate control device. Many of the activities occurred simultaneously; however, most were dependent upon completion of one or more areas. Because of this interdependence, combustor modifications were not completed until May of this year. System modifications included: 1) the rearrangement of heat exchangers to accommodate the tubular ESP and sampling cyclones that were installed, 2) the installation of the mechanical-aerodynamic coal sizer, new coal feeder, meters and control valves for the furnace wall slag probes, and the data acquisition system, 3) the design and construction of the baseline heat-flux probe and HVT, and 4) modification of the probe bank and associated cooling air system. Schematics of the combustion test furnace before and after modification, can be seen in Figures 1 and 2.

4.1.1 Coal feed characterization and control

Changes in the particle size of the pulverized coal entering the combustion system can skew combustion results by decreasing the stability of the flame under a given set of conditions, decreasing the rate of carbon

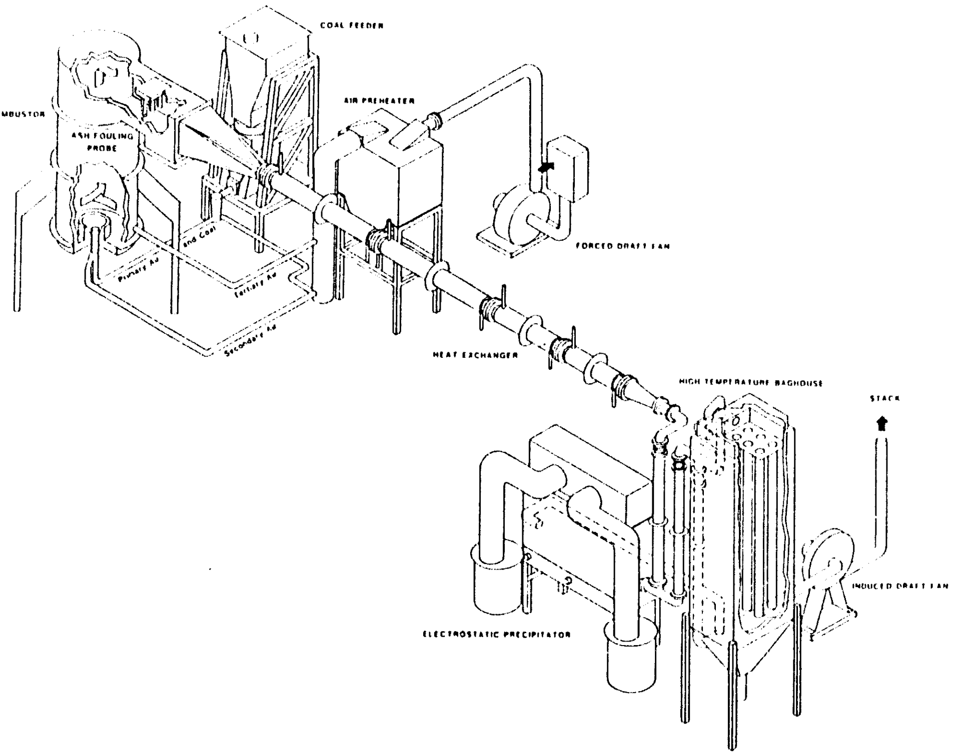

Figure 1. Schematic of combustion test system prior to changes.

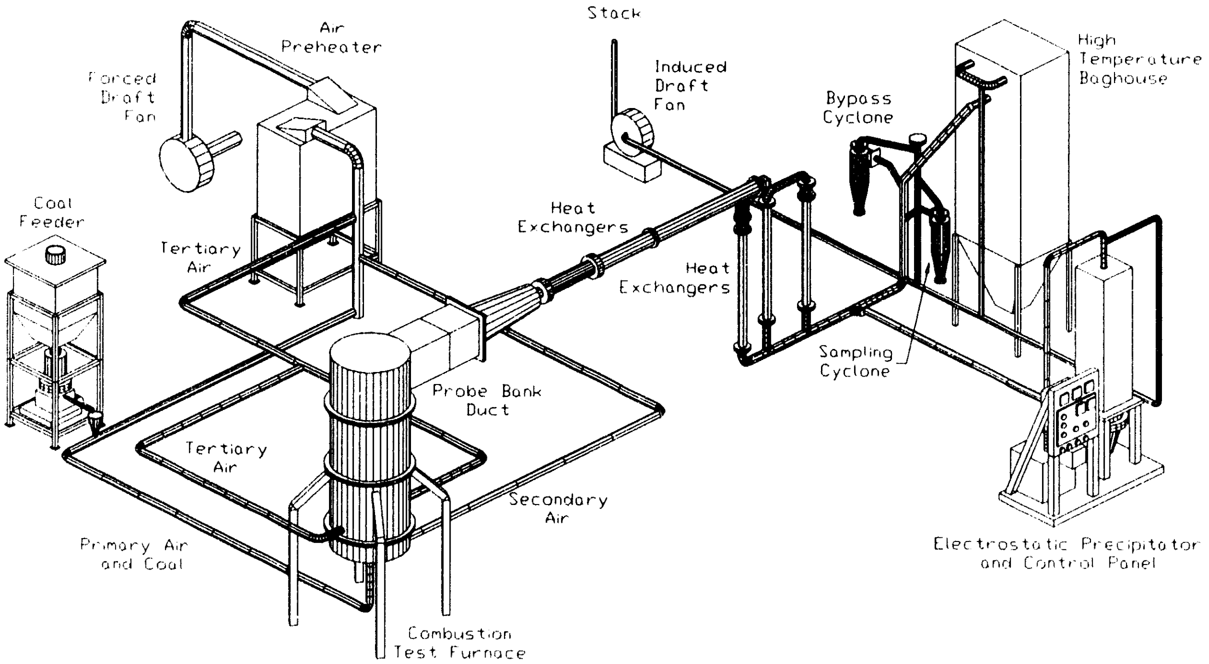

Figure 2. Schematic of combustion test system after modification.

burnout. This type of change cannot be tolerated for a series of tests on the same fuel. For the low-rank coal fuels test program, a mechanical-aerodynamic separator (Figure 3) was installed in EERC's coal preparation facility. The separator provides a consistent particle size for each combustion test, which is approximately 70% less than 200 mesh for testing on the ash fouling furnace. The separator was mounted near a 400-lb/hr pulverizer and was calibrated prior to shakedown testing. The sizer has two levels of 24 fan blades that can be removed in any combination to achieve a normal distribution about any specified feed size.

The microprocessor-controlled weight-loss coal feeder, seen in Figure 4, allows on-line monitoring of the feed rate and was used during shakedown testing to maintain combustion conditions such as air/fuel ratio and furnace exit gas temperature. Because of the sensitivity of the weigh mechanism, the feeder has a small hopper (two cubic foot volume), and an additional hopper is required for coal storage. The microprocessor controls a pneumatic valve that opens to fill the feed hopper when the fuel level is below a given set point. Initial shakedown of the feeder indicated problems with the power supply and tachometer which are described below (4.2 Task B). These parts were replaced and the system appears to work very well.

4.1.2 Combustion characterization

To aid in the characterization of the flame and the combustion environment, an adjustable secondary air swirl burner was installed, a high velocity thermocouple was constructed, and a baseline heat flux probe was also constructed. During flame stability testing, the swirl burner (shown in Figure 5) can be adjusted to determine the level of swirl required to achieve proper backmixing of secondary air. Visual observation of the flame can be used to evaluate flame standoff as a function of swirl setting. Combustor sight ports were modified to give a better view of the burner cone and flame, and photographs (standard 35mm and 35mm infra-red) can be used to record the results at each condition. In addition, a high-velocity thermocouple (HVT) and a baseline heat flux probe are also used to evaluate the furnace temperature profile and heat flux to the furnace wall as a function of secondary air swirl.

A high-temperature extraction probe, designed by Northwest Research Inc. in conjunction with EERC engineers, was under construction and not available for use during shakedown testing. The extraction probe uses nitrogen to quench ash intermediates as they are collected and will be used to characterize carbon burnout as a function of residence time and swirl setting.

Samples collected by the extraction probe can also be submitted for advanced analytical techniques using a scanning election microscope (SEM). Results of these analyses can aid in determining the reactions of mineral matter in the flue gas prior to deposition and can be compared with SEM analyses of deposits collected on heat transfer surfaces in the simulated convective pass.

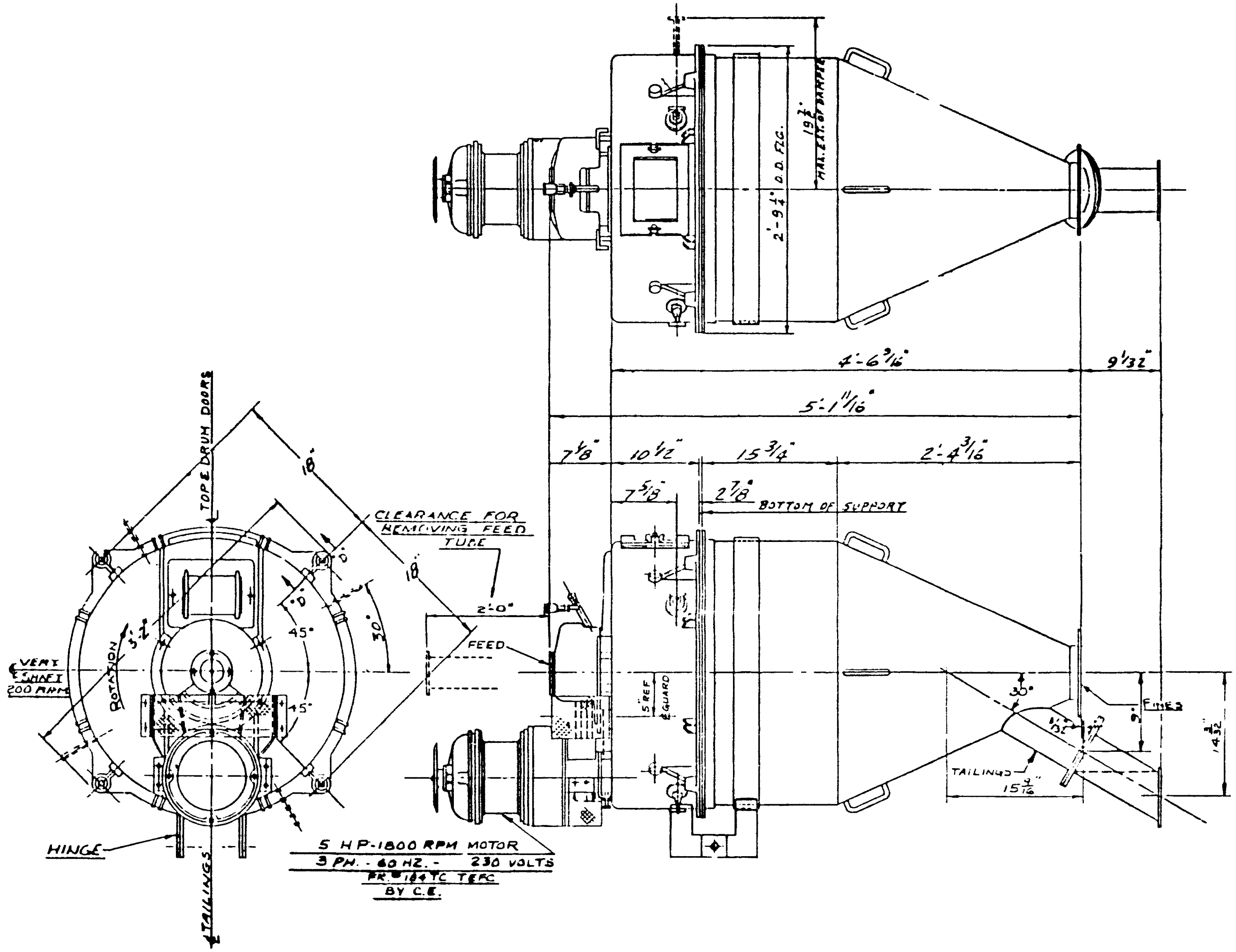

Figure 3. Schematic of 30" double whizzer mechanical separator.

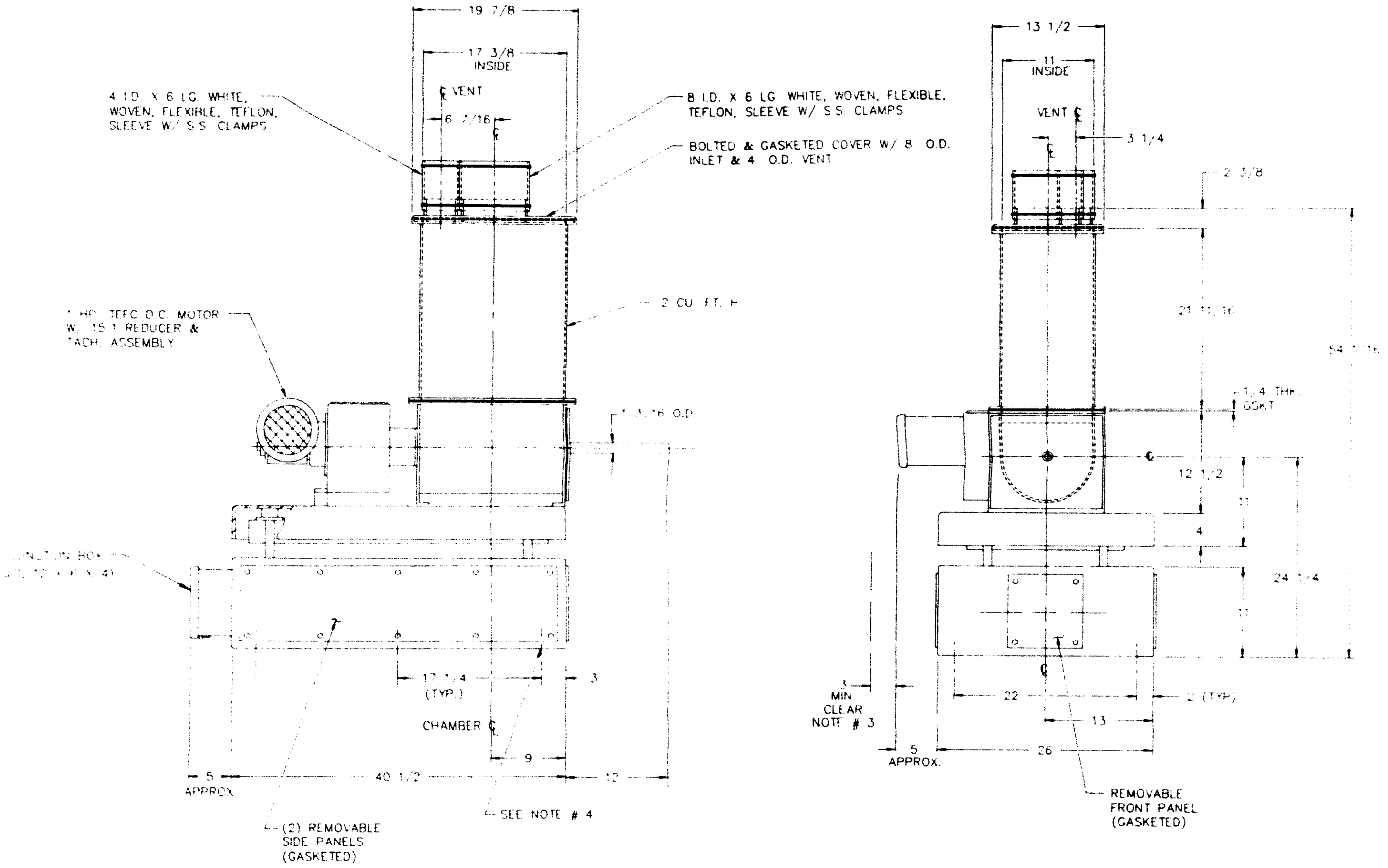

Figure 4. Schematic of new coal feeder.

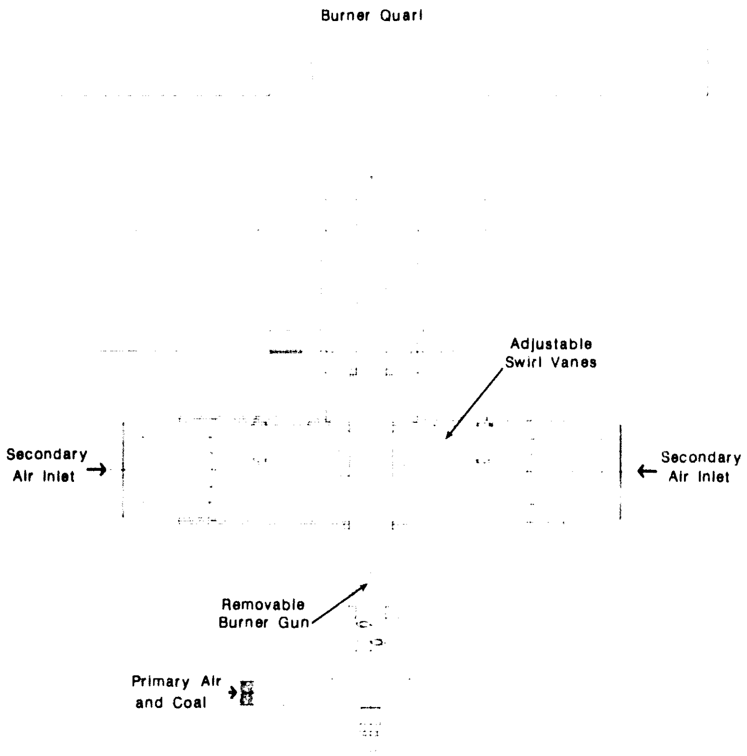

Figure 5. IFRF adjustable swirl burner.

4.1.3 Fly Ash Characterization

Changes were made to the combustion furnace's particulate collection and control systems to increase fly ash characterization capabilities. The newly-installed tubular ESP, which can be operated under cold-side (300°F to 350°F) or hot-side (750°F) conditions, adds a degree of flexibility previously unavailable for particulate control testing. The rearrangement of heat exchange equipment allows the flue gas to bypass a pair of heat exchangers to achieve the higher gas temperature. The ESP has electric heaters to maintain gas temperature and will be operated at a flue gas velocity of 5.0 ft/sec through the ESP. Excess flue gas (if any) will be bypassed through one of the cyclones. This arrangement should also give EERC the capability of testing ESP collection efficiency as a function of rapping frequency and/or flue gas conditioning at a constant SCA (standard collection area). The ESP is instrumented to monitor current through the collected ash layer to aid in determining the optimum rapping frequency. A photograph of the ESP and associated power supply is shown in Figure 6.

A bypass cyclone and a sampling cyclone were also added to the particulate control section of the furnace. The bypass cyclone will be used during heatup to prevent moisture from accumulating in the sampling cyclone or the ESP. The sampling cyclone will be used to collect a high volume sample

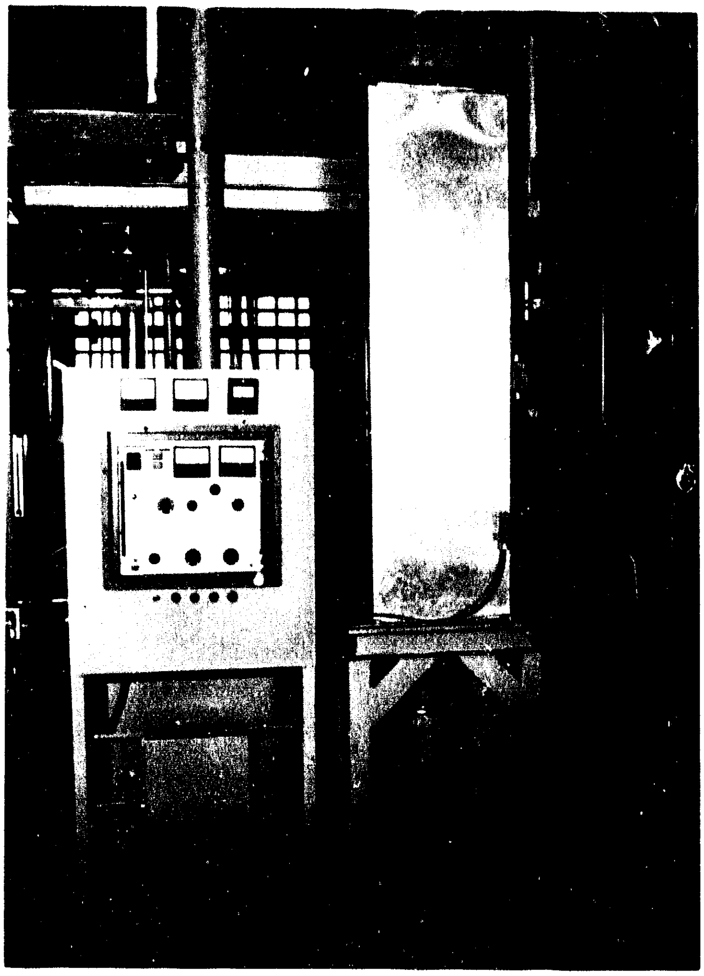

Figure 6. Tubular ESP and control panel.

for resistivity testing and other analyses (SEMPC, XRF, XRD, ESCA, etc.). During sample collection, the sampling cyclone will be operated at a pressure drop of 4 inches of water column to achieve a cut size of 3 micrometers. The identical cyclones will also be calibrated to measure flue gas flow rate as a function of pressure drop.

4.1.4 Data acquisition and control

A Kiethly 5000 data acquisition and control system was installed in the instrumentation panel board of the ash fouling furnace. The system can read up to 48 temperature inputs and sixteen 4-20 mA inputs (from pressure transducers and flow meters), and can be used to control up to eight peripheral devices (control valves, and various other equipment) using a 0 to 5 volt signal. At present, the acquisition and control program reads 45 temperature inputs, 9 pressure inputs, and 2 flow meter inputs (attached to the water-cooled slag probes), and controls the slag probe's water supply valves. The computer-driven acquisition and control system will be used to gather information from recording analyzers (O_2, CO_2, NO_x, and SO_x), orifice meters, and the coal feeder to monitor air/fuel ratio and suggest changes in fuel or air settings to achieve the desired furnace exit gas temperature and the appropriate level of excess air. The system will also be used to alarm operations personnel to any run-threatening condition (ESP inlet temperature too high or FEGT out of range, etc.).

4.2 Task B. Shakedown and Baseline Testing

Shakedown testing on the ash fouling test combustor at EERC was begun in May of this year. The original estimate for completion of shakedown testing called for 6 complete tests. To date, 3 complete tests and 3 partial tests have been run. Shakedown testing used readily available North Dakota Indian Head and Beulah lignites. The shakedown testing program was designed to identify problem areas with the newly installed equipment, establish performance criteria for the tubular ESP and sampling cyclone, establish the procedure for flame stability testing, evaluate the data acquisition and control system, and also to evaluate the use of external probes (high velocity thermocouple and baseline heat flux probe) for combustion characterization.

4.2.1 Test 1a and 1b

Initial shakedown of equipment was to include evaluating the microprocessor-controlled weight-loss coal feeder, checking for system leaks, and establishing the temperature entering the ESP. In conjunction with these activities, the mechanical-aerodynamic particle sizer was to be evaluated for the purpose of obtaining a normal distribution of coal feed size in the 70% less than 200 mesh range. A secondary objective for each combustion test was to evaluate the furnace preheat on natural gas to determine the optimal heating rate required to achieve the appropriate furnace exit gas temperature.

Results from these tests indicated problems with the coal feeder. During the first test, coal was burned in the furnace for approximately one hour before the coal feeder had difficulty reading the tachometer signal used to monitor the screw speed. Under this condition, the feeder cannot make adjustments in screw speed to achieve the desired feed rate, and the system turned off as a safety precaution. The tachometer alarm was reset for the second test and the feeder appeared to work well for approximately two hours prior to a second shutdown. Troubleshooting procedures indicated that the feeder power supply and tachometer were inoperable and required replacement. A new power supply and tachometer were installed prior to the next shakedown test.

The furnace is preheated with natural gas to the desired exit gas temperature prior to the switch to coal. Under the set procedure for preheating the furnace, the level of natural gas is gradually increased over an 8 hour period until the desired furnace exit gas temperature has been maintained for approximately 1.5 hours. In an effort to evaluate the effectiveness of the preheat period, an orifice meter was installed on the natural gas line and air flow measurements were taken during the combustor preheat period of the first two tests. The data indicated that the furnace was operated at near 75% excess air during this period and that adjustments in fuel feed rate and air flow rate could reduce the preheat period to less than 8 hours.

4.2.2 Test 2a

Shakedown 2 was designed to establish the effectiveness of the newly constructed sampling cyclone as a means of obtaining a high volume ash sample for study. The cyclone was designed to provide a cut point of approximately 3 microns when operated at 4 inches of water pressure drop. Three EPA Method 5 dust loadings were taken at the inlet and outlet of the sampling cyclone during the combustion test and the results indicated that the cyclone was oversized. The maximum pressure drop achieved was approximately 2 inches of water and the overall efficiency of the cyclone was about 75%. Based on these findings, a smaller cyclone will be required for collection of fly ash for study.

Combustion testing utilized a North Dakota Indian Head lignite, fired at the rate of 72.5 lb/hr. The furnace exit gas temperature was maintained at 2,000°F and the excess air level was maintained near 25%. Air-cooled deposition probes were inserted into the duct and a deposit was collected over a 5.25-hour test period. The surface temperature of the probes was maintained near 1,000°F. Samples of the coal were taken during the test and submitted for proximate and ultimate analyses, gross heating value, and determination of the mineral oxides present in the coal ash using x-ray fluorescence. Results of these analyses can be seen in Tables 1 and 2. The high moisture fuel was more difficult to feed, as a screw speed of nearly 100% of maximum was required to achieved the feed rate reported above. The size of the feeder screw was designed to deliver up to 100 lb/hr of a high moisture Lignite. The results indicate that a larger screw may be required for combustion testing of low-rank coals.

The deposit was gray to brown in color and weighed 369 grams. Deposits collected over 5.25 hours in the range from 300 to 500 grams are considered high fouling. Observations of the strength of the deposit were made by a pilot-plant operator and a strength rating factor (SRF) of 3.5 was assigned to the deposit. The SRF is a rating from 1 to 10, with 1 indicating soft and crumbly, and 10 indicating hard and unfragmented. The fouling deposit collected during the test was typical of North Dakota Lignites and reflects the total ash content and ash sodium level (4.3% SO_3-free) of the coal. Furnace wall slag probes were inserted in the furnace at the locations indicated in Figure 7. Deposits collected on the simulated water-wall probe and a horizontal probe weighed 1.7 grams and 5.5 grams, respectively. The deposits were lightly sintered and indicated a low slagging potential for the Indian Head coal.

The fuel appeared to burn very well in the test furnace and no major problems were noted during the combustion test. The feeder appeared to work well, as coal feed rate and screw speed were continuously monitored. However, the totalizer on the feeder was not reading properly and troubleshooting indicated that a new CPU was required for the microprocessor. The newly

TABLE 1

COAL PROPERTIES OF INDIAN HEAD LIGNITE

Test Number	Shakedown 2a	
Date Sampled	5/9/90	
Sample Number	90-0526	
	As-fired	Moisture-free
Proximate Analysis, wt%		
Moisture	35.90	---
Volatile Matter	26.90	42.00
Fixed Carbon	28.88	45.02
Ash	8.31	12.98
Ultimate Analysis, wt%		
Hydrogen	2.93	4.57
Carbon	39.03	60.92
Nitrogen	0.54	0.85
Sulfur	0.69	1.08
Oxygen	12.54	19.58
Ash	8.31	12.98
Moisture	35.90	---
Heating Value		
BTU/lb	6,631	10,352

TABLE 2

COAL ASH PROPERTIES OF INDIAN HEAD LIGNITE

Test Number	Shakedown 2a	
Date Sampled	5/9/90	
Sample Number	90-0526	
Coal Ash Analysis	wt% of Ash	wt% SO_3-Free
SiO_2	30.9	38.4
Al_2O_3	14.8	18.4
Fe_2O_3	5.1	6.3
TiO_2	0.7	0.9
P_2O_5	0.9	1.1
CaO	16.9	21.0
MgO	6.6	8.2
Na_2O	3.5	4.3
K_2O	1.0	1.2
SO_3	19.5	00.0
Total	99.9	99.8

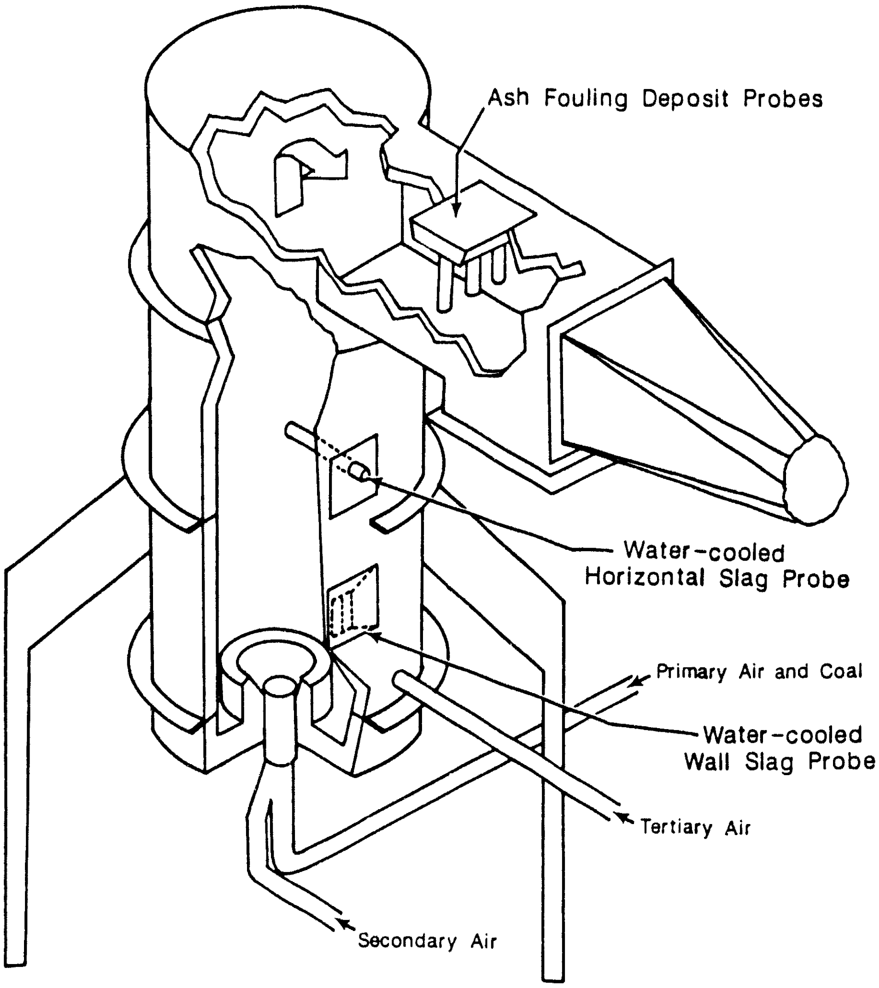

Figure 7. Location of water-cooled slag probes.

constructed HVT was also used during this test to measure the flame temperature near the tip of the flame. Combustion temperatures were measured in the furnace between 2,550°F and 2,600°F. This compares favorably with HVT readings taken at the same location during previous tests using a larger HVT. A photograph of the HVT and associated sampling system is shown in Figure 8. The temperature of the gas entering the heat exchanger was measured at 86°F, indicating that no additional cooling of the gas was required. Velocity past the thermocouple junction was over 285 ft/sec.

During the combustor preheat period, the natural gas flow rate was increased and the air flow rate was maintained to achieve approximately 10% excess air. The furnace exit gas temperature and the refractory temperature near the duct were monitored and indicated that preheat had been achieved in five hours, nearly three hours less than under standard operating procedures used in Tests 1a and 1b.

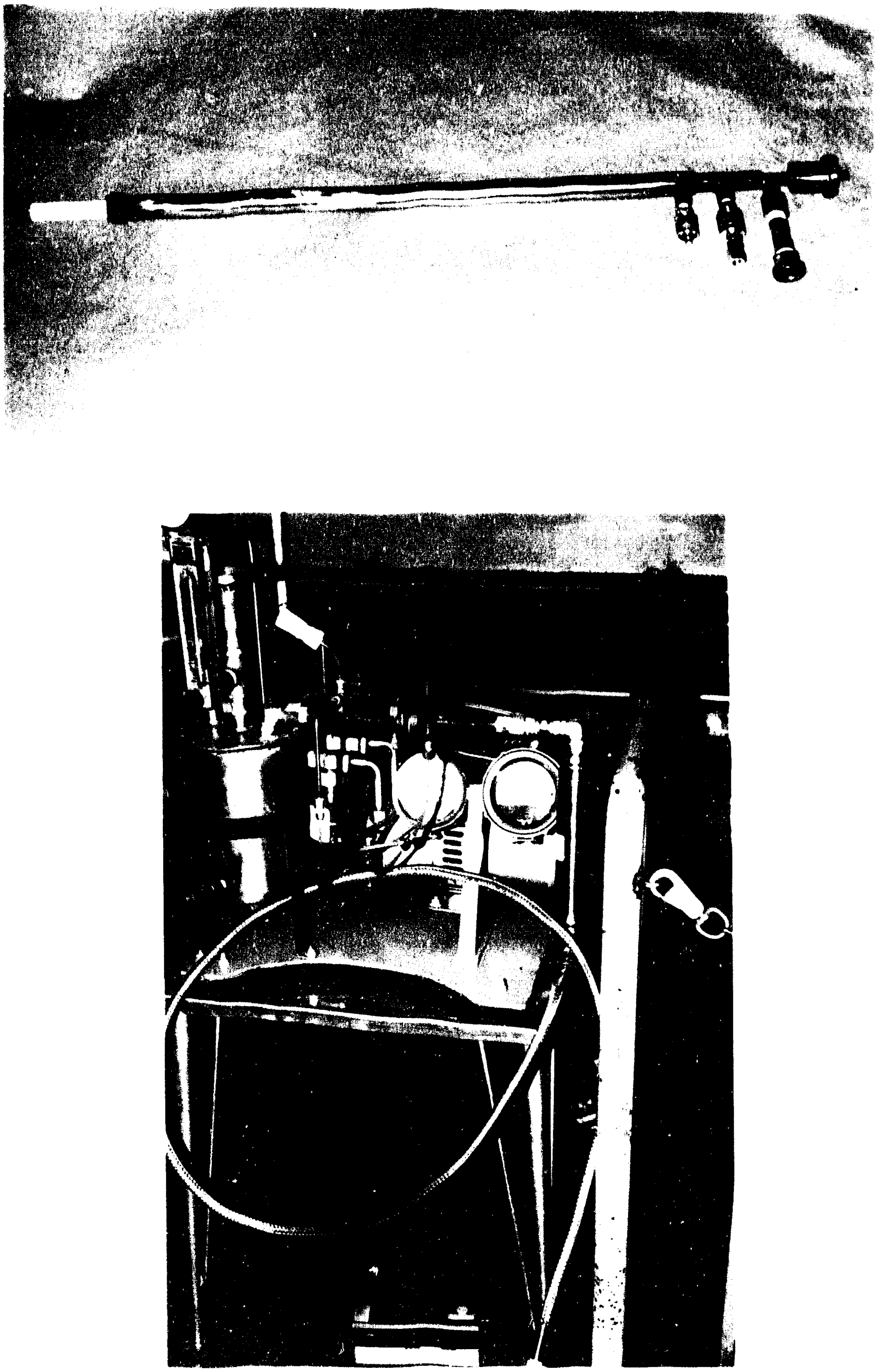

Figure 8. Photographs of the HVT and associated sampling system.

4.2.3 Test 2b

This test was designed to establish the procedure used during flame stability testing. Originally, this test was also designed to compare the difference in deposition rate between the adjustable swirl burner and EERC's standard burner (which had been used in over 670 previous combustion test). In preparation for a privately sponsored combustion test on the unit, a 2-inch refractory liner had to be installed and cured prior to combustion testing, thereby eliminating the opportunity to compare swirl burner deposition versus non-swirl burner deposition. The test was successful in establishing the procedure for flame stability testing, as stability was monitored at varying levels of swirl from 1.9 to 0.2 under both full load and turndown conditions. Swirl is defined as the ratio of radial momentum to axial momentum and indicates the level of back-mixing of secondary air in the burner zone.

Flame stability testing used primary and secondary air only, at approximately 15% and 85% of the total air, respectively. The general test method set the burner at its maximum level of swirl and monitored system parameters such as fuel feed rate, excess air, gaseous emissions (CO_2, CO, and SO_x), combustor static, and air flow rates. Photographs of the flame and burner zone were then taken through a sight port in the furnace proper just above the burner cone using standard 35 mm film. Flame temperature was also measured using a high velocity thermocouple (HVT) at a set location in the furnace. The swirl setting was then reduced to the next lower setting and data was taken as described above. The procedure was repeated until the flame was visually observed to lift off the burner quarl. At this point the flame was characterized as unstable under full load conditions (furnace exit gas temperature maintained at 2,000°F). Photographs of the flame were taken at each setting and are shown in Figures 9 - 12. The photographs indicate that under full load, flame stability was maintained at swirl settings of 1.9, 1.5, and 1.0, while flame liftoff was observed at a swirl setting of 0.5.

Flame stability under turndown conditions was characterized by reducing the feed rate of the fuel to one-half the full load rate, maintaining the same primary air flow rate and adjusting the secondary air flow to meet excess air requirements. The procedure described above was repeated until the flame was determined to be unstable. Figures 13 - 16 indicate that flame liftoff occurred at a swirl setting of 1.0 under turndown conditions (one-half load in this case).

4.2.4 Test 3

The purpose of this test was to establish the procedure for operating the newly installed tubular ESP. The APS sampling system was used to measure outlet dust loadings as a function of rapping frequency and duration, to determine the length of time required to condition the ESP electrodes. The ESP test was discontinued when an apparent short caused the current-carrying wire to spark, disrupting the electric field and contaminating the outlet dust loading sample. Flue gas was routed through the bypass cyclone for the remainder of the test.

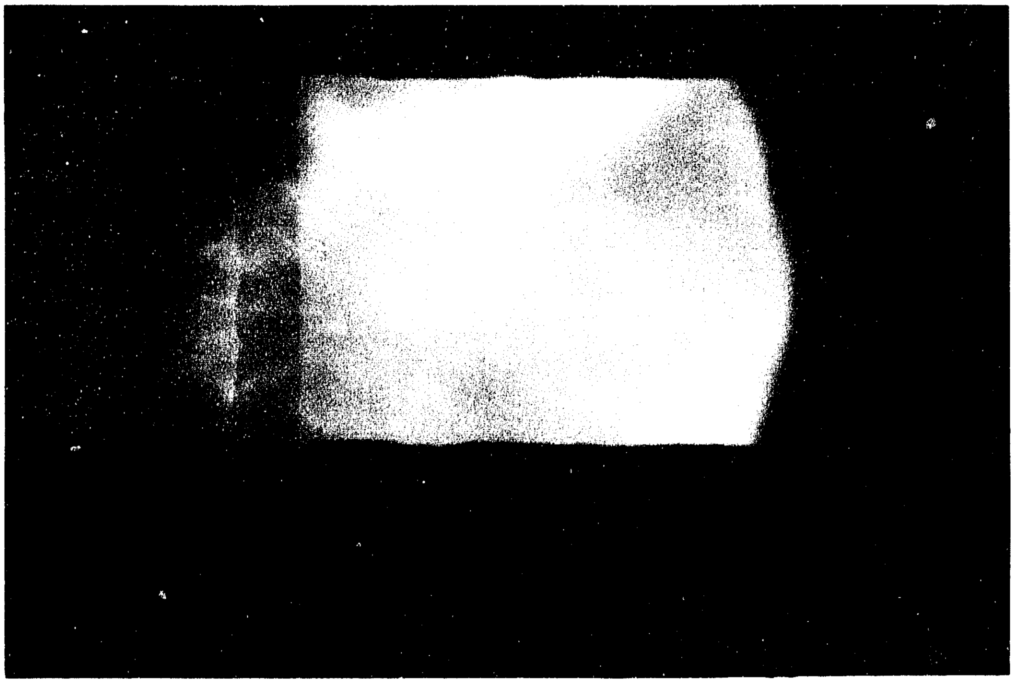

Figure 9. Indian Head flame at full load and swirl setting of 1.9.

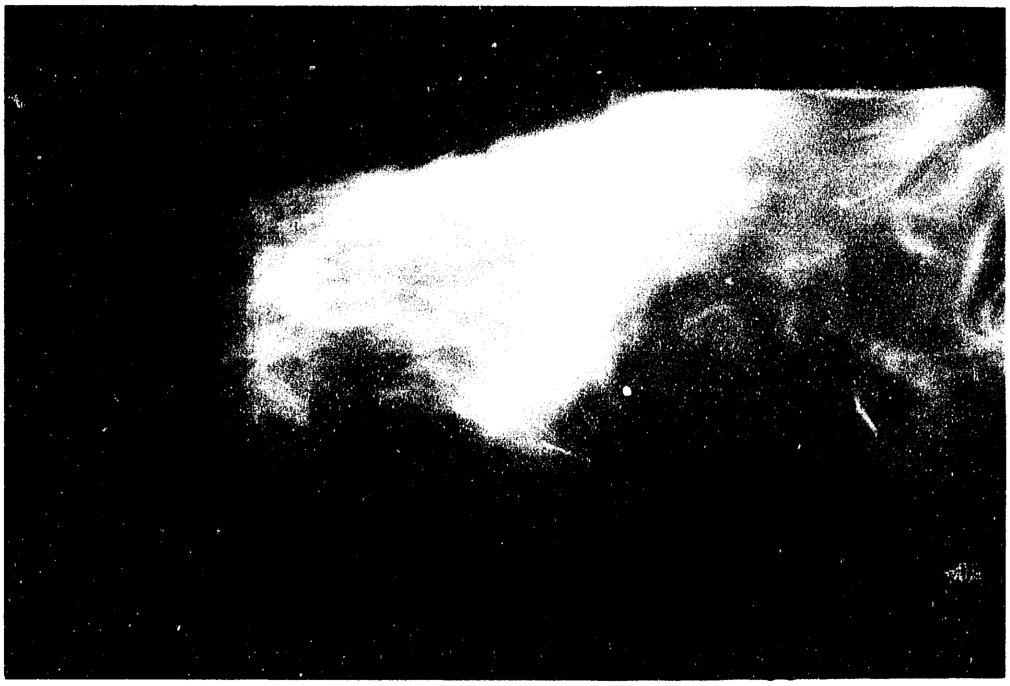

Figure 10. Indian Head flame at full load and swirl setting of 1.5.

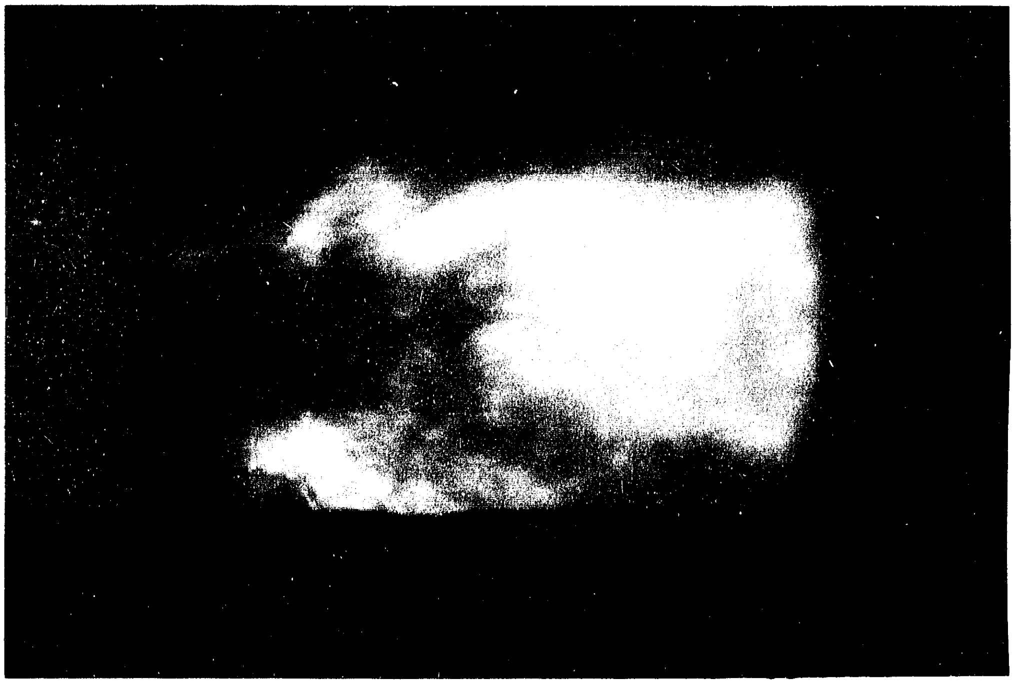

Figure 11. Indian Head flame at full load and swirl setting of 1.0.

Figure 12. Indian Head flame at full load and swirl setting of 0.5.

Figure 13. Indian Head flame at one-half load and swirl setting of 1.9.

Figure 14. Indian Head flame at one-half load and swirl setting of 1.5.

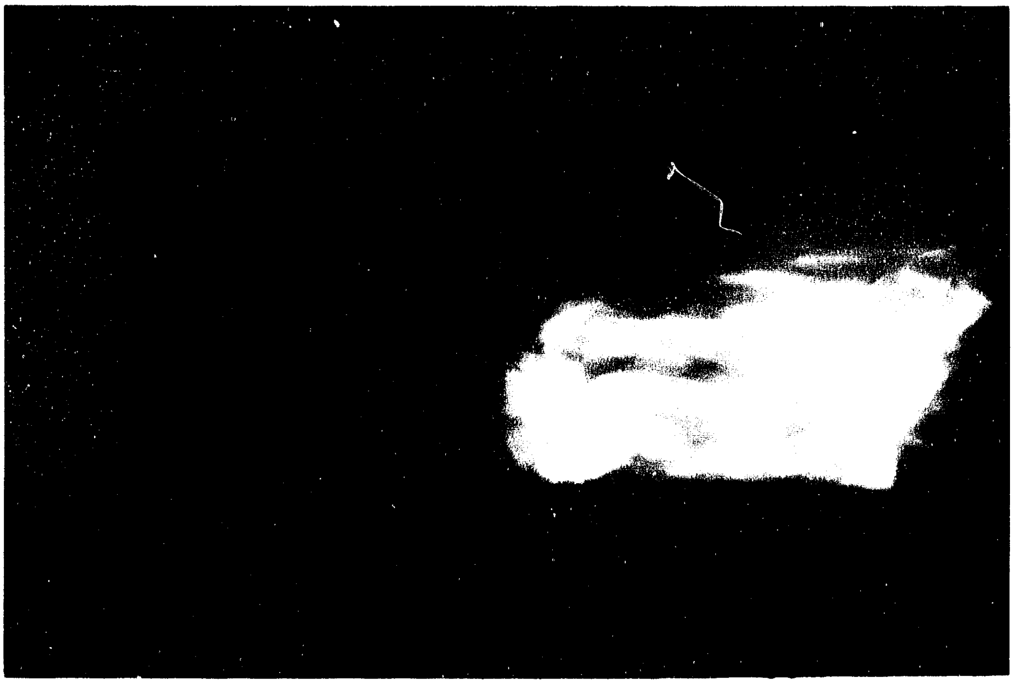

Figure 15. Indian Head flame at one-half load and swirl setting of 1.0.

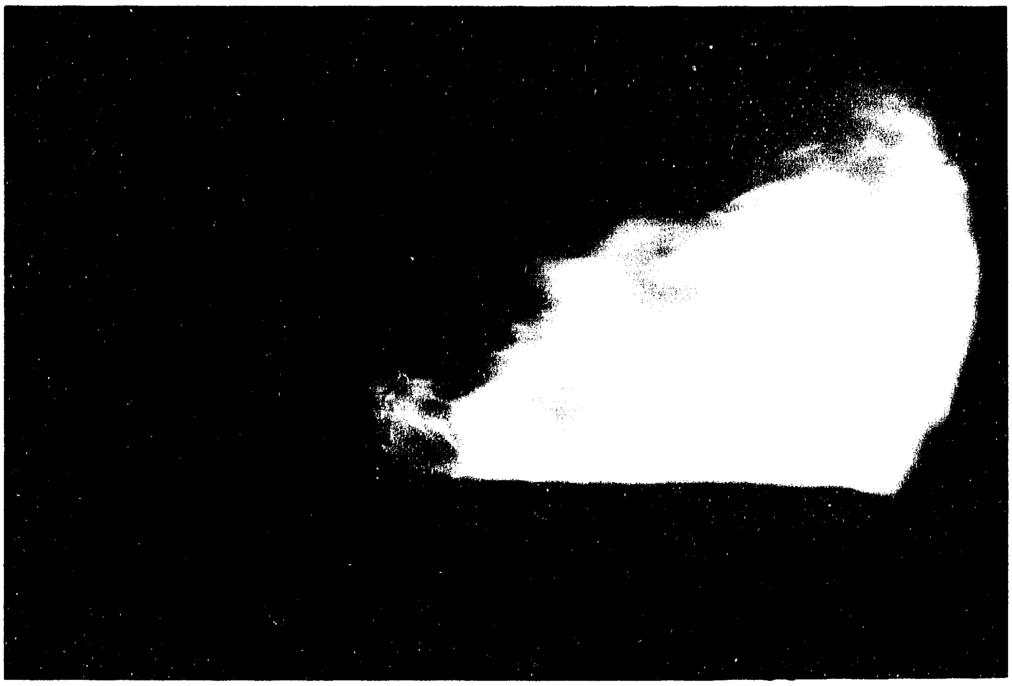

Figure 16. Indian Head flame at one-half load and swirl setting of 0.5.

A recently obtained Beulah lignite was used during the combustion test. Analyses of the coal and ash can be seen in Tables 3 and 4, respectively. The adjustable swirl burner was used to determine the effect of burner type on the observed deposition rate. Because the EERC standard burner had been used in over 670 tests on the combustion test furnace, effects of burner type need to be evaluated to determine whether the adjustable swirl burner can be used during combustion testing as well as flame stability testing.

The furnace exit gas temperature was maintained at 2,000°F for the duration of the test. Excess air was maintained near 25%, and the deposit probe surface temperature was maintained at 1,000°F. The deposit collected was gray, very soft, and uncharacteristic of a Beulah lignite. The deposits were sloughed from the tubes when removed from the furnace duct. The total weight of the ash collected at the bottom of the duct was 288 grams, and the rated strength of the deposit was 2 (on a scale from 1 to 10, as previously mentioned). Previous testing of Beulah lignite on this furnace had indicated high fouling rates and high strength deposits that were tan or brown in color. At this point it was uncertain whether this change in the deposit characteristics was due to the swirl burner or the coal sample.

4.2.5 Test 4

Shakedown 4 was designed to compare the observed deposition rate (using the standard burner) to the rate observed in Test 3 while using the same fuel, and to again establish the operating procedure for the ESP. This run was terminated after 3.5 hours of operation due to coal feed problems. Inspection of the feed indicated that the coal during the latter stages of the test had a significant level of surface moisture, which caused the coal to cake in the feed auger. This reduced the feed rate, which caused the furnace exit gas temperature to drop below an acceptable test level. Dust loadings indicated that the ESP was operating at 95.6% efficiency during the run, and at 90.5% efficiency when the run was terminated. The deposit collected over the shortened test was similar to the deposit collected in Test 3, and also had an uncharacteristic deposit strength for a Beulah coal.

4.3 Task C. Characterization of a lignite and Petroleum Coke Blend

A commitment has been made by Manalta Coal, Ltd. to participate in a blending study using a Great Plains lignite and petroleum coke blend. The actual work on the project was expected to start in June, 1990; however, the program was rescheduled to begin in the fall of 1990. The scope of the work for this program includes detailed analytical characterizations of the coal and coke, drop-tube furnace tests of a range of blends and operating conditions, and a 32-hour combustion test on the optimal blend to determine combustion characteristics.

4.4 Task D. Characterization of a Subbituminous Coal and Petroleum Coke Blend

Final fuel selections for the upcoming year for Detroit Edison did not include petroleum coke. Therefore, Detroit Edison decided not to fund this task.

TABLE 3

COAL PROPERTIES OF BEULAH LIGNITE

Test Number	Shakedown 3	
Date Sampled	6/15/90	
Sample Number	90-0655	
Proximate Analysis, wt%	As-fired	Moisture-free
Moisture	22.30	---
Volatile Matter	32.46	41.78
Fixed Carbon	33.68	43.62
Ash	11.34	14.60
Ultimate Analysis, wt%		
Hydrogen	3.38	4.34
Carbon	47.02	60.51
Nitrogen	0.58	0.75
Sulfur	1.87	2.41
Oxygen	13.49	17.37
Ash	11.34	14.60
Moisture	22.30	---
Heating Value		
BTU/lb	7,789	10,023

TABLE 4

COAL ASH PROPERTIES OF BEULAH LIGNITE

Test Number	Shakedown 3	
Date Sampled	6/15/90	
Sample Number	90-0655	
Coal Ash Analysis	wt% of Ash	wt% SO_3-free
SiO_2	26.3	34.0
Al_2O_3	14.7	19.0
Fe_2O_3	9.9	12.8
TiO_2	1.3	1.7
P_2O_5	0.9	1.2
CaO	14.2	18.4
MgO	6.2	8.0
Na_2O	3.8	4.9
K_2O	0.1	0.1
SO_3	22.7	00.0
Total	100.1	100.1

4.5 Task E. Characterization of a Low-Sulfur Subbituminous Coal and a High-Sulfur Bituminous Coal Blend

Due to funding limitations, no work has been performed on this task.

5.0 SUMMARY AND CONCLUSIONS

Modifications to the EERC pilot-scale ash fouling test combustor were completed and shakedown testing was begun in May, 1990. Changes made included the rearrangement of heat exchangers and the redesign of the fly ash sampling and collection system. A microprocessor-controlled weigh feeder and a secondary air adjustable swirl burner were purchased and installed on the ash fouling test furnace at EERC. A mechanical-aerodynamic particle sizer was also purchased and installed in EERC's coal preparation facility.

Initial shakedown indicated that a new tachometer and power supply were required for the newly installed coal feeder. Two shakedown tests were terminated due to problems associated with the feeder. These items were replaced and three full-length combustion tests were completed. The final shakedown test was terminated at 3.5 hours due to coal feed problems resulting from an abnormal amount of surface moisture in the fuel, which caused caking in the feed auger.

Results from the completed shakedown tests indicated that the sampling cyclone, constructed at EERC for the purpose of obtaining a high-volume fly ash sample, was too large and that a smaller cyclone would be required for future sampling. Shakedown testing was unable to establish the operating procedure for the single wire tubular electrostatic precipitator (ESP) recently installed on the combustion test furnace. During ESP performance testing, the current-carrying wire sparked, contaminating the outlet dust loading during the second set of readings on the ESP. Readings obtained indicated that the ESP could be operated at 95.6% efficiency under the conditions utilized during that test. Further shakedown of the ESP is required to establish the operating parameters for future testing. The flame stability testing procedure was successfully established in the final full-length combustion test. Visual observation of the burner cone and flame (recorded with 35 mm film) and high velocity thermocouple readings were used to verify differences in flame stability at varying levels of secondary air swirl and under both full load and turndown conditions.

The high-velocity thermocouple (HVT) sampling system, designed and constructed by EERC engineers and technicians, was successfully tested during the course of shakedown testing. Temperatures near the flame tip were recorded in the range of 2,550°F to 2,600°F, which compares favorably with previous HVT sampling on a similar fuel at the same location in the furnace.