4.0 COMBUSTION RESEARCH

4.1 Atmospheric Fluidized Bed Combustion

ATMOSPHERIC FLUIDIZED-BED COMBUSTION

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by

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ATMOSPHERIC FLUIDIZED-BED COMBUSTION

1.0 BACKGROUND

The main driving forces behind the use of fluidized-bed combustion have primarily been environmental concerns, fuel flexibility, and compatibility with low-cost fuels. Both bubbling and circulating designs have been developed for operation at atmospheric pressure, and many industrial-scale units of both types are currently in operation. A limited number of larger utility boilers have recently been commissioned. In addition, pressurized fluidized-bed combustion (PFBC) is making its entrance on the utility scale with the PFBC which recently began operation at the Tidd Station.

Even though fluidized-bed combustion (FBC) technologies are being commercialized, a number of areas require further research. An integrated approach should be taken toward FBC research interrelating those problems generic to bubbling, circulating, and pressurized fluidized-bed combustion systems. The program should also be designed to address specific problems related to each of these areas. Major issues facing fluidized-bed combustion are listed below:

- Methods are needed to minimize corrosion and erosion of in-bed and convective pass tubes, refractory and support surfaces, and expander turbines. Work should focus on the following:

  - Understanding mechanisms
    - ▸ Mineralogical properties of the bed and coal
    - ▸ Fluid mechanics of the bed
    - ▸ Corrosion versus erosion mechanisms
    - ▸ Stress forces on tubes
  - Assessing acceptable wastages
  - Identifying cost-effective methods of combating tube wastage
  - Developing systematic test devices

- Retrofit applications should be addressed for all types of FBCs. According to information from the American Boiler Manufacturing Association (ABMA), approximately 200 existing units are candidates for retrofit technologies. The FBC retrofits at NSP's Black Dog station, MDU's Heskett station, and Colorado Ute's Nucla station have demonstrated the feasibility of such applications.

- Fuel flexibility and characterization issues should be addressed to help users understand the constraints of fuel switching, the design considerations, and, most importantly, the economics involved in having fuel flexibility for the FBC.

- Agglomeration/sintering of bed materials and deposition on tubes, support surfaces, and refractory have been identified as problems by both manufacturers and users of FBC technology. Problem cases have been documented for both bubbling and circulating beds using a variety of fuels, including coal. The Energy and Environmental Research Center (EERC) has extensive experience in this area to help understand and solve this operational problem.

- Scaleup effects need to be addressed so that vendors and users can take pilot-scale data and be assured that the large-scale system will perform as anticipated. This database has been growing rapidly with all of the new units starting operation; however, much information is still required.

- Advanced systems should be designed to resolve problems and improve overall FBC performance. These systems should:

  - Increase the volumetric heat release rate.
  - Improve overall boiler efficiency.
  - Simplify fuel feed and ash removal systems.
  - Decrease capital and operating costs.
  - Improve turndown.
  - Decrease the size of units to enable modular construction.

- Several problems related to emissions from FBC systems need to be addressed:

  - Better sorbent utilization would improve the economics of FBC.

  - NO_x control is currently not a major problem, but could become more difficult with bubbling beds if standards become more stringent.

  - Information indicates that particulate-control problems may exist for certain types of ash. These ashes should be identified, and the use of specific equipment, conditioning, or other methods should be applied to resolve the problems.

  - Hot-gas cleanup is required for PFBC to meet turbine specifications in addition to New Source Performance Standards (NSPS).

These problems and concerns could limit FBC from reaching its full potential. Special efforts should be made to perform the necessary research to help FBC evolve into a mature technology that meets the technical, economic, and environmental needs of the future.

2.0 GOALS AND OBJECTIVES

A number of major issues have been identified that warrant further research. There are several issues in atmospheric bubbling FBC that the EERC has the capability to investigate; some of these issues are proposed in this work plan. Other FBC research should be funded, at least partially, by the industrial sector, either through the Electric Power Research Institute (EPRI) or private companies. Efforts should continue to transfer the expertise gained under previous Cooperative Agreements to the private sector.

The overall goal of the low-rank coal (LRC) FBC program at the EERC is to develop a technology database so that industry can introduce economically and environmentally acceptable coal technology options to the marketplace. Research will address those areas where data gaps exist in fuel flexibility and performance, potential operating problems, environmental compliance, advanced concepts, and system simplification.

2.1 Three-Year Objectives (7/89 - 6/92)

The EERC has developed an extensive database on the corrosion and erosion of boiler tubes, agglomeration and sintering of bed material, fuels and sorbent characterization, and particulate emissions through testing funded under the Cooperative Agreement. To successfully transfer this information to the private sector, the EERC will continue to publish results from this work at conferences and in refereed journals. The existing database will be supplemented by low-level experimentation, paper studies, economic evaluations, and surveys of operating plants and other researchers' data where needed to fill gaps.

Pilot-scale work has been performed evaluating the corrosion and erosion of boiler tube surfaces in bubbling beds. The EERC has done extensive analysis and characterization of samples generated from this testing. Over the next three years, available samples from industrial- and utility-scale boilers will be analyzed and the results correlated to bench- and pilot-scale work as well as to each other. The EERC will also attempt to obtain funding from non-DOE sources to perform more work on large-scale systems.

Efforts in corrosion and erosion will instead be focused on circulating fluidized-bed combustion (CFBC) during this time period. A pilot-scale CFBC is being constructed as part of another project. Initial work on this unit will involve system, coal, and sorbent characterization. During the characterization testing, an assessment will be made to determine if any meaningful corrosion and/or erosion data can be obtained. If meaningful data can be generated, EERC and METC personnel will discuss the possibility of incorporating CFBC corrosion and erosion work into this program.

Work will continue on the coal pretreatment cell currently being developed at the EERC. After each phase of the project, an assessment will be made to determine if the concept is still technically and economically feasible. The end result of this effort is expected to be a design of the pretreatment cell in conjunction with a bubbling and circulating FBC and an economic evaluation of the concept. Testing will be done at the pilot scale, with no demonstration planned as part of this program.

As part of the advanced concepts task and other non-Cooperative Agreement work, the EERC will work toward simplifying the control and operation of the FBC. As part of this task, users of FBC technology will be polled to identify operational problems with fuel and sorbent feed and ash removal systems. Based on priorities identified from this poll and results of other work, the EERC will work toward the simplification of control and operation of the FBC. Specific systems will be identified in either the second- or third-year work plans.

First-generation PFBC technology has reached commercialization, as indicated by the two recent Clean Coal awards for utility-scale plants. Second-generation concepts are now being developed. The success of these concepts will depend, in part, on an understanding of the effects of fuel properties on performance. The EERC will consult with developers of second-generation technology regarding the effect of fuel properties on drying, pyrolysis, combustion, and topping cycles.

The EERC is in the process of designing and constructing a pilot-scale CFBC as a part of a multiclient-funded program. This unit will be used to investigate the impact of CFBC and coal properties on performance. The test unit will not be available to this program for at least one year. Once the CFBC is available for testing, the current status of the technology will be assessed to determine if and how the unit should be incorporated into this test program.

Atmospheric fluidized-bed combustion (AFBC) has become an acceptable option for the generation of steam and electricity. A number of units are currently on-line in both the industrial and utility sector. Great market potential exists for the use of AFBC technology in both commercial and industrial sectors; however, to increase the acceptability of this technology, low cost, reliability, and ease of operation must be inherent to the system. The purpose of this task is to simplify the control and operation of the FBC for boilers in the range of 10,000 to 200,000 lb/hr. The goal of this system simplification is to make the unit easier to operate, reduce capital and operating costs, and increase the overall reliability of the system.

2.2 Proposed Third-Year Research (7/91 - 6/92)

Specific objectives of the Fluidized-Bed Combustion Project for Year Three of this three year period are as follows.

2.2.1 Task 1. Advanced Concepts--Coal Pretreatment Cell

During the past year, modifications were made to the pilot-scale pretreatment cell to allow operation under conditions that more closely simulate a full-scale unit. Also during the last program year, testing of a lignite and bituminous coal under low-temperature conditions was performed to understand and develop data under this mode of operation. Based on this preliminary information, a patent application will be prepared. Additional testing under the full range of operating conditions (temperature, air-to-fuel ratio, feed size, and residence time) will be completed using a lignite, subbituminous, and bituminous coal. The goal of this additional testing is to complete the proof-of-concept phase of the product. At the end of this program year, the coal pretreatment cell should be developed to the point where it can be marketed to commercial interests.

2.2.2 Task 2. Effects of Ash Chemistry--Corrosion/Erosion/Deposition

Understanding the impact of ash chemistry on the operation of all types of fluid beds (bubbling, circulating, and pressurized) is critical for the success of the FBC industry. To help industry understand and cope with problems related to ash chemistry, samples of corroded/eroded boiler tubes, deposits, agglomerates, sintered bed material, and other ash-related phenomena will be collected from full-scale users. These samples will be analyzed using the advanced techniques developed at the EERC, with the results correlated back to the original bed chemistry and the operating conditions of the boiler. The bed chemistry will be a function of the coal, sorbent, and bed material used, as well as operating temperature, excess air level, and residence time.

3.0 RESULTS AND DISCUSSION--COAL PRETREATMENT CELL

3.1 Equipment Description

Figure 1 shows a schematic of the EERC pilot-scale unit used for testing during the last reporting period. In the current configuration, coal is fed via a screw feeder into the coal bed approximately 26 inches above the distributor plate. A stainless steel conic section is located within the 8-inch ID coal pretreatment cell (CPC) combustor section just above the distributor plate and has the following dimensions: 24-inch length, 4-inch inlet diameter, and 8-inch outlet diameter. A combination of nitrogen and air is used as the fluidizing medium within the CPC. The addition of nitrogen allows for close control of combustion within the bed (air-to-fuel ratio) and provides additional gas volume for control of superficial gas velocity through the bed. Flue gas would be used in place of nitrogen in a commercial unit. The fluidizing gas is passed through three electric resistance heaters arranged in series within an insulated enclosure to preheat the air/nitrogen mixture to up to 1000°F, if needed, before flowing to the CPC.

Treated char flows by gravity from the top of the bed into a barrel. The char outlet is located approximately 4 feet above the distributor plate which, in combination with the in-bed conic section, allows for a bed volume of 1.0 ft^3. Fine coal particles, moisture, and volatile gases from the fluidized bed are carried out of the CPC and into a pulverized coal test furnace. The temperature within the test furnace is maintained at

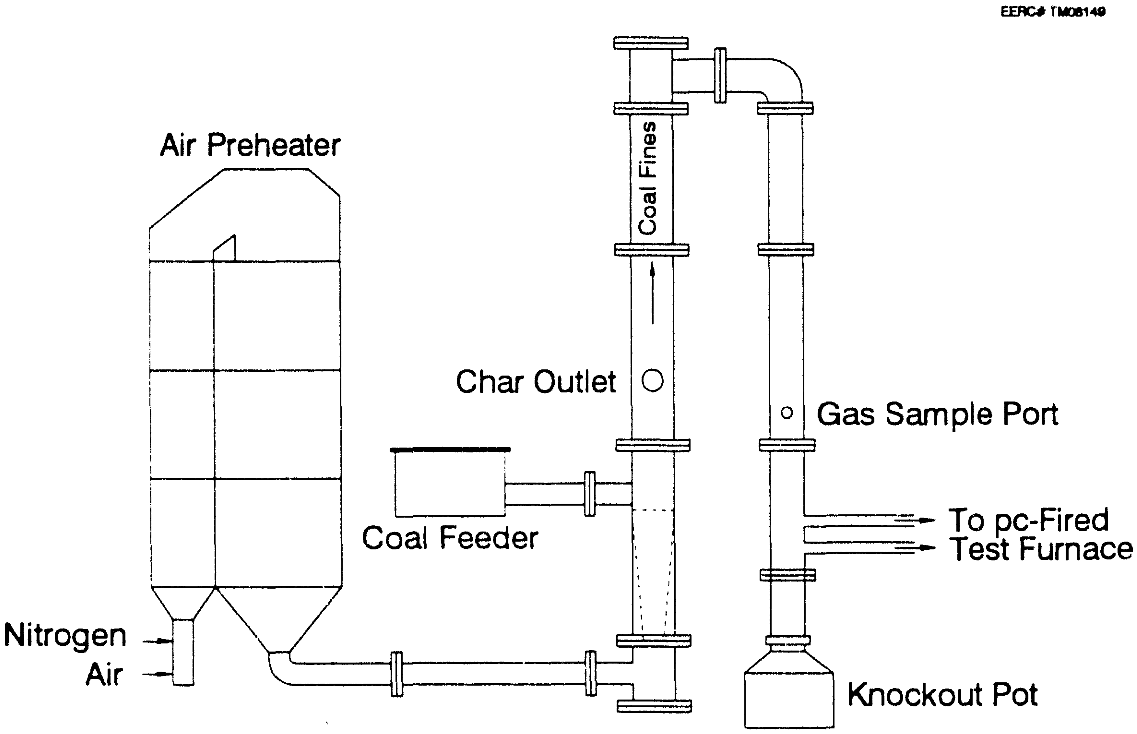


Figure 1. Schematic of the EERC pilot-scale coal pretreatment cell.

approximately 1600°F with natural gas as fuel to ensure combustion of the fines and volatiles from the CPC. A stainless steel knockout pot with a volume of 1.5 ft^3 is attached to the base of the CPC downcomer section to collect any coal fines too large to be carried through to the test furnace.

3.2 Test Procedures

A total of five tests were performed on the pretreatment cell using Blacksville bituminous coal to investigate the potential for beneficiation of the bituminous coal by simple drying only. All tests were run on feed coal having a top size of 1.25 inches. Bed inlet temperature, bed residence time (coal feed rate), and the air-to-fuel ratio (on a molar basis) were selected as independent variables for matrix testing. The superficial gas velocity through the top of the bed was set at a level high enough to maintain proper fluidization throughout the bed and to allow for a given size fraction to be retained within the CPC bed/freeboard, or to be drained through the product char outlet port. Table 1 shows the nominal test conditions under which the pretreatment cell was operated for the Blacksville bituminous coal drying tests.

3.3 Results of Blacksville Bituminous Coal-Drying Tests

Only limited results were obtained from operation of the coal pretreatment cell on the Blacksville coal. Each test period was plagued with fluidization problems due to the swelling nature of the coal. It was not possible to maintain steady-state operation for longer than about 45 minutes at any of the planned operating conditions without some type of plug occurring somewhere in the system, usually within the bed. These plugs would force either a significant increase in superficial gas velocity to reestablish fluidization, or a complete shutdown of the system to locate and clean out the problem area. Because of the operational problems encountered as a result of plugging, very little significant data were obtained from the tests that were run, and subsequent testing of the system with the Blacksville coal was discontinued.

3.4 Coal Pretreatment Cell Testing--Devolatilization Mode

Recently, two series of tests have been completed in a "devolatilization" mode of operation, one with a Beulah, North Dakota, lignite and the

TABLE 1

Blacksville Bituminous Coal Drying Tests
Nominal Test Conditions

Test Number	Inlet Temperature (°F)	Air/Fuel Ratio	Coal Feed Rate (lb/hr)
AC1-91	750	0.143	125
AC2-91	750	0.049	160
AC3-91	750	0.049	225
AC4-91	750	0.143	125
AC5-91	100	0.143	95

other with a Montana subbituminous coal. The coal pretreatment cell was operated with these two fuels at bed temperatures sufficiently high to initiate partial combustion of the fuel in the bed and allow for self-sustained operation at elevated temperatures (greater than or equal to 800°F). The purpose of the tests under these conditions is to determine the level of devolatilization of the coal; the quantity and size distribution of the char produced; and the amount of ash reduction in the product char at various levels of operating temperature, air-to-fuel ratio, and bed residence time. An overview of the results generated to date follows.

Tables 2 and 3 are the average output data and a material balance, respectively, generated from operation of the CPC on the North Dakota lignite. Included with Table 2 is a listing of the nominal test conditions under which the unit was operated. Table 2 shows that the lignite tests were run over a temperature range of about 840°-1125°F, bed residence times of 9 minutes to 18 minutes, and air-to-fuel molar ratios between 0.10 and 0.18. Calculated superficial gas velocities for the top of the expanded bed ranged from 4.4 ft/sec to about 10 ft/sec. Table 3 shows that, over the range of conditions tested, 100% of the moisture was driven out of the product char for each test, with the exception of Run CPC-BS1-0292 (99% moisture removal). The removal of volatile matter ranged from about 34% up to about 60%.

The ratio of the amount of char produced to the amount of coal fed ranged from 12% to about 40%, and there appears to be a relationship between temperature and char production, as shown in Figure 2. Char production data from three tests were plotted as a function of superficial gas velocity, and are shown in Figure 3. Operating temperature, air-to-fuel ratio, and bed residence time for each of the three tests were all similar at about 900°F, 0.10, and 11 minutes, respectively. Figure 3 suggests that, with all other conditions held constant, char production increases with increasing gas velocities. Figure 4 illustrates the relationship between the evolution of volatile matter and operating temperature. As expected, volatile matter evolution increased with increasing temperature. Figure 5 is a plot of particle-size distributions from Run CPC-BS1-0992 of various solids sampled associated with the pretreatment cell. The figure shows that the pretreatment cell is an effective solids classifier, and the size distributions of the product char and the collected fines (designated as drip leg fines) fall within rather narrow ranges.

A set of tests similar to those described above was also performed on a Montana subbituminous coal from the Colstrip Mine. The feed coal was screened prior to testing to remove the fine fraction less than 1/4 inch. The feed coal was screened to simulate more closely the size of material on which the pretreatment cell was originally designed to operate. The original design concept was to use a minimally prepared coal feedstock, possibly run-of-mine coal crushed once, and let the physical and chemical processes within the pretreatment cell reduce the average particle size and act as a classifier. The size of the product char would then be determined by specific operating conditions, such as gas velocities, temperature, residence time, and/or air-to-fuel ratio.

Tables 4 and 5 are similar to Tables 2 and 3 and show the average output data (operating conditions) and a material balance, respectively, for the Montana subbituminous coal testing. The nominal operating conditions under which the tests were performed are also listed on Table 4. As with the North

TABLE 2

North Dakota Lignite Average Output Data

Run No.	0292	0392			0492	0592		0692		0892		0992	1092
Test No.	N/A[a]	1	2	3	N/A	1	2	1	2	1	2	N/A	N/A
Average Feed Rate	117.1	121.3	121.3	121.3	182.8	199.1	199.1	233.0	233.0	173.5	173.5	168.9	181.2
TOB[b] Avg. Temp. (°F)	1075	1122	984	849	1105	840	947	990	1108	978	932	955	986
TOB Flow (acfm)	182.8	156.9	151.1	133.7	168.4	166.2	196.4	179.8	203.1	147.2	91.8	209.5	145.7
TOB SGV (ft/sec)	8.70	7.47	7.20	6.37	8.02	7.91	9.35	8.56	9.67	7.01	4.37	9.98	6.94
Fuel Input (mol/hr)	6.0	6.3	6.3	6.3	9.4	10.3	10.3	12.0	12.0	9.0	9.0	8.7	9.4
Air Input (mol/hr)	1.05	0.94	1.00	0.97	1.17	0.99	1.05	1.55	1.47	1.29	0.95	1.54	1.19
A/F Ratio (mol/mol)	0.174	0.150	0.160	0.155	0.124	0.097	0.102	0.129	0.122	0.144	0.106	0.177	0.128
O_2 Input (%)	11.4	12.1	12.3	12.1	13.9	10.0	9.7	16.0	14.5	16.2	18.4	13.4	15.2
Avg Residence Time[c] (min)	17.93	17.31	17.31	17.31	11.49	10.55	10.55	9.01	9.01	12.10	12.10	12.43	11.59

Run No.	Test No.	Comments
CPC-BS1-0292	N/A	Test #2 scrapped due to bad odors from AFU (improper operation)
CPC-BS1-0392	1	Preheat at 300°F only.
	2	
	3	Preheat turned back up to 800°F.
CPC-BS1-0492	N/A	Preheat at 300°F only.
CPC-BS1-0592	1	Preheat at 800°F.
	2	Preheat at 300°F.
CPC-BS1-0692	1	Preheat at 300°F only.
	2	Preheat at 300°F only.
CPC-BS1-0792	N/A	Drying tests for Kerr-McGee Proposal (with M. Musich).
CPC-BS1-0892	1	Preheat at 300°F only. Adjusted ρ_s[d] from 750 to 1200 kg/m³.
	2	Preheat at 300°F only.
CPC-BS1-0992	N/A	Repeat BS1-0592, Test 2 with new ρ_s
CPC-BS1-1092	N/A	Repeat BS1-0892, Test 1 with new ρ_s and. product drain extension

Nominal Conditions

Run No.	0292	0392			0492	0592		0692		0892		0992	1092
Test No.	N/A[a]	1	2	3	N/A	1	2	1	2	1	2	N/A	N/A
Bed Residence Time (min)	16.8	16.8	16.8	16.8	11.2	11.2	11.2	8.4	8.4	11.2	11.2	11.2	11.2
Feed Rate (lb/hr)	125	125	125	125	187.5	187.5	187.5	250	250	187.5	187.5	187.5	187.5
TOB Avg. Temp. (°F)	1100	1100	950	800	1100	800	950	950	1100	950	950	950	950
Product Size (in)	1/2	3/8	3/8	3/8	1/2	1/2	1/2	1/2	1/2	1/4	1/8	1/2	1/4

[a] N/A - Not applicable.

[b] TOB - Top of bed.

[c] Assuming an average bulk density of 35 lb/ft³ for material within the bed and a nominal bed volume of 1.0 ft³.

[d] ρ_s - nominal particle density.

TABLE 3

North Dakota Lignite Material Balance

Run No.:	0292	0392			0492	0592		0692		0892		0992	1092
Test No.:	N/A	1	2	3	N/A	1	2*	1	2	1	2	N/A	N/A
Run Duration, min	84.0	197.8	197.8	197.8	59.8	135.0	135.0	140.1	140.1	125.2	125.2	89.5	61.6
Test Duration, min	60.0	60.0	60.0	60.0	56.0	71.8	60.0	69.6	60.0	60.4	50.0	84.7	56.9
Coal Fed per Run, lb	164.0	400	400	400	182	448	448	544	544	362	362	252	186
Average Feed Rate, lb/hr	117.1	121.3	121.3	121.3	182.8	199.1	199.1	233.0	233.0	173.5	173.5	168.9	181.2
Product Char Collected, lb	14	30	34	45	51.5	89.8	95	85.5	47	38	43	51	47
Drip Leg Fines Collected, lb	25	23	18	14.5	20.5	22.5	19.7	46.5	43.0	35.5	17	77	23
Product Moisture Content, %	0.2	0	0	0	0	0	0	0	0	0	0	0	0
Product Volatile Content, %	20.95	19.24	22.16	27.64	18.70	28.80	23.98	23.00	17.57	22.84	25.47	23.18	24.55
Avg. Moisture Loss, lb	22.3	23.4	23.4	23.4	32.8	45.9	38.3	52.0	44.9	33.6	27.8	45.9	33.1
Avg. Volatile Matter Loss, lb	26.6	29.7	26.1	19.5	42.6	35.5	39.2	55.9	60.9	36.4	26.4	48.9	32.9
Avg. Moisture Loss, %	98.96	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00
Avg. Volatile Matter Loss, %	52.05	55.96	49.28	36.74	57.20	34.08	45.11	47.36	59.78	47.72	41.70	46.94	43.81
Total Accountable, lb	88.0	106.0	101.5	102.3	147.5	193.7	192.3	239.9	195.7	143.6	114.2	222.8	136.0
Closure, %	75.1	87.4	83.6	84.3	86.4	81.3	96.6	88.8	84.0	82.2	78.9	93.5	79.1
Uncollected Fines (calc.), lb	29.2	15.3	19.9	19.0	23.1	44.7	6.8	30.3	37.3	31.2	30.4	15.6	35.9
Total Fines, lb	54.2	38.3	37.9	33.5	43.6	67.2	26.5	76.8	80.3	66.7	47.4	92.6	58.9
Product Char d_{90}, micron	5025	4989	4888	6632	4759	4653	5279	4208	4601	4945	4761	4987	4187
Product Char d_{50}, micron	1949	2107	2040	2457	1919	1777	2090	1690	1913	2136	1881	1962	1662
Product Char d_{10}, micron	788	790	784	744	806	751	859	776	841	978	640	894	801
Bed Material @ EOR, lb	10	N/A	N/A	8.5	9	N/A	16.5	N/A	?	N/A	?	?	?
Char/Feed Ratio, %	12.0	24.7	28.0	37.1	30.2	37.7	47.7	31.6	20.2	21.7	29.7	21.4	27.4
Drip Leg Fines/Feed Ratio, %	21.3	19.0	14.8	12.0	12.0	9.4	9.9	17.2	18.5	20.3	11.8	32.3	13.4
Uncoll. Fines/Feed Ratio, %	24.9	12.6	16.4	15.7	13.6	18.7	3.4	11.2	16.0	17.8	21.1	6.5	20.9
Total Fines/Feed Ratio, %	76.0	96.7	97.0	106.6	80.9	71.8	96.2	64.5	56.2	68.8	84.3	60.6	73.4

* The "Product Char Collected" value for Run BS1-0592, Test 2 is a best-guess estimate.

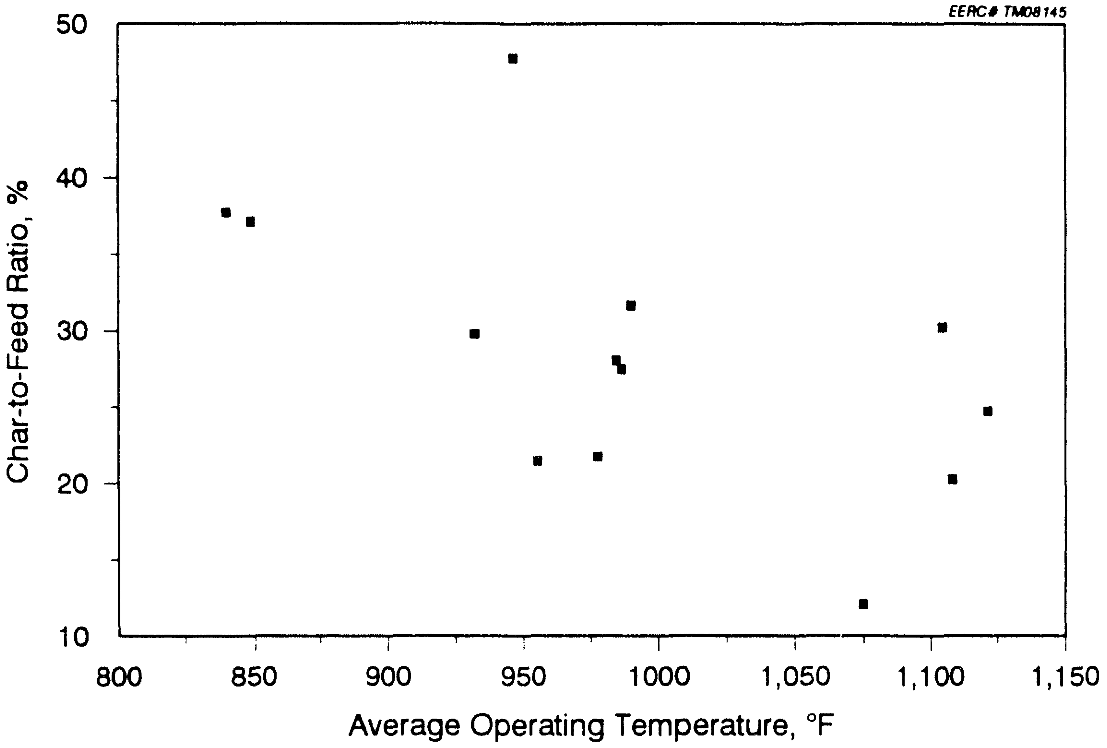


Figure 2. The ratio of char to coal feed as a function of operating temperature for the North Dakota lignite devolatilization testing.

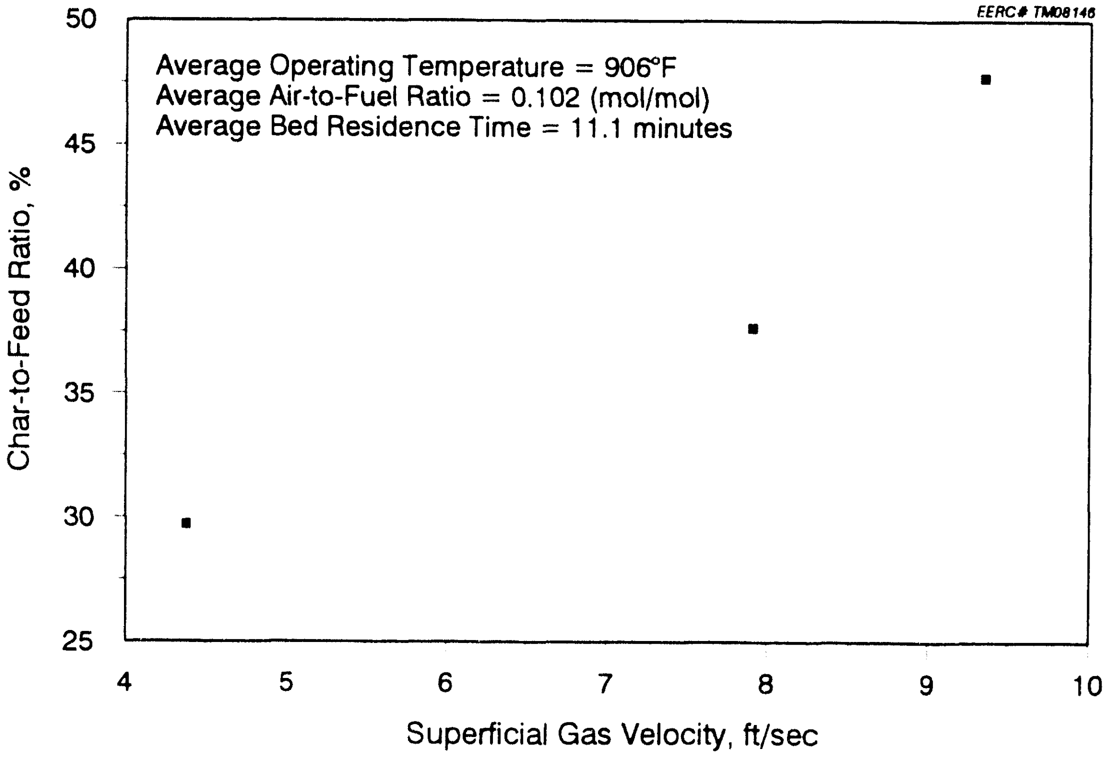


Figure 3. The ratio of char produced to coal fed as a function of superficial gas velocity for the North Dakota lignite devolatilization testing.

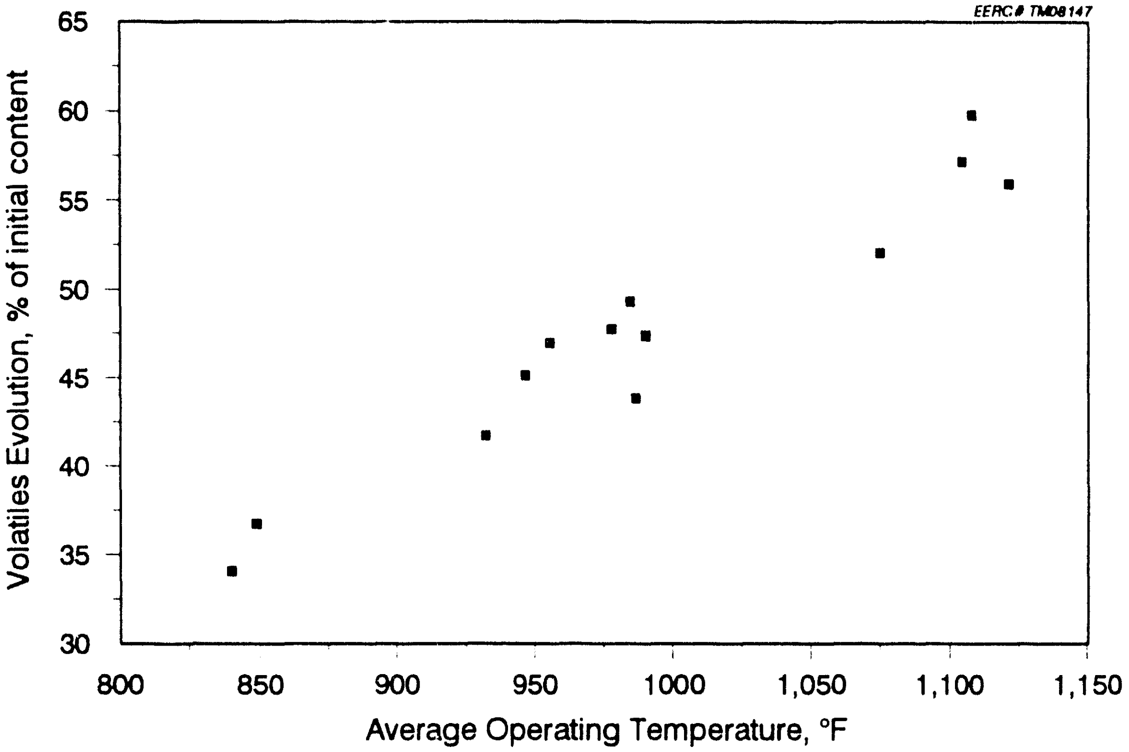


Figure 4. Volatile matter evolution as a function of operating temperature for the North Dakota lignite devolatilization testing.

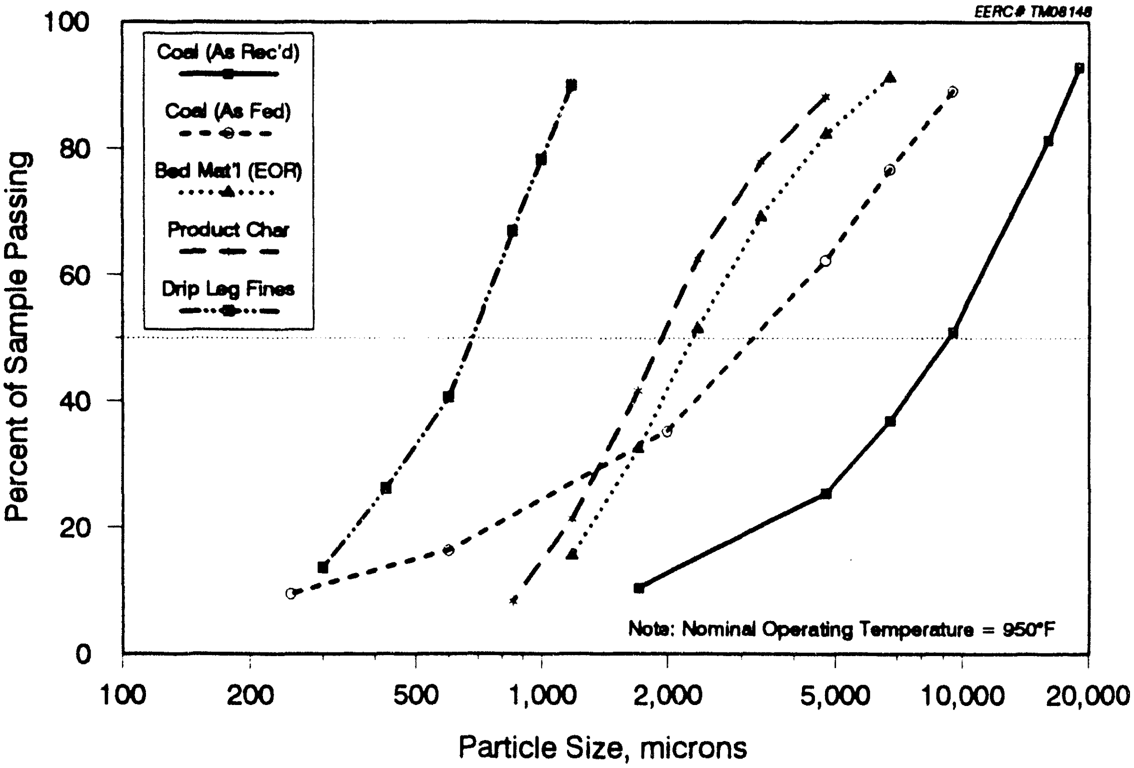


Figure 5. Particle-size distributions for various solid samples taken during North Dakota lignite Test CPC-BS1-0992.

Dakota lignite tests, the Montana subbituminous coal was tested at temperatures ranging from about 850° to 1075°F. Three additional simple drying tests were run at the conclusion of the devolatilization tests to determine the level of beneficiation possible with this type of coal at relatively low temperatures. The operating temperatures for these tests ranged from about 250° to 520°F.

Bed residence times and air-to-fuel ratios similar to those used for the lignite testing were employed for the subbituminous coal tests and ranged from 8 minutes to 19 minutes and between 0.10 and 0.20, respectively. Calculated superficial gas velocities for the top of the expanded bed were generally higher for the subbituminous coal tests because of the larger feed-coal size and ranged from 6.9 ft/sec to about 11.4 ft/sec. Table 5 shows that, over the range of conditions tested, 100% of the moisture was driven out of the product char for each test, with the exception of the simple drying tests, i.e., those tests under the run number designation CPC-MT1-1492 (89% to 98% moisture removal). The removal of volatile matter ranged from less than 1% to about 14% for the drying tests, and from 40% up to about 57% for devolatilization testing. The ratio of the amount of char produced to the amount of coal fed ranged between 21% and about 40% for the devolatilization mode of testing, and 33% to 52% during the drying tests.

A more complete analysis of the data is currently underway, and a comprehensive final report covering the results of the pretreatment cell testing is in preparation.

TABLE 4

Montana Subbituminous Coal[a] Average Output Data

Time-Weighted Averages

Run No.	Test No.	Average Feed Rate (lb/hr)	TOB[b] Avg. Temp. (°F)	TOB Flow (acfm)	TOB SGV (ft/sec)	Fuel Input (mol/hr)	Air Input (mol/hr)	A/F Ratio (mol/mol)	Oxygen Input (%)	Avg.[c] Residence Time (min)
CPC-MT1-1192	1	204.6	935	195.48	9.31	12.0	1.55	0.129	14.2	10.26
	2	204.6	947	157.40	7.50	12.0	1.20	0.100	13.8	10.26
CPC-MT1-1292	1	180.8	968	238.70	11.37	10.6	1.74	0.164	13.3	11.62
	2	180.8	856	175.50	8.36	10.6	1.08	0.102	10.4	11.62
CPC-MT1-1392	1	211.5	1076	212.66	10.13	12.4	1.54	0.124	14.3	9.93
	2	111.1	972	207.82	9.90	6.5	1.23	0.189	10.8	18.90
	3	111.1	911	215.75	10.27	6.5	1.30	0.199	10.6	18.90
CPC-MT1-1492	1	130.1	518	160.63	7.65	7.6	1.25	0.163	9.9	16.14
	2	130.1	299	145.11	6.91	7.6	1.03	0.135	6.9	16.14
	3	254.7	254	157.49	7.50	14.9	2.11	0.141	12.3	8.24

NOMINAL CONDITIONS:

Run No.	Test No.	Residence Time (min)	Feed Rate (lb/hr)	TOB Avg. Temp. (°F)	Product Size (in)	Comments
CPC-MT1-1192	1	11.20	187.5	950	3/8	Preheaters on.
	2	11.20	187.5	950	1/4	Preheat at 300°F only.
CPC-MT1-1292	1	11.20	187.5	950	1/2	Fluidization problems.
	2	11.20	187.5	800	1/4	Lost fluidization @ bottom of bed.
CPC-MT1-1392	1	11.20	187.5	1100	3/8	
	2	16.80	125	950	3/8	Preheat at 300°F only.
	3	16.80	125	950	3/8	Preheaters up to 800°F.
CPC-MT1-1492	1	16.80	125	N/A	3/8	Relatively low-temperature drying test.
	2	16.80	125	N/A	3/8	Repeat of MT1-1492 Test 1 with lower bed temperatures.
	3	8.40	250	N/A	3/8	Drying test at higher feed rate.

[a] All feed coal was sized to +1/4" prior to testing.

[b] TOB - Top of bed.

[c] Assuming an average bulk density of 35 lb/ft^3 for material within the bed and a nominal bed volume of 1.0 ft^3.

TABLE 5

Montana Subbituminous Coal Material Balance

Run No.: Test No.:	MT1-1192 1	MT1-1192 2	MT1-1292 1	MT1-1292 2	MT1-1392 1	MT1-1392 2	MT1-1392 3	MT1-1492 1	MT1-1492 2	MT1-1492 3
Run Duration, min	143.7	143.7	179.9	179.9	73.2	143.7	143.7	105.2	105.2	38.2
Test Duration, min	61.7	64.8	63.6	70.7	70.0	63.3	72.2	69.2	36.0	38.2
Coal Fed per Run, lb	490	490	542	542	258	266	266	228	228	162
Avg. Feed Rate, lb/hr	204.6	204.6	180.8	180.8	211.5	111.1	111.1	130.1	130.1	254.7
Product Char Collected, lb	62	75	51	72	52.5	25	54	71	26	84
Drip Leg Fines Collected, lb	48	20.5	51.5	13.5	39	22	24.0	5.1	3.5	15.0
Product Moisture Content, %	0	0	0.	0	0	0	0	0.5	1.7	2.7
Prod. Volatile Content, mf, %	23.31	22.11	24.48	18.69	21.47	26.13	21.68	37.25	42.83	41.00
Avg. Moisture Loss, lb	52.5	55.2	47.8	53.1	61.6	29.2	33.3	36.7	18.1	36.0
Avg. Volatile Matter Loss, lb	42.0	46.8	36.0	52.3	53.8	20.1	28.8	9.0	0.3	3.7
Avg. Moisture Loss, %	100.00	100.00	100.00	100.00	100.00	100.00	100.00	98.00	93.19	89.18
Avg. Volatile Matter Loss, %	46.12	48.89	43.41	56.80	50.37	39.60	49.88	13.89	0.99	5.22
Total Accountable, lb	204.4	197.4	186.2	190.9	206.8	96.3	140.2	121.8	48.0	138.7
Total Accountable w/ Bed Mat'l, lb					219.8			142.8	74.5	164.7
Closure, %	97.2	89.3	97.3	89.7	83.8	82.2	104.9	81.2	61.5	85.6
Closure w/ Bed Material, %					89.1			95.2	95.4	101.7
Uncollected Fines (calc.), lb	5.9	23.7	5.2	22.0	39.9	20.9	-6.5	28.2	30.1	23.3
Total Fines, lb	53.9	44.2	56.7	35.5	78.9	42.9	17.5	33.3	33.6	38.3
Bed Material @ EOR, lb	N/A	28.5	N/A	20.9	13	N/A	13	21	26.5	26
Char/Feed Ratio, %	29.5	33.9	26.6	33.8	21.3	21.3	40.4	47.3	33.3	51.9
Drip Leg Fines/Feed Ratio, %	22.8	9.3	26.9	6.3	15.8	18.8	18.0	3.4	4.5	9.3
Uncoll. Fines/Feed Ratio, %	2.8	10.7	2.7	10.3	16.2	17.8	-4.9	18.8	38.5	14.4
Total Fines/Feed Ratio, %	25.6	20.0	29.6	16.7	32.0	36.6	13.1	22.2	43.0	23.6

FORM EIA 459F
(10/80)

U.S. DEPARTMENT OF ENERGY

FEDERAL ASSISTANCE MANAGEMENT SUMMARY REPORT

FORM APPROVED
OMB NO 1900-0127
Page 1 of 2

1. Program/Project Identification No. DE-FC21-86MC10637
2. Program/Project Title Atmospheric Fluidized-Bed Combustion (4.1)
3. Reporting Period 3-1-92 though 6-30-92
4. Name and Address Energy and Environmental Research Center
University of North Dakota
Box 8213, University Station, Grand Forks, ND 58202 (701) 777-5000
5. Program/Project Start Date 4-1-86
6. Completion Date 9-30-92

7. FY 91/92
8. Months or Quarters Quarters
1991: JUL AUG SEP OCT NOV DEC
1992: JAN FEB MAR APR MAY JUN

9. Cost Status a. Dollars Expressed In Thousands b. Dollar Scale

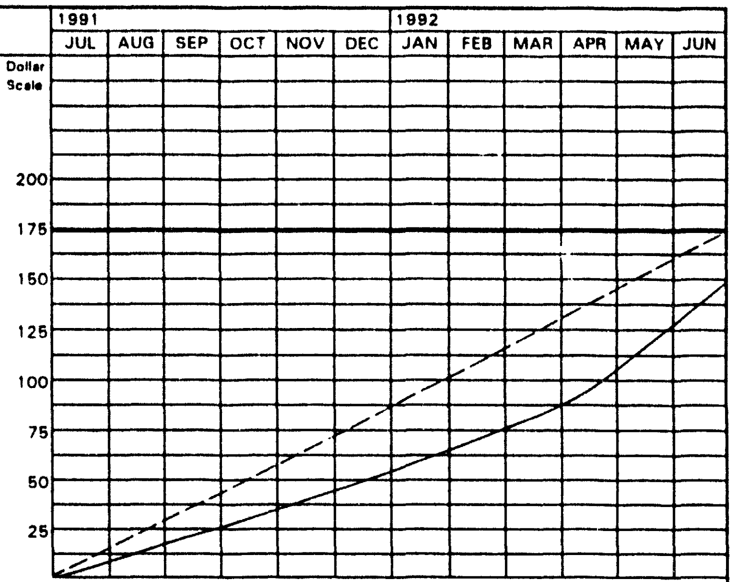
10 Cost Chart

Fund Source		Quarter 1st	2nd	3rd	4th	Cum. to Date	Tot. Plan
DOE	P	44	44	44	43	175	175
	A	26	29	31	63	149	
	P						
	A						
	P						
	A						
	P						
	A						
Total P		44	44	44	43	175	175
Total A		26	29	31	63	149	
Variance		18	15	13	(20)	26	

P = Planned A = Actual

Total Planned Costs for Program/Project

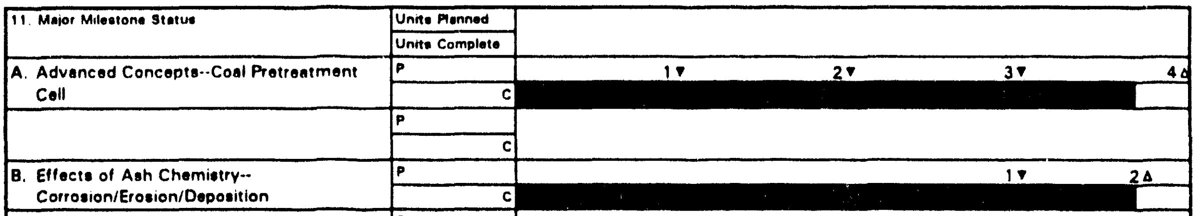
$175



c. Cumulative Accrued Costs

	JUL	AUG	SEP	OCT	NOV	DEC	JAN	FEB	MAR	APR	MAY	JUN
Planned			44			88			132			175
Actual			26			55			86			149
Variance			18			33			46			26

11. Major Milestone Status



12. Remarks

Due to EERC fiscal year end, the June books do not close until July 25, 1992. Costs posted through July 8 have been included.

13. Signature of Recipient and Date Ann K Henderson for Michael D. Mann

14. Signature of DOE Reviewing Representative and Date

FORM E1A-459E
(10/80)

U.S. DEPARTMENT OF ENERGY
FEDERAL ASSISTANCE MANAGEMENT SUMMARY REPORT

FORM APPROVED
OMB NO. 1900 0127

1. Program/Project Identification No. DE-FC21-86MC10637	2. Program/Project Title Atmospheric Fluidized-Bed Combustion (4.1)	3. Reporting Period 3-1-92 through 6-30-92
4. Name and Address	Energy and Environmental Research Center University of North Dakota Box 8213, University Station Grand Forks, ND 58202 (701) 777-5000	5. Program/Project Start Date 4-1-86 6. Completion Date 9-30-92

Milestone ID. No.	Description	Planned Completion Date	Actual Completion Date	Comments
Task A	Advanced Concepts--Coal Pretreatment Cell:			
a.1	Complete test matrix on lignite coal	9-30-91	12-31-91	
a.2	Complete test matrix on subbituminous coal	12-31-91	5-30-92	
a.3	Complete test matrix on bituminous coal	3-31-92	3-31-92	
a.4	Proof-of-concept with design information for full-scale plant	6-30-92		
Task B	Effects of Ash Chemistry--Corrosion/Erosion/Deposition:			
b.1	Obtain and analyze materials	3-31-92	3-31-92	
b.2	Correlate data	6-30-92	*	

* No special analysis were requested.

4.2 Beneficiation of Low-Rank Coals

BENEFICIATION OF LOW-RANK COALS

Semiannual Technical Progress Report
for the Period January 1, 1992 - June 30, 1992

by

Mark A. Musich, Research Supervisor
Chris M. Anderson, Research Engineer
Raymond A. DeWall, Research Chemist
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for

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July 1992

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

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BENEFICIATION OF LOW-RANK COALS

1.0 GOALS AND OBJECTIVES

The overall objectives of the Low-Rank Coal Beneficiation Project are to study methods to reduce the inorganic content and increase the heating value of low-rank coal (LRC) to produce high-quality dry fuels and/or coal/water fuels (CWF). The Energy and Environmental Research Center (EERC) has previously demonstrated with a 600-lb/hr continuous pilot plant that hot-water drying (HWD) of lignite or subbituminous coal can produce high-energy content, dry or slurry fuels. Integration of continuous chemical and physical cleaning processes with HWD can produce low-ash, low-moisture CWF suitable for advanced combustion systems.

Presently the LRC Beneficiation Project is continuing the development, on the bench scale, of a LRC agglomeration process which combines a chemical cleaning step with an aggregation step using coal-derived oil. Previous statistical testing has studied the effects of major variables on agglomerate qualities. The variables studied included coal particle size, acid concentration, acid mixing speed and acid contact time, and oil mixing speed and oil mixing time. The process has been proven effective with all coals tested including North Dakota and Texas lignites, and Alaska, Wyoming, and Montana subbituminous coals. A limited amount of testing had been performed on deoiling techniques to recover agglomerating oil for recycle or resale as an upgraded, light oil fraction.

The proposed third-year research efforts for oil agglomeration (Task A) as described in the Sixth Annual Program Plan were:

1. <u>Agglomeration with Carrier Solvent</u> - Testing will determine if LRCs can be agglomerated when the agglomerating oil is dissolved in a carrier solvent. The carrier solvent ideally would aid in dispersion of the oil on the coal and reduce the quantity of oil for agglomeration. Reducing the quantity of oil could eliminate the deoiling step. The carrier solvent would be recoverable from the wastewater and agglomerates for recycle. Carrier solvents will be tested that are compatible with both coal- and petroleum-derived oils.

2. <u>Recycle and Makeup Requirements</u> - Analytical techniques will be tested to determine the oil content of agglomerates and wastewater. Similarly, analytical techniques will be developed to determine the residual acid concentration in the wastewater. Wastewater will be recycled to determine if the presence of soluble cations will deleteriously affect the ash content of the acid-cleaned or agglomerated coal. The determination of oil and acid consumption will allow estimation of preliminary economics and equipment sizing for process scaleup.

3. <u>Process Modifications for Agglomerate Formation and Dewatering</u> - Agglomeration aids will be tested which can provide the ionic strength required for agglomeration. The agglomeration aids could eliminate or reduce acid use when inorganic reduction is not

essential but dewatering is still desired. Also, briquetting will be tested as a method of compacting the oil/coal mixture when agglomeration is not successful. A pellet or briquette could be made having the characteristics of successfully agglomerated coal.

4. <u>Mass Agglomerate Formation and Deoiling Techniques</u> - The batch agglomeration process will be tested on a slightly larger scale in order to minimize the agglomeration experiments required to produce material for deoiling and product stability testing. Techniques will be further investigated to determine the appropriate conditions and method for agglomerate deoiling.

5. <u>Development of Agglomerate Stability Analysis Techniques</u> - Analysis techniques will be tested to determine chemical and physical stability. Stability parameters of importance are crushing strength, durability, abrasion resistance, water immersion resistance, and freeze/thaw resistance. Chemical stability will address resistance to oxidation and spontaneous combustion.

Additional beneficiation techniques described in Task B include froth floatation and colloidal coal cleaning. Techniques will be tested which aid in increasing the floatability of LRCs, and the chemical solubilization of coal humates will be tested as a method of producing low inorganic-content fuels.

2.0 ACCOMPLISHMENTS

Progress was made toward addressing Tasks A.2, A.3, and A.4 and Task B.1. Analytical techniques for determining the oil content of agglomerates and process water were evaluated by agglomerating a lignite and a subbituminous coal with crude phenol/tar oil and then performing oil, moisture, and solids balances. The analytical techniques include azeotropic distillation and Soxhlet extraction for determining agglomerate moisture and oil contents, respectively, and total carbon to determine the oil content of the process water. Several improvements were made to the agglomerate oil measurement technique and the resulting balance closures indicated that the analysis methods are not grossly in error.

Additional agglomerating oils were tested, with varying degrees of success, in the EERC agglomeration process. The oils tested included a commercial cresylic acid, liquefaction-derived oils, mild gasification-derived tars, and used motor oil. The ability of the oils to agglomerate easily at low-shear mixing conditions was qualitatively correlated against the polar and oxygen contents of the oils. Preliminary results indicate that the high polar and oxygen content oils are easily agglomerated at low-shear conditions while the low polar and oxygen content oils require high-shear mixing or surfactants to promote agglomerate formation. The determination of oxygen and polar content may progress into a screening method for determining candidate LRC agglomerating oils.

Several tests indicated that the acid precleaning step may be eliminated when inorganic reduction is not necessary. Alkali and alkaline earth metal salts successfully replaced the acids during agglomeration by, hypothetically, providing the necessary solution ionic strength. Carbonic acid also successfully promoted agglomeration at an ionic strength 10^{-3} to 10^{-4} of nitric, sulfuric, or hydrochloric acids. Persistence in testing paid off when the crude phenol/tar oil successfully agglomerated the Beulah lignite without a prior acid-cleaning step or the addition of acid or metal salt during the agglomeration step.

The EERC agglomeration process was scaled up from 50 g of coal per test to 200 g of coal per test to allow production of large quantities of agglomerates for deoiling and strength and stability tests. The process was scaled up further, resulting in the successful agglomeration of 2.5 kg of coal in a 5-gallon pail.

Agglomerate deoiling tests with a batch autoclave have resulted in inefficient oil removal and poor temperature control. Preliminary tests with a batch fluid-bed reactor have indicated that this system may be ideal for thermally deoiling agglomerates. The hydrodynamics of the fluid bed provide excellent temperature control using either an inert gas or steam as a deoiling/fluidizing medium. The fluidizing action was not severe enough to cause significant agglomerate degradation. Presently the system is limited by its small agglomerate capacity and slightly inefficient condensate-recovery system.

Additional tests conducted during the period of January through June 1992 included:

1. Removal of soluble oil from the process water by solvent extraction.
2. Removal of soluble oil from the process water by absorption on coal.
3. Durability testing of air-dried and oven-dried agglomerates.

3.0 INTRODUCTION

LRCs (lignitic, subbituminous, and brown) are characterized by a number of qualities which make them ideal for combustion and/or conversion processes. The inherent alkali content and high volatile-matter content of the LRCs make them ideally suited as a gasification feedstock. The low-sulfur content of some subbituminous coals makes them compliance coals for power generation. Additionally, most LRCs are low in ash and are inexpensively strip-mined. Even though these LRCs are plentiful in southern coast states, between the Mississippi River and the Rocky Mountains, and, most certainly, in Alaska, their marketability has been restricted by their high moisture and, consequently, low energy contents. Additionally, the fouling problems associated with the high sodium content have inhibited the use of some LRCs. Considerable effort has been put forth to develop processes which increase energy content and reduce mineral content, yet produce a competitively priced feedstock from LRCs.

Although oil agglomeration of bituminous coals has been conducted for many years, the extension of these techniques to LRCs had achieved limited success, until recently. The primary reason for the inability to agglomerate LRCs lay in the incompatibility of the agglomerating oil and the coal surface.

The low moisture and low exchangeable-ion contents plus relatively few oxygen functionalities make bituminous coals compatible with nonpolar oils. The converse is true for the LRCs in that the high moisture content, oxygen functionalities, and cations make nonpolar oils unsuitable for agglomeration. Polar, coal-derived oils, however, can be used to agglomerate LRCs, and the polar oils are aided by surface modification and high ionic-strength aqueous treatment. Surface modification consists of removing the cations which contribute to the hydrophilicity and consequent oleophobicity of the coal. The cation (ash) level of the coal can be reduced by aqueous phase ionic exchange. Simultaneously, the high ionic strength of the low-pH solution is theorized to establish a concentration gradient between the water in the coal capillaries and the bulk water of the aqueous solution. This concentration gradient allows the polar oils, which are more compatible with the oxygen functionalities, to displace the capillary water. The agglomerating oil also thinly covers the carbonaceous material on the coal surface, and, as a result of mixing, the oil-covered surfaces come into contact and aggregate together. The aggregate size can be controlled by the speed of mixing, the amount of agglomerating oil, and the agglomeration time.

The EERC has demonstrated that low-ash fuel can be produced by a combination of physical cleaning followed by chemical cleaning of fine coal. Laboratory testing has reduced LRC ash contents to less than 1 wt% using float/sink techniques followed by dilute acid leaching (1). LRCs with ash contents as low as 2 wt% have been produced on the pilot scale using dense-media separation followed by continuous acid leaching (2,3). Each method has also shown that the sodium content can be significantly reduced. These processes, however, produce a clean coal which would be restricted to utilization at a site adjacent to the preparation facility. The hazards associated with finely ground coal, such as dustiness and oxidation, would prohibit long-range transport of this form of beneficiated coal. However, the EERC has demonstrated that a process of dilute acid leaching followed by oil agglomeration accomplishes similar ash reductions and, equally important, produces a low-moisture, high heating-value LRC with the potential to be easily transported and resistant to weathering.

4.0 AGGLOMERATION DEVELOPMENT AND TESTING

4.1 Agglomeration Experimental Procedure

The EERC agglomeration procedure is divided into two steps: acid cleaning followed by oil agglomeration. The same sample cell is used throughout the process. The coal is pulverized to a top size of 30 mesh prior to agglomeration, with the agglomerates being recovered on a 30-mesh screen. All the steps in the process are carried out at room temperature and atmospheric conditions. Table 1 contains the experimental parameters needed to achieve agglomeration. The acid-cleaning (leaching) step consists of mixing a quantity of coal with a quantity of dilute acid solution, typically using 1 part coal to 2 parts of a dilute acid solution. The coal/acid solution is stirred for 1 to 30 minutes, typically less than 5 minutes, at a speed of 5500-6000 rpm in a high-shear mixer.

TABLE 1

Oil Agglomeration Experimental Parameters

Coal Particle Size	less than 30 mesh by 0
Acid Concentration	6.0, 3.0, 1.5, and 0.75 wt%
Acid-Cleaning Speed	5500 - 6000 rpm
Acid-Contact Time	1 - 30 minutes
Oil-Mixing Speed	600 - 800 rpm
Oil-Mixing Time	5 - 10 minutes

Agglomeration consists of adding a quantity of agglomerating oil, typically equivalent to the weight of the coal, to the coal/acid solution. The sample is mixed using a low-speed T-bar mixer until agglomeration occurs. Agglomeration is normally indicated by a change in solution turbidity. The raw agglomerates are recovered on the 30-mesh screen, and the coal that passes through the screen is recovered by filtration using a Buchner funnel and filter paper. The agglomerates are washed, and the samples are allowed to air-dry overnight prior to weighing and analysis. Figure 1 is a representation of the agglomeration process.

The raw coal moisture and solids contents are determined according to ASTM D3173. The agglomerating oil moisture content is determined by Karl-Fischer analysis and ash content is determined by proximate analysis according to ASTM D3174. The agglomerating oil carbon content is determined from ultimate analysis by ASTM D3176. The process water is analyzed by Standard Method No. 505A to determine total carbon content, in mg/L (4). The total soluble oil content in the process water is calculated as follows:

$$\text{Total Soluble Oil Content (mg/L)} = \frac{\text{Process Water Total Carbon Content (mg/L)}}{\text{Oil Carbon Content (wt\%)} \times 100}$$

The composition (i.e. wt% carbon) of the soluble oil is assumed to be equivalent to that of the agglomerating oil composition.

The moisture contents of raw, air-dried, or oven-dried agglomerates are determined by azeotropic distillation with xylene, and oil contents are determined by Soxhlet extraction with methylene chloride (5). The tailings are analyzed using the agglomerate analysis techniques if sufficient quantities are produced.

Oil, water, and solids balances are calculated using the weights and compositions of the raw agglomerates and raw tailings. The raw agglomerate moisture is always directly determined by azeotropic distillation. However, the oil content of the raw agglomerates is either directly determined by Soxhlet extraction or calculated from analysis of the air- or oven-dried agglomerates. Consequently, four separate methods can be used to determine the raw agglomerate oil content. Method 1 determines the raw agglomerate oil

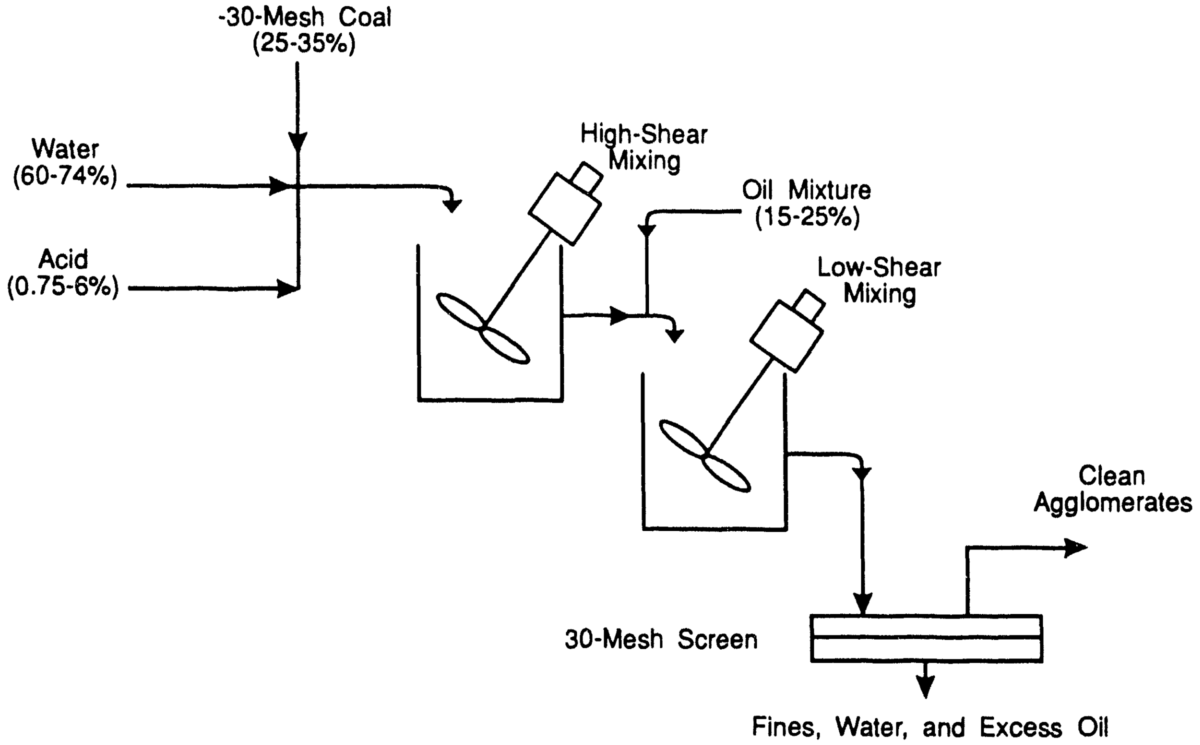

Figure 1. Schematic diagram of the EERC oil agglomeration process.

content directly by Soxhlet extraction. Method 2 calculates the raw agglomerate oil content based on the air-dried agglomerate moisture and oil contents. Method 3 calculates the raw agglomerate oil content based on the oven-dried agglomerate moisture and oil contents. Method 4 calculates the raw agglomerate oil content based on the air- and oven-dried agglomerate moisture and oil contents. The analysis sequence for determining the moisture and oil contents of the raw agglomerates is illustrated in Appendix A.

The oils tested in the agglomeration process are each analyzed to determine oxygen and polar oil content. The oxygen and polar contents are correlated with the effectiveness of the oil to promote agglomeration.

The agglomerating oil oxygen content is determined by difference from ultimate analysis by ASTM D3176. The polar oil content is determined by open column chromatography using silica gel (-28 x +200-mesh) activated by oven-drying at 250°C for 2 hours. The linear and small aliphatics are eluted from the oil sample using pentane; isooctane was used to elute branched and large aliphatics; methylene chloride was used to elute aromatics; methanol was used to elute the polars. The extracting solvents are evaporated to recover the oil fractions. The total mass of recovered oil is determined for each test to assess the efficiency of extraction and to calculate a mass balance closure.

4.2 Results and Discussion

4.2.1 Agglomeration Process Scale-up

The amount of coal that can be agglomerated using the EERC oil agglomeration process was increased from 50 to 200 g per test. This was done in order to produce enough agglomerates to satisfy requirements for analytical testing techniques such as oil content measurements, stability testing, and deoiling for oil recovery. The major changes that were necessary in order to accomplish scale-up were increases in vessel size and tip-to-tip width of the T-bar mixer. A total of 14 tests were conducted using crude phenol/tar oil agglomerant and 200 g of coal. The first two tests, using 200 mL of oil, failed to form agglomerates, presumably because of an excess of agglomerant. Successful agglomeration was achieved after reducing the agglomerant quantity from 200 mL to 180 or 190 mL. Additional operational changes were made to reduce the amount of tailings (-30-mesh agglomerates). Mixing speed (shaft rpm) reduction was sufficient to allow the small agglomerates to grow larger than 30 mesh. At comparable shaft speeds the larger T-bar mixer had a higher tangential tip speed that was presumed to inhibit agglomerate growth. Consequently, the tailings produced from 180 mL of oil and 200 g of coal were proportional to using 50 g of coal and 50 mL of oil.

Following the success of this scale-up, two agglomeration tests were performed in which the coal was agglomerated in a 20-liter pail. A T-bar mixer was fabricated using the same tip-to-tip ratio as that employed in the 200-gram mixer. The first test was performed using approximately 2700 g of coal and crude phenol/tar oil and 5400 g of 0.63 M nitric acid solution. A 1/4-horsepower mixer was mounted on a special pail lid to drive the T-bar mixer. The test produced about 450 g of +30-mesh, air-dried agglomerates; however, a vast majority of the coal was an oily mass. Upon termination of the experiment, it was observed that a large quantity of coal was on the bottom of the pail, presumably a consequence of the T-bar mixer not extending far enough into the pail to allow thorough mixing. It was also concluded that the tip speed was slightly high; therefore, the oil and coal contact was too fast to promote agglomeration. Another experiment was attempted following the modifications to the mixer. This experiment used approximately 1500 g of coal and crude phenol/tar oil and 3000 g of 0.63 M nitric acid solution. A smaller quantity was used because the bucket was too full with the earlier experiment. The shaft speed was reduced from the previous experiment. This test very successfully produced approximately 2000 g of agglomerates. A small quantity of agglomerates were lost during washing on a 30-mesh screen. To perform more agglomeration testing in the 20-liter pail, it will be necessary to utilize much more efficient methods to recover the agglomerates because a considerable amount of water was necessary to wash the fines from the agglomerates.

4.2.2 Testing of Additional Agglomerating Oils

Several additional coal- and petroleum-derived oils were tested as agglomerants in the EERC oil agglomeration process. These oils included commercially produced cresylic acid No. 83 black (CA83BL), generically known as alkylated phenols, from the Merichem Company of Houston, Texas; two liquefaction-derived oils from run #263J at Wilsonville, Alabama; two mild gasification-derived tars from the EERC mild gasification project; and used 10W-40 crankcase oil.

4.2.2.1 Cresylic Acid

Successful agglomeration using cresylic acid was only possible after the oil content was reduced from 50 mL (standard with crude phenol/tar oil) to as low as 30 mL per 50 g of coal. At oil volumes above 30 to 35 mL, an oily mass or clumped agglomerates were formed. However, to achieve agglomeration at these lower agglomerant volumes, low-shear mixing had to be increased from the traditional 2 to 5 minutes to as long as 7 to 15 minutes. Successful agglomeration also occurred when the majority of CA83BL was added during high-shear mixing. This resulted in almost immediate agglomerate formation, and slight agglomerate growth was achieved with low-shear mixing and slight oil addition. The high-shear mixing resulted in almost no tailings production although the +30-mesh agglomerates were smaller than typically formed agglomerates. Agglomerates produced from this oil had physical characteristics similar to the agglomerates produced using crude phenol/tar oil.

4.2.2.2 Liquefaction Oils

The liquefaction oils numbered V161 and V178 were produced at the liquefaction plant by distilling the "raw liquefaction product." Both oils are classified as light oils or naphthas by Wilsonville. The V161 is 45.9 wt% of the product, a light fraction with over 97 wt% boiling under 550°F. The V178 is 54.1 wt% of the product, a heavier fraction with over 85 wt% boiling in the range of 550°F to +850°F.

Five agglomeration tests were conducted with V161, with four tests being considered successful. The single failure was based on the traditional agglomeration methods employed when using crude phenol/tar oil. This oil could agglomerate the coal at volumes as low as 30 mL when a surfactant such as Triton X-100 (2 g/50 g coal) or crude phenol (10 g/50 g coal) was used. Also, a single test produced the results that, during high-shear mixing, the oil requirements could be reduced.

The agglomeration tests performed with V178 oil used a surfactant to help promote agglomeration. The surfactant was chosen based on the results with the V161 oil. One test used 2 g Triton X-100 to 50 g coal, and the other used 5 g crude phenol to 50 g coal. The volume of V178 oil ranged from 25 to 30 mL with no tailings production during either test. The agglomerates produced from these two oils appeared to be very brittle after air-drying, compared to the crude phenol/tar oil agglomerates.

4.2.2.3 Mild Gasification Tar

Two mild gasification tars were used for agglomeration testing. The first tar tested was from the knockout pots, and the other one was from the back cyclone of the mild gasification unit. The knockout pot tar appeared to contain a considerable amount of very fine coal that was probably blown over during the fluidization process. Neither of the tars mixed well with water or flow at room temperature so they were thinned in a variety of solvents. The best solvent for thinning the knockout pot tar was butanol; for the back cyclone tar, it was pentane. A 60:40 wt% knockout pot tar/butanol mixture did not form agglomerates using normal low-shear mixing methods. Similarly, an 83:17 wt% back cyclone tar/pentane mixture did not form agglomerates under typical agglomerating conditions. However, after mixing the tar/pentane mixture under high-shear conditions, agglomerates were formed using 36 g of the mixture per 50 g coal. The sample was placed under the low-shear mixer to promote agglomerate growth. These agglomerates were extremely small compared to the normal agglomerates, probably due to the high-shear mixing that was employed.

4.2.2.4 Used Crankcase Oil

Using normal agglomerating conditions with a sample of 10W-40 used crankcase oil was unsuccessful at producing agglomerates. However, high-shear mixing of this sample caused a distinct phase separation. Phase separation is an indication that the coal is preferentially wetted by oil and no longer dispersed in the water phase. Since there is no oil dispersed in the water phase, oil recovery could be easy if agglomeration were performed with crankcase oil.

The high-shear mixing results presented above were employed using incremental addition of oil until phase separation was observed. Following the phase separation, the oil/water/coal was mixed with the low-shear mixer until agglomeration was complete. The test was repeated by adding the oil in a single dose during high-shear mixing. Once again agglomeration was successful with only 13 mL of oil required to agglomerate 50 g of coal. This compares with approximately 45 to 50 mL of crude phenol/tar oil to agglomerate 50 g of Beulah lignite.

4.2.3 Ionic Strength Modification Testing

It was hypothesized that other sources of ionic strength may be just as effective as dissociating acids, i.e., nitric acid. Investigations were done to verify this by using potassium nitrate, calcium acetate, and carbonic acid as sources of ionic strength.

A 1 M solution of potassium nitrate was used for a source of ionic strength in the agglomeration process. The initial solution was made by mixing potassium hydroxide with nitric acid. The solution pH was adjusted to 4.0, much higher than normally achieved during agglomeration. Agglomeration was performed using the normal agglomerating conditions. The experiment was successful with only slight bridging of agglomerates. Several more experiments were performed using another solution of potassium nitrate that was made using potassium nitrate crystals. The pH of this 1 M solution was 6.7. Agglomeration was very successful with this solution using 45 mL of oil. The success of potassium nitrate led to the use of calcium acetate as an ionic solution. This test was also successful with the conditions employed using potassium nitrate in the agglomeration process.

Carbonic acid was used as a source of ionic strength because of the low cost of producing it compared to nitric acid. The carbonic acid source for this testing was carbonated sparkling water. Normally the carbonic acid could be produced by bubbling carbon dioxide in water. The original pH of the sparkling water was 4.6, and it increased to 5.9 after acid cleaning. Agglomeration was carried out in the normal manner with very few fines produced. This test shows that inexpensively produced carbonic acid could be used for agglomeration when little, if any, inorganic reduction is required.

4.2.4 Agglomeration Without Coal-Surface Modification

Several tests were performed without acid cleaning or surface modification of the coal. These tests were done using crude phenol/tar oil as the agglomerant. The first test performed with Beulah lignite was done using incremental oil addition until agglomeration was observed. This test required a quantity of 40 mL oil for agglomeration to occur. The agglomerates produced were very tiny. This successful test led to another test where the oil was added all at once in the normal manner. Agglomeration was very successful with very little fines production. Oil agglomeration conditions were optimized because testing was performed using 30-50 mL oil. At 30 mL, small agglomerates were formed, but they were much smaller than the required 30-mesh size. For 50 mL, the agglomerates formed were oil-bound and clumped together. Duplicate analysis using 40 mL oil produced the same results as the successful initial testing.

4.2.5 Characterization of Agglomerating Oils

The ultimate analysis results of the agglomerating oils are presented in Table 2, and the aliphatic, aromatic, and polar contents are presented in Table 3. The agglomerating oils tended to increase in oxygen content as the polar content increased, although the oil with the highest oxygen content did not have the highest polar content. Similarly, the oil with the lowest oxygen content did not have the lowest polar content. Preliminary review of the analyses suggests that there may be a correlation between the composition of the oil and its ability to promote agglomeration. The oils with the highest oxygen and polar contents tended to be the most suitable for agglomeration at low-shear conditions. Conversely, the oils with lower oxygen and polar contents required high-shear mixing or the addition of surfactants to promote agglomeration.

The preliminary results of oxygen- and polar-content analyses suggest these techniques can be used for screening candidate oils for LRC agglomeration. However, because of the length of time required to generate these analyses, the applicability of an oil for agglomeration may be more quickly determined by performing agglomeration experiments. Consequently, determination of oxygen and polar contents may be used to substantiate why a particular oil did not work at low-shear conditions, but instead required high-shear mixing or surfactants.

4.2.6 Moisture, Oil, and Solids Balances

Three agglomeration balance tests were performed, two with Beulah lignite and one with Spring Creek subbituminous coal. The balance tests were similar to the one discussed in the July-December 1991 semiannual when m-cresol was used as the agglomerating oil (6). The balances differed primarily in the calculation method for the raw agglomerate oil content.

The agglomerating oil used in each test was a 94/6 vol% blend of coal-derived crude phenol and tar oil, each obtained from the Dakota Gasification Company, Beulah, North Dakota. The analyses of the two coals are presented in Table 4.

TABLE 2

Ultimate Analysis Results for Agglomerating Oils, wt%

	Phenol/ Tar Oil	Cresylic Acid	Mild Gas Tar	Used Motor Oil	V-161	V-178	V-1074	ROSE Resid
As-Determined Moisture	5.39	0.17	21.82	0.18	0.50	0.08	1.38	0.15
Moisture-Free Carbon	77.02	79.18	88.25	84.31	84.43	87.64	89.93	89.95
Hydrogen	6.91	8.67	8.71	12.95	12.00	10.17	9.14	7.24
Nitrogen	1.04	0.16		0.46	0.49	0.89	0.83	1.13
Sulfur	0.03	0.00	0.17	0.60	0.00	0.00	0.00	0.00
Oxygen	14.54	11.99	2.02	0.67	3.05	1.28	0.00	1.61
Ash	0.45	0.00	0.04	1.00	0.04	0.03	0.09	0.10

TABLE 3

"Short" Column Chromatography Analysis Results for Agglomerating Oils, wt%

	Phenol/ Tar Oil	Cresylic Acid	Mild Gas Tar	Used Motor Oil	V-161	V-178	V-1074	ROSE Resid
Aliphatics	0.99	1.15	13.30	61.5	32.2	21.31	19.2	3.44
Branched Aliphatics	5.45	0.85	1.13	1.58	1.29	3.10	0.48	0.80
Aromatics	12.60	1.33	31.16	23.9	33.85	58.17	65.6	76.5
Polars	80.96	96.67	54.41	12.9	32.64	17.42	13.8	19.2

TABLE 4

Proximate, Ultimate, and Heating Value Analyses for Beulah Lignite and Spring Creek Subbituminous Coal

	Beulah	Spring Creek
Moisture, wt%	33.20	25.30
Proximate, mf, wt%		
Volatile Matter	46.08	44.54
Fixed Carbon	46.52	51.25
Ash	7.40	4.21
Ultimate, mf, wt%		
Carbon	64.18	71.39
Hydrogen	5.03	5.00
Nitrogen	0.70	0.99
Sulfur	1.10	0.29
Oxygen	21.57	18.10
Ash	7.40	4.21
Heating Value, mf, Btu/lb	10,960	12,390

The azeotropic moisture and Soxhlet-determined oil contents plus the air- and oven-drying losses of the agglomerates are presented for the three balance tests in Tables 5, 6, and 7. The analytically determined total carbon content and the calculated oil contents of the filtrates are presented in Table 8.

Moisture, oil, and solids balances were performed for each agglomeration test using one or more of the previously described balance methods. The range of raw agglomerate compositions and closures for the balance tests are presented in Table 9. Complete balances are presented in Appendix B.

The range of raw agglomerate compositions and balance closures indicates, in some instances, considerable variability among the different balance methods. Methods 3 and 4, which used oven-drying in conjunction with Soxhlet extraction, resulted in poor water and oil balance closures. The oil balance closure by these methods ranged from 85 to 92 wt% and the solids balance closure ranged from 117 to 127 wt% during the first Beulah agglomeration balance test. The poor closures were hypothesized to have resulted when some of the agglomerating oil polymerized during agglomerate oven-drying. Presumably the polymerized oils were inextractable by the methylene chloride during Soxhlet analysis causing an erroneously low raw agglomerate oil content. Balance Method 2, which did not use oven-drying in the agglomerate analysis scheme, resulted in more reasonable oil and solids balance closures of 102 and 105 wt%, respectively.

TABLE 5

Beulah Lignite Agglomerate Analysis
Balance Test 1, wt%

Agglomerate Type	Raw	Air-Dried	Oven-Dried Raw	Oven-Dried Air-Dried
Azeotropic Moisture	31.6	2.78	1.32	1.45
Soxhlet Oil	ND*	42.9	11.3	15.1
Air-Drying Loss	41.6	ND	ND	ND
Oven-Drying Loss	55.5	26.3	ND	ND

* Not determined.

TABLE 6

Beulah Lignite Agglomerate Analysis
Balance Test 2, wt%

Agglomerate Type	Raw	Air-Dried
Azeotropic Moisture	31.6	4.79
Soxhlet Oil	23.7	42.7
Air-Drying Loss	40.0	ND*

* Not determined.

TABLE 7

Spring Creek Subbituminous Agglomerate Analysis
Balance Test 1, wt%

Agglomerate Type	Raw	Air-Dried
Azeotropic Moisture	28.4	6.28
Soxhlet Oil	37.2	40.9
Air-Drying Loss	33.6	ND*

* Not determined.

TABLE 8

Agglomeration Filtrate Analysis
Beulah Lignite and Spring Creek Subbituminous Balance Tests

Balance Test	Beulah Test 1	Beulah Test 2	Spring Creek Test 1
Total Carbon Content, mg/L	12,800	8,770	9,440
Total Oil Content, mg/L	16,600	11,400	12,300

TABLE 9

Summary of Agglomeration Balance Tests
Beulah Lignite and Spring Creek Subbituminous, wt%

Balance Test	Beulah Test 1	Beulah Test 2	Spring Creek Test 1
Raw Agglomerate Composition			
Moisture	31.6	31.6	28.4
Oil	29.5 - 36.6	23.7 - 36.9	36.5 - 37.2
Solids	31.8 - 38.9	31.5 - 44.7	34.4 - 35.1
Balance Closure			
Moisture	99.0	97.9	99.1
Oil	85.2 - 102	70.1 - 100	89.6 - 90.7
Solids	105 - 117	105 - 150	102 - 104

Subsequent agglomeration tests with Beulah lignite and Spring Creek subbituminous used only Methods 1 and 2 to calculate raw agglomerate oil contents. The results with these two methods were not always consistent though Method 2 did tend to give the most reasonable oil and solids balance closures. The oil and solids balance closures with Method 2 for Beulah lignite were 100 and 105 wt%, respectively. Similarly, the oil and solids balance closures with Method 2 for Spring Creek subbituminous were 91 and 102 wt%, respectively.

Comparison of the balance calculation methods indicates that Methods 1 and 2 are preferred to Methods 3 and 4. The former methods require fewer analyses and calculations. Also, these methods avoid the potential for oil polymerization during oven-drying and the deleterious effect on oil content determination.

Based on the more reasonable balance test closures with Beulah lignite, approximately 83 to 87 wt% of the agglomerating oil added during the process is collected on the agglomerates and tailings. The balance of the oil, 14 to 17 wt%, remains solubilized in the process water. The oil in the process water and on the tailings would be recovered by recycling the process water and the tailings to the agglomeration stage during continuous processing.

The raw Beulah lignite agglomerates were determined to lose approximately 31 to 32 wt% of their oil and 91 to 95 wt% of their moisture to evaporation during air-drying at room temperature. The evaporated oil and moisture would also be condensed and recycled for use in the agglomeration stage.

The single balance test with Spring Creek produced similar results with approximately 27 and 85 wt% of the oil and moisture evaporated during air-drying. Approximately 17 wt% of the agglomerating oil remained solubilized in the process water; the balance of the agglomerating oil was on the agglomerates and tailings.

5.0 OIL RECOVERY FROM PROCESS WATER BY SOLVENT EXTRACTION

Solvent extraction was evaluated as a method to recover, for recycle, soluble agglomerating oil from the agglomeration process water and to reduce the phenolics concentration to a level suitable for conventional biological wastewater treatment methods. Also, solvent extraction was tested as a method to verify the process water total oil content as calculated from the total carbon content.

5.1 Experimental Procedure

Solvent extraction was performed in a 2-L separatory funnel on 500 g of low-pH agglomeration process water using methylene chloride as the solvent. Prior to solvent extraction, the total soluble oil content of the process water was determined by the method described in Section 4.1. The acidic oil fractions (phenols, etc.) were eluted first using 50 mL of solvent, then the process water was neutralized with a dilute NaOH solution prior to extraction of polyaromatics (benzene, naphthalene, phenanthrene, etc.). The process water was then basified to allow extraction of basic nitrogen compounds (quinoline, aniline, etc.). The methylene chloride was evaporated from the eluted fractions to recover the acidic, neutral, and basic oil fractions. The total mass of extracted oil was then compared to the oil content calculated from the total carbon content.

5.2 Results and Discussion

Process water generated during an agglomeration test was analyzed to determine the concentration of organics resulting from solubilized agglomerating oil. The process water was analyzed to determine biological oxygen demand (BOD), chemical oxygen demand (COD), total organic carbon (TOC), total carbon (TC), total solids (TS), total suspended solids (TSS), and total phenolics (4). The results of the analysis are presented in Table 10.

The high COD to BOD ratio of 2.71:1 indicated that a significant level of refractory COD (+30,000 mg/L) would remain in the water, presuming biological treatment could remove +90% of the BOD. However, the exceedingly high phenolics content of almost 10,000 mg/L would probably prevent biological treatment of this water without prior reduction of phenolics by a pretreatment

TABLE 10

Process Water Analysis
Solvent-Recovery Testing

Constituent	Concentration, mg/L
BOD	17,700
COD	48,000
TOC	12,800
TC	16,500
TS	15,100
TSS	106
Phenolics	9,980

process. Consequently, solvent extraction was tested as an agglomeration process water pretreatment step.

Extraction of the process water with methylene chloride resulted in elution of 13.6 g of phenolic oils (acidic), 1.4 g of neutral oils, and 10.9 g of basic oils. The total extracted-oil concentration, based on 500 mL of process water, was approximately 51,500 mg/L. Comparatively, the total oil concentration, determined from the total carbon content, was approximately 21,400 mg/L, or 42% of that determined by solvent extraction. Similarly, the phenolic oil concentration determined by solvent extraction was 27,200 mg/L which compares to 9,980 mg/L as determined by standard water analysis techniques.

The oil concentration determined by solvent extraction was concluded to be inaccurate, probably due to inadequate evaporation of solvent during the solvent recovery process. Consequently, the solvent extraction technique has been temporarily dismissed as a method of determining oil content and recovering the oil for recycle.

6.0 OIL RECOVERY FROM PROCESS WATER BY ABSORPTION ON COAL

Raw and acid-cleaned coals, prepared for subsequent agglomeration tests, were investigated as an absorbant for recovering soluble oil from the agglomeration process water. This method was investigated as an alternative to solvent extraction for oil recovery and process water pretreatment. Presumably, the raw or acid-cleaned coal used to recover oil from the process water would be fed directly to the agglomeration step, thus reducing the quantity of makeup agglomerating oil in a continuous process.

6.1 Experimental Procedure

The raw or acid-cleaned -30-mesh Beulah lignite was added to 100 mL of process water and agitated using a T-bar mixer at approximately 800 rpm. Soluble oil absorption/desorption equilibria were assumed to be achieved within the 5-minute mixing period. The effect of coal quantity on oil removal was observed by varying the coal-to-process water ratio (g/mL). The "clean" process water was separated from the absorbent (raw or acid-cleaned coal) by

Buchner filtration. The "raw" and "clean" process waters were analyzed by Standard Methods 505A and 510C to determine total carbon and total phenolics content, respectively (4).

6.2 Results and Discussion

Process water generated in several different agglomeration experiments was treated using coal-to-water ratios in the range of 0.1 to 1.0 g/mL, and the effect on total carbon and total phenolics content is illustrated in Figures 2 and 3.

The acid-cleaned coal reduced the total carbon and total phenolics concentrations by 87 and 89 wt%, respectively, at a coal/water ratio of only 0.8. The raw coal was slightly less effective, absorbing 85% of the total carbon and 81% of the total phenolics at a coal/water ratio of 1.0 g/mL. At coal/water ratios as low as 0.25 to 0.50, the acid-cleaned and raw coals were able to reduce the total carbon concentration by 50% to 70%.

Preliminary testing indicated that approximately 90% of the soluble agglomerating oil could be recovered from the process water at coal/water ratios of around 1.0 g/mL. The level of removal would need to be enhanced because up to 2 wt% of the agglomerating oil added to the process still remains solubilized in the process water. This technique will be investigated further to determine the maximum level of total carbon and total phenolics reduction and the associated coal/water ratio.

7.0 AGGLOMERATE DEOILING

Thermal deoiling was investigated as a method for reducing the oil content of agglomerates and recovering the oil for recycle to the agglomeration stage. Thermal deoiling was also investigated as a method of producing a stronger, more stable lump fuel suitable for transport and resistant to moisture reabsorption and spontaneous combustion.

7.1 Experimental Procedure

Agglomeration deoiling tests were performed using two different systems, a 2-gallon batch reactor autoclave and a batch fluid-bed reactor. The two deoiling systems are illustrated in Figures 4 and 5.

The first deoiling system was an externally heated 2-gallon autoclave equipped with a single temperature controller and a single thermocouple to monitor agglomerate bed temperature. A test consisted of charging a 30-mesh basket with approximately 300 g of air-dried agglomerates and placing the basket into the room temperature autoclave. The heaters were activated after sealing the reactor, and an unheated nitrogen purge was introduced at the bottom of the autoclave to aid evacuation of the vaporized agglomerating oil and moisture. The agglomerates were maintained at temperature for 45 minutes, after which the reactor was allowed to cool before recovering the deoiled agglomerates. The vaporized agglomerating oils and moisture were recovered by water- and ice water-cooled condensers. The weights of the air-dried and deoiled agglomerates plus the condensables were recorded to determine the deoiling efficiency and material recovery.

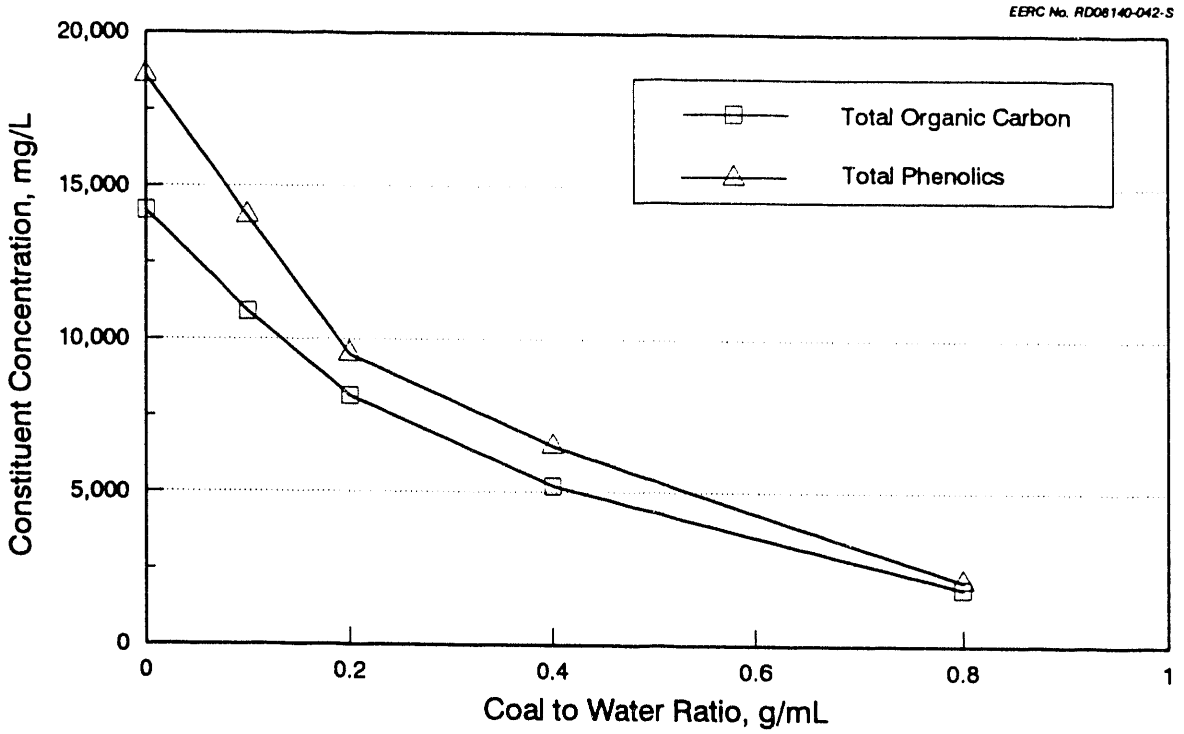

Figure 2. Reduction of process water total carbon and total phenolics by absorption with acid-cleaned Beulah lignite.

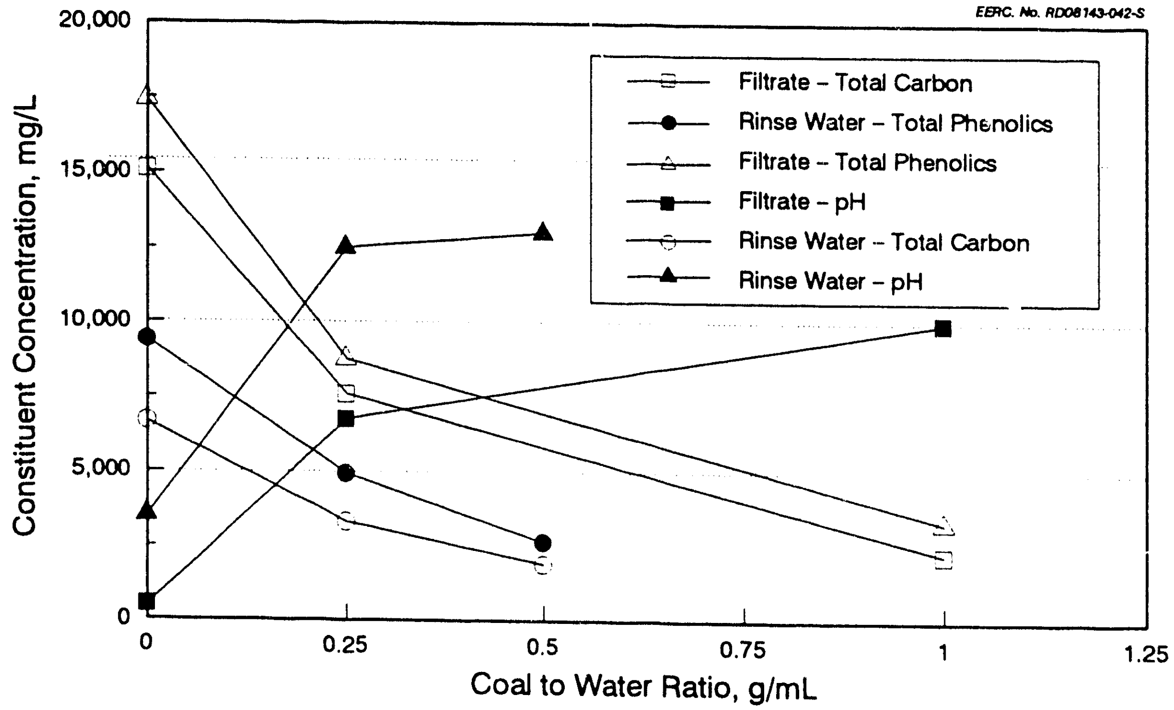

Figure 3. Reduction of process water total carbon and total phenolics by absorption with raw Beulah lignite.

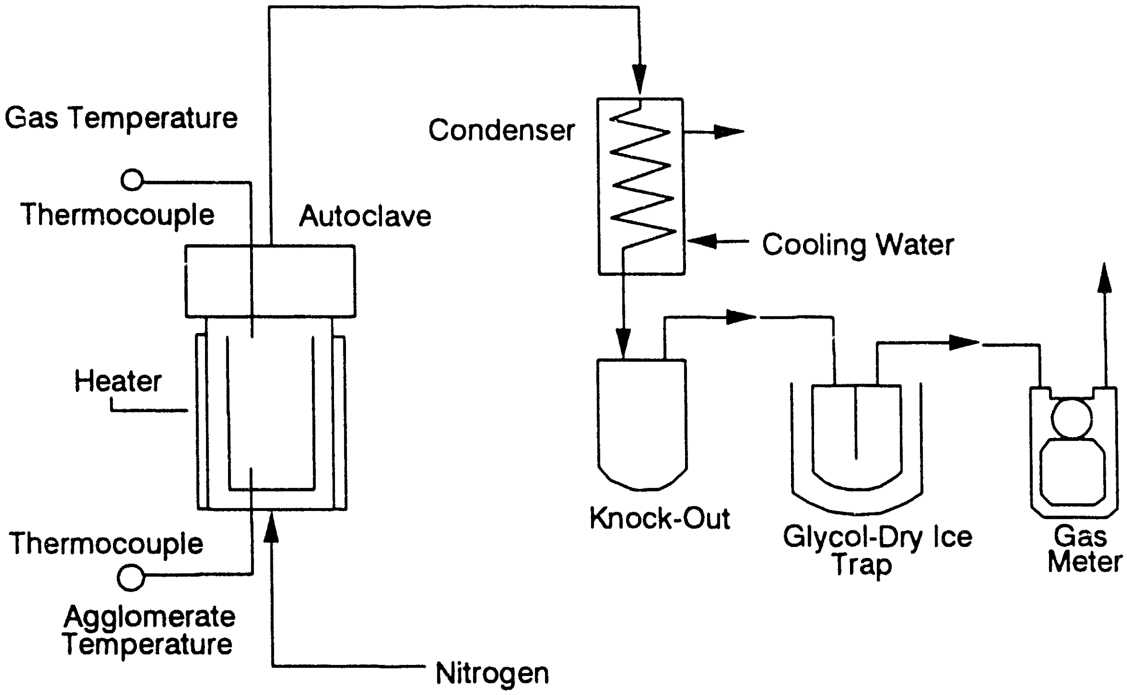

Figure 4. Batch autoclave thermal deoiling system.

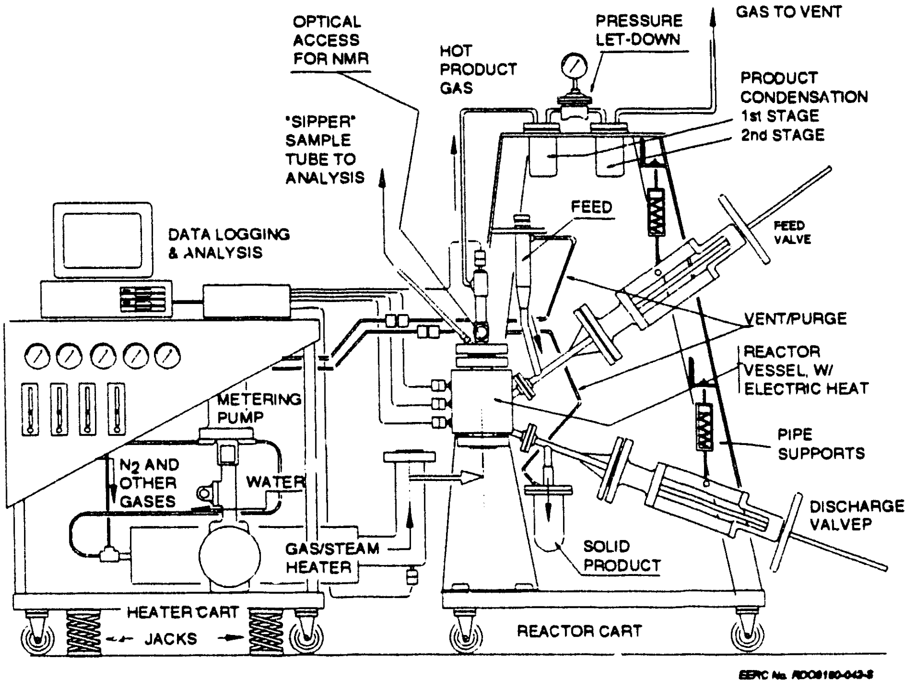

Figure 5. Batch fluid-bed thermal deoiling system.

The second deoiling system was an externally heated batch fluid-bed reactor with an internal diameter of 1.5 in and a heated zone 9 in long. The agglomerates, determined from shakedown testing, were 100 g. Prior to charging the air-dried agglomerates, the fluid-bed reactor was preheated to the desired deoiling temperature using superheated steam. The external heaters were used to help maintain deoiling temperature. The air-dried agglomerates were charged to the preheated reactor from a pressurized hopper through a manually operated gate valve. Temperature monitoring and recording were provided at the reactor inlet; 2, 4, and 6 in above the distributor plate; and at the reactor outlet. A 100-mesh screen was installed at the reactor outlet to prevent entrainment of agglomerates or agglomerate fines. The agglomerates were treated for 40 minutes using superheated nitrogen or steam as the fluidizing and thermal deoiling medium. The vaporized agglomerating oil and moisture were recovered using two water-cooled condensers. The agglomerates were discharged from the pressurized reactor through a gate valve into a collection vessel. The weights of the air-dried and deoiled agglomerates and the condensables were recorded to determine the deoiling efficiency and material recovery.

7.2 Results and Discussion

7.2.1 Superheated-Nitrogen Deoiling Tests

A single deoiling test was performed using the batch autoclave deoiling system. Approximately 315 g of air-dried agglomerates were deoiled at a nominal temperature of 150°C for 1 hour. The deoiling test was considered a failure, however, for a number of reasons. First, temperature control was difficult and, over the test period, the agglomerate bed temperature increased to approximately 170°C. Second, the mass loss during the deoiling test was only 28 wt%, which is considerably less than evaporative loss by oven-drying at 105°C. Third, the mass balance closure was only 93.5 wt%, suggesting poor recovery of evaporated oil and moisture. As a consequence of these difficulties, the batch autoclave was temporarily abandoned as a thermal deoiling system, and subsequent deoiling tests were attempted with the batch fluid-bed reactor.

A series of four batch fluid-bed deoiling tests was performed at 200°C using superheated nitrogen as the deoiling and fluidizing medium. The deoiled agglomerates from the four tests were combined into a composite sample as was the condensed oil/moisture fractions. The fluid-bed reactor proved to be an ideal system for maintaining a proper and uniform bed temperature. The agitation induced by the fluidizing gas produced little agglomerate decrepitation or fines production. A total of 401 g of air-dried agglomerates were deoiled, producing 251 g of deoiled agglomerates and 117 g of condensed oil/moisture. The material balance closure was approximately 92 wt% with the loss attributed to poor recovery of evaporated oil and moisture.

The Soxhlet oil and azeotropic moisture contents of the air-dried and nitrogen-deoiled agglomerates are presented in Table 11. The air-dried agglomerate moisture and oil contents were 5.9 and 38 wt%, respectively, which compare to the deoiled agglomerate moisture and oil contents of 2.0 and 0.3 wt%, respectively. A moisture and oil balance, assuming 100% recovery of agglomerate solids, indicated that the agglomerate weight loss did not equal the oil and moisture weight losses. The Soxhlet oil content of the deoiled agglomerates was estimated to be low by 7.5 wt%, presumably due to the

TABLE 11

Beulah Lignite Agglomerate Moisture and Oil Contents
Superheated-Nitrogen and Steam-Deoiling Tests, wt%

Agglomerates	Air-Dried	Nitrogen Deoiled	Steam Deoiled
Azeotropic Moisture	5.85	2.04	2.47
Soxhlet Oil	37.7	0.33	0.24

hypothesized polymerization of agglomerating oil during the deoiling tests. It was not possible to verify this estimate however, because of the poor overall material balance closure.

The condensate from the nitrogen deoiling experiment was analyzed by open column chromatography to determine the concentration of aliphatics, aromatics, and polars. The concentrations of linear and small aliphatics, branched and large aliphatics, aromatics, and polars were determined to be 0.85, 1.31, 2.93, and 94.9 wt%, respectively. These concentrations compare to the original agglomerating oil compositions of 0.96, 5.29, 12.2, and 78.5 wt%, respectively. The apparent increase in the polar content may have been caused by one or more concurrent phenomena including preferential absorption of specific oil components during agglomeration, evaporation of specific oil components during deoiling, and the inefficient recovery of specific volatile oil components during deoiling. The recovery of vaporized oils and moisture would have to be greatly enhanced and the soluble oil in the process water would have to be isolated and analyzed to substantiate these suggestions.

Considering the relative simplicity, the uniform temperature control, and the minimal agglomerate degradation, fluid-bed deoiling with superheated nitrogen will be investigated further, using this or a larger batch system.

7.2.2 Superheated-Steam Deoiling Tests

Two batch fluid-bed deoiling tests were performed at 230°C using superheated steam as the deoiling and fluidizing medium. The deoiled agglomerates from the two tests were combined into a composite sample as were the condensed oil/moisture fractions. A total of 202 g of air-dried agglomerates were deoiled, producing 113 g of deoiled agglomerates. The material balance closure was low at 89 wt% and this was attributed to the inefficient condensation of agglomerating oil and superheated steam.

The analysis of the air-dried and superheated steam deoiled agglomerates are presented in Table 11. The azeotropic moisture content and Soxhlet oil content of the deoiled agglomerates were 2.5 and 0.2 wt%, respectively, which compare to the air-dried agglomerate moisture and oil contents of 5.9 and 38 wt%, respectively. The extremely low oil content suggests almost complete removal during deoiling. Presumably this could have been substantiated by determining the mass of oil in the condensate stream. This effort was confounded by poor condensate recovery and by the inability to determine the condensate oil concentration. In addition to being solubilized in the condensate, the oil existed as discrete droplets. The oil droplets would not settle out during decantation, but adhered to the separatory funnel walls. Because of the dispersed oil droplets and the resulting non-homogeneity of the condensate, the standard technique of determining total carbon content was not applicable.

Owing to the difficulties in accurately determining the oil content of the condensate and to the necessity to superheat, condense, and then treat large quantities of water/condensate, superheated steam deoiling will be temporarily abandoned as an oil recovery method.

8.0 AGGLOMERATE STRENGTH AND STABILITY TESTING

Preliminary strength and stability tests for agglomerates included durability toward abrasion and resistance toward moisture reabsorption. Strength and stability testing are necessary to determine if the agglomerates can maintain their integrity during the rigors of transportation and storage. Ideally the strength and stability of the agglomerates would be correlated to the moisture and oil contents as determined by the previously described analysis techniques.

8.1 Experimental Procedure

The resistance to abrasion was determined at the relatively mild conditions achieved by continuous vibration on the screens of a Ro-Tap sieve apparatus. Prior to durability testing, the agglomerates were carefully screened, avoiding breakage, to determine the agglomerate size distribution. The sized agglomerates were placed on the top screen, and the Ro-Tap machine was operated for 10 minutes. The size distribution of the agglomerates after durability testing was compared to the original distribution to determine the abrasion loss.

The resistance to moisture reabsorption was determined using a modified ASTM D1412 equilibrium moisture procedure. The agglomerates were not water soaked prior to equilibrium moisture testing in order to avoid possible agglomerate decrepitation. The agglomerates were incubated at 30°C at 96%-97% relative humidity until the weight change was less than 0.1 g per day. The final moisture content of the incubated agglomerates was calculated from the weight loss/gain and the initial azeotropic moisture content. The moisture content at equilibrium was not determined by the traditional oven-drying weight loss method because of potential agglomerating oil evaporation.

8.2 Results and Discussion

Durability and moisture reabsorption tests were performed on air-dried and oven-dried (105°C) agglomerates. The azeotropic moisture and Soxhlet oil contents of the air- and oven-dried agglomerates are presented in Table 12. The size distributions of agglomerates after durability testing were compared to the original distributions and the results are presented in Figures 6 and 7. The test results indicated that the moderate abrasion conditions during Ro-Tap sieve testing did not significantly reduce the air- or oven-dried agglomerate size. The moisture and oil content appeared to have little

TABLE 12

Beulah Lignite Agglomerate Moisture and Oil Contents
Strength and Stability Tests, wt%

Agglomerate Type	Air-Dried	Oven-Dried
Azeotropic Moisture	6.0	1.0
Soxhlet Oil	44.0	14.3
Equilibrium Moisture	ND*	10.4

* Not determined.

effect on agglomerate strength as the air- and oven-dried agglomerates exhibited similar size degradation.

The equilibrium moisture content of the oven-dried agglomerates was approximately 10 wt% which compares to the original oven-dried moisture content of 1.0 wt% and a raw Beulah lignite equilibrium moisture content of 27 wt%. The calculation of the air-dried agglomerate equilibrium moisture content was confounded by the simultaneous evaporation of agglomerating oil.

Future agglomerate strength tests will include drop, tumble, and compressive load tests similar to those used on raw, pelletized, or briquetted coals. These tests will be performed after development of a reliable deoiling system which can process the required quantities of agglomerates.

9.0 ACID-CLEANING TESTS

The inorganics of the LRCs tested in the EERC agglomeration process are predominantly associated with the organic matrix as the carboxylic or phenolic salts of alkali and alkaline earth metals. Physical cleaning is only effective for removing extraneous minerals and has a limited effect on the removal of deposit-causing inorganic species such as sodium and calcium. Consequently, ion exchange via acid cleaning is necessary to remove these constituents and significantly lower the inorganic content. Added benefits of acid cleaning prior to agglomeration are the dissolution of acid-soluble extraneous minerals and the surface conditioning that enhances the effectiveness of the agglomerating oil to bind the coal. The acid is hypothesized to change the surface chemistry of the LRCs by reducing oleophobic sites.

Several experiments were performed using nitric acid leaching on Beulah lignite and Spring Creek subbituminous coal. The results from this testing helped determine the optimum conditions for achieving maximum ash reduction with these two coal samples. The acid-cleaning experiments were performed under the same conditions as those utilized prior to agglomeration.

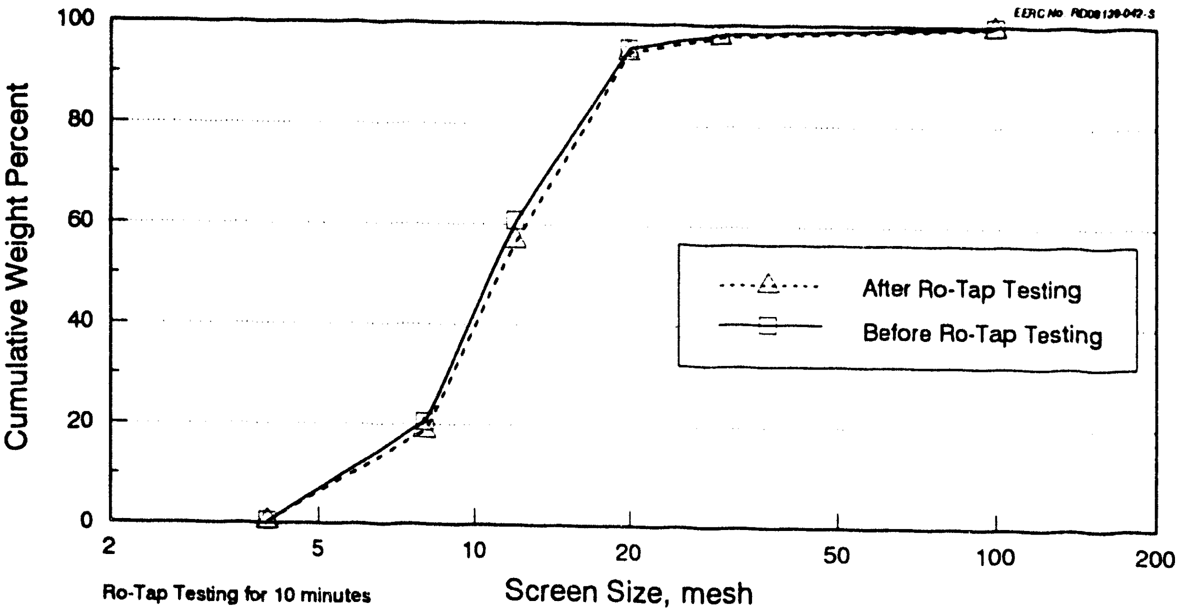

Figure 6. Size distribution of air-dried agglomerates before and after Ro-Tap durability testing.

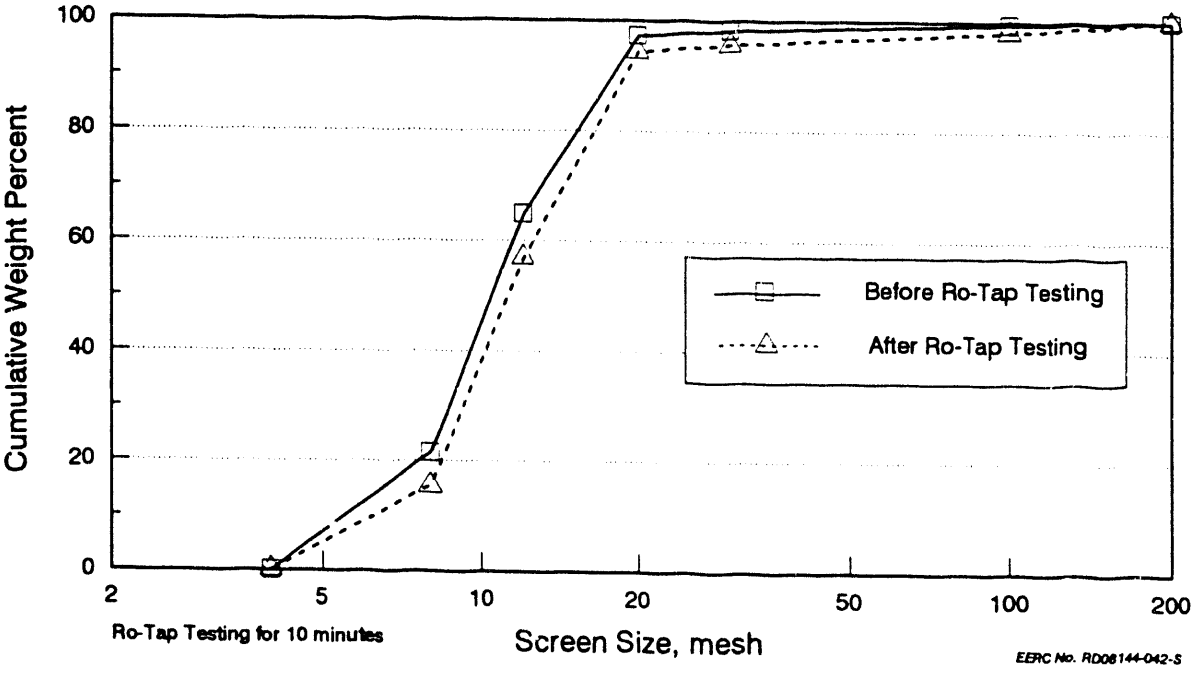

Figure 7. Size distribution of oven-dried agglomerates before and after Ro-Tap durability testing.

A titrimetric procedure using 0.1 N sodium hydroxide was developed to determine acid consumption as a function of acid solution strength and ash reduction.

Table 13 presents the results of cleaning Beulah lignite with nitric acid solutions of 0.12 to 1.29 M. Ash reductions ranged from 18 to 63 wt%, and the highest ash reductions were achieved when the acid concentration was greater than 0.5 M. At acid concentrations less than 0.5 M, the nitric acid is almost completely consumed.

Tables 14 and 15 present the results of cleaning Spring Creek subbituminous coal with nitric acid solutions of 0.14 to 0.89 and 0.16 to 1.28 M, respectively. Ash reductions in Table 14 ranged from 15 to 40 wt% and acid consumption ranged from 50 wt% at the highest concentration to 96 wt% at the lowest acid concentration. The acid cost from this testing produced results that range from $4.29 to $14.67 per ton coal. Ash reductions in Table 15 ranged from 27 to 47 wt% and acid consumption ranged from 35 to 97 wt%. Acid cost for this experiment ranged from $5.07 to $14.58 per ton of coal. The results from acid cleaning the coal presented in Table 15 indicate that the coal sample used in Table 14 was slightly oxidized. This was substantiated by the moisture analysis that indicated a moisture content for the Table 14 sample was 17 wt%. This is considerably lower than the moisture for the Table 15 sample that had a moisture content of 25 wt%, which is more typical for this coal. Oxidation of the coal surface inhibits the ability of the acid to ion-exchange with the coal, and the ash reduction is not as great for an oxidized sample.

Figures 8 and 9 indicate the effect of nitric acid cleaning on the ash constituents of Beulah lignite and Spring Creek subbituminous coal, respectively. For Beulah lignite, maximum removal of ion-exchangeable species is essentially achieved at a concentration of ~0.5 M. Sodium, although low in this coal sample, is nearly completely removed, and calcium and magnesium are reduced by ~82 wt% at these acid concentrations. At an acid concentration of 0.5 M, the acid cost per ton of coal cleaned, from Table 13, is approximately $13, based on $185/ton for 70 wt% concentrated nitric acid.

Spring Creek subbituminous coal results in Figure 9 indicate significant removal of ion-exchangeable species is essentially achieved at a concentration of 0.16 M. Reduction in sodium, magnesium and calcium content at this concentration is 81, 69 and 34 wt%, respectively. In comparison at 1.28 M these species are reduced in concentration by 95, 79 and 68 wt%, respectively. This comparison indicates that cost-effective cleaning of this coal could be achieved at 0.16 M. Since the ion-exchangeable species are significantly reduced at this concentration and the acid cost is approximately $5.1/ton coal, based on $185/ton for 70 wt% concentrated nitric acid, this should be the acid concentration of choice for achieving ion-exchangeable mineral reduction. If higher acid concentrations are chosen, it is apparent that substantial reduction of ion-exchangeable inorganics in LRCs is more costly. As with Beulah, acid cleaning of Spring Creek does not appreciably change the discrete mineral content.

The data presented indicate that Spring Creek acid cleaning is optimized at ~0.16 M, and for Beulah it is optimized at ~0.5 M. From the data in the tables and figures, it is apparent that substantial reduction of ion-exchangeable inorganics in LRCs is costly when using nitric acid.

TABLE 13

Nitric Acid-Cleaning Results for Beulah Lignite

Acid Molarity:	1.29	0.93	0.64	0.47	0.32	0.23	0.16	0.12
Ash Content[1]	2.84	3.08	2.91	3.47	4.25	5.43	5.70	6.26
Ash Reduction, wt%	63.4	60.2	63.1	54.7	44.1	29.8	23.6	18.6
Acid Consumption, wt%	41.5	51.6	63.3	84.0	95.0	98.6	99.1	99.6
lb Conc. Acid / 100-lb Coal	9.48	8.50	7.17	6.99	5.40	4.02	2.81	2.11
Acid Cost, $/Ton Coal[2]	17.54	15.73	13.26	12.93	9.99	7.43	5.19	3.91

[1] Raw coal ash content = 7.4 wt% mf.
[2] $185/ton; 70 wt% concentrated nitric acid.

TABLE 14

Nitric Acid-Cleaning Results for Spring Creek Subbituminous Coal

Acid Molarity:	0.89	0.46	0.25	0.14
Ash Content[1]	2.93	3.28	3.61	4.17
Ash Reduction, wt%	40.2	34.5	27.7	15.0
Acid Consumption, wt%	50.2	66.8	88.6	95.6
lb Conc. Acid / 100-lb Coal	7.93	5.46	3.85	2.32
Acid Cost, $/Ton Coal[2]	14.67	10.10	7.12	4.29

[1] Raw coal ash content = 5.05 wt% mf.
[2] $185/ton; 70 wt% concentrated nitric acid.

TABLE 15

Nitric Acid-Cleaning Results for Spring Creek Subbituminous Coal

Acid Molarity:	1.28	0.63	0.32	0.16
Ash Content[1]	2.24	2.53	2.76	3.20
Ash Reduction, wt%	46.9	41.2	35.7	26.9
Acid Consumption, wt%	34.8	44.2	70.3	96.6
lb Conc. Acid / 100-lb Coal	7.88	4.96	3.99	2.74
Acid Cost, $/Ton Coal[2]	14.58	9.18	7.38	5.07

[1] Raw coal ash content = 4.37 wt% mf.

[2] $185/ton; 70 wt% concentrated nitric acid.

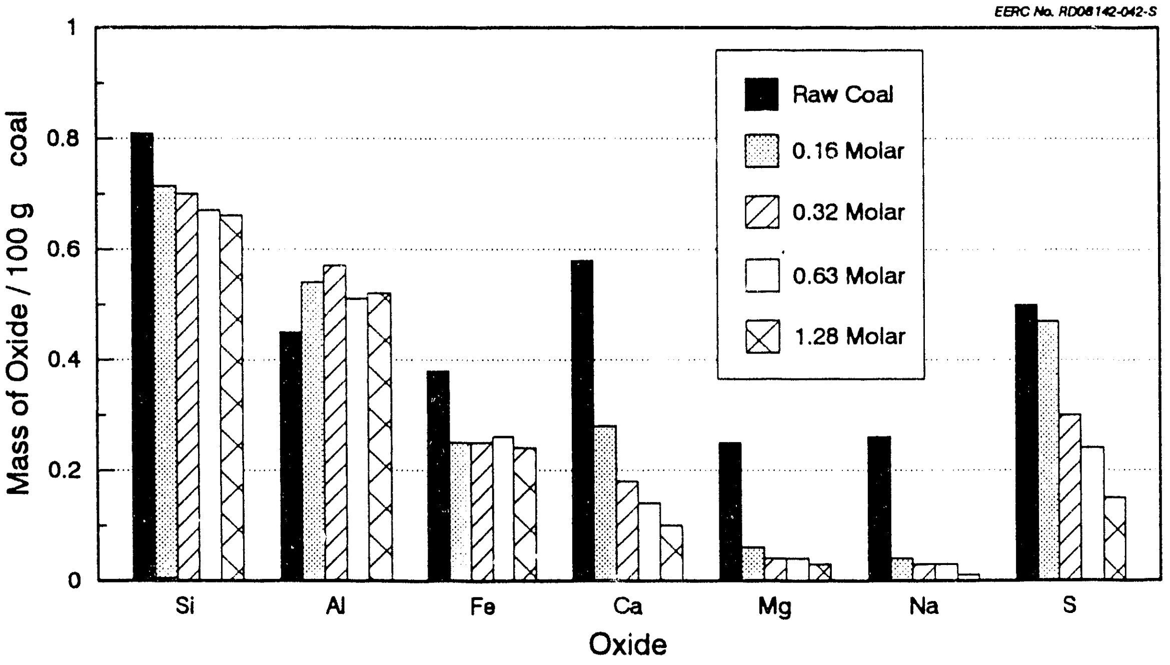

Figure 8. Mass of ash constituents for raw coal and nitric acid-cleaned fractions of Beulah lignite.

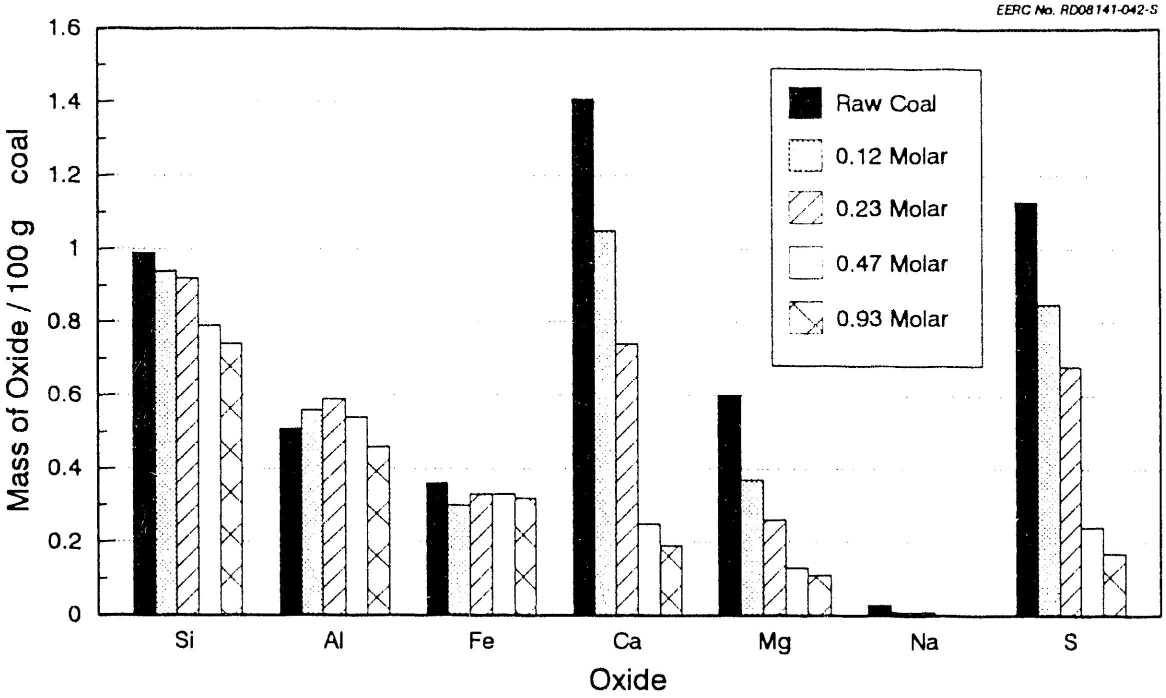

Figure 9. Mass of ash constituents for raw coal and nitric acid-cleaned fractions of Spring Creek subbituminous coal.

In addition to nitric acid cleaning, acid cleaning was performed on these two coals using a solution of carbonic acid. The carbonic acid source was carbonated sparkling water. The acid only slightly reduced the ash content of Beulah (7.87 compared to 7.43) and has essentially no effect on the ash content of Spring Creek (3.92 compared to 3.76). Although this acid did not appear to be effective, an ash analysis should be performed to see if any ion-exchange was achieved with the sodium in the coals.

In order to utilize acid cleaning in the agglomeration process, methods have to be developed to either recycle the acid or neutralize it with a basic solution. Sodium hydroxide would be very costly to use for neutralization; therefore, testing was initiated using hydrated lime as a neutralizing agent. Initially the filtrate was neutralized using hydrated lime powder. This was not successful because an endpoint was difficult to observe since the hydrated lime did not readily go into solution. Therefore, a solution of 1 M hydrated lime was made for addition to filtrate solutions. The target pH was 7.0; however, attainment of a pH very near 7.0 was difficult to achieve, especially at low initial residual acid concentrations. Experiments with filtrates from the higher acid filtrates indicated that approximately 1 mole of hydrated lime is required to neutralize 2 moles of acid. For example, 30 mL of 0.31 M filtrate solution required 5 mL of 1.0 M hydrated lime solution to raise the pH to 7.89.

Further testing needs to be performed using the hydrated lime solution in order to better establish the conditions necessary to neutralize the filtrate.

10.0 COLLOIDAL COAL CLEANING

Colloidal coal cleaning was investigated as a method to reduce the ash content of LRC by separating the carbonaceous coal material from the inorganics. Extraction of a LRC with an alkaline medium can produce a colloidal humate dispersion or a hydrosol from which the humic acid can be precipitated (7) resulting in low-ash fuel suitable for advanced combustion systems.

10.1 Experimental Procedure

Approximately 50 g of coal was added to 250 g of a 10 wt% NaOH solution, and the mixture was blended at 11,000 rpm for 60 minutes using a Virtis high-shear homogenizer. The blended slurry was centrifuged at 3600 rpm for 30 minutes using an IEC Model K centrifuge to separate the supernatant hydrosol from the extracted coal residue. The humic acid was precipitated by adding concentrated nitric acid to the hydrosol until the pH was slightly below 1. The humic acid was recovered by centrifugation at 3600 rpm for 30 minutes. The residue and humic acid were analyzed by standard methods to determine ash and moisture contents. The weights of the residue, humic acid, wash water, and centrate were recorded for determination of ash reduction and material recovery.

10.2 Results and Discussion

A preliminary colloidal coal-cleaning test was performed using pulverized Beulah lignite in order to become familiar with the process. The products from colloidal coal cleaning of the Beulah lignite were a dark residue, presumably containing unextracted coal and coal inorganics, plus the brown, low-density, semifluid humic acid which was devoid of distinct particles. The acidified process water was essentially clear, indicating that hydrosol had been completely precipitated as humic acid.

Approximately 188 g of residue containing 12.3 wt% solids and approximately 49 g of humic acid containing 13.6 wt% solids were recovered from 50 g (33 g solids) of coal. The residue and humic acid ash contents were 50.7 and 22.2 wt%, respectively, on a moisture-free basis. The high combustible content of the residue suggests that a considerable quantity of coal solids remained unextracted. This may have resulted from insufficient mixing time, improper coal size, or a low NaOH/coal ratio. Contrastingly, the high ash content of the humic acid suggests that the precipitate contained as considerable quantity of the sodium humate. Previous colloidal coal-cleaning tests have indicated the necessity for numerous washings with water to reduce the ash (Na^+) content (7).

Additional colloidal coal-cleaning tests will be performed with Beulah lignite to determine the NaOH/coal ratio required to optimize coal inorganic reduction and material recovery. The number of water washings required to achieve the minimum humic acid ash content will also be determined. The process will also be modified to eliminate the acidification step by precipitating a calcium humate instead of humic acid. The calcium humate could be fired in conventional or advanced combustion systems with high sulfur coals so that the atomically dispersed calcium can act as a sulfur oxide sorbent.

11.0 CONCLUSIONS

1. Agglomeration process scale-up was successful using a 20-liter pail to form larger quantities of agglomerates for analysis.

2. Ash reductions of greater than 50 wt% for Beulah lignite were achieved when the acid concentration was ~0.5 M.

3. Ash reductions for Spring Creek subbituminous coal ranged from 15 to 47 wt% for acid concentrations from 0.14 to 1.28 M.

4. Significant removal of ion-exchangeable species for Beulah lignite using nitric acid is essentially achieved at ~0.5 M, and for Spring Creek subbituminous coal it is achieved at ~0.16 M.

5. The most effective oils in the EERC agglomeration process have high oxygen and polar contents.

6. Liquefaction oils and used crankcase oil are only effective in oil agglomeration using high-shear conditions or surfactant addition prior to low-shear mixing conditions.

7. Agglomeration was successful when sources of ionic strength were potassium nitrate, calcium acetate, and carbonic acid.

8. Agglomeration can be achieved without surface conditioning or ion-exchange prior to agglomeration.

9. Methods of analysis have been investigated that produce material balances ranging from 91% to 105%.

10. Oil remaining solubilized in the process water following agglomeration ranges from 14% to 17%.

11. Solvent extraction methods are not acceptable for recovering oil from process water, due to inadequate evaporation of solvent during the solvent recovery process.

12. Agglomerant can be recovered from process water using raw or acid-cleaned coal to absorb the solubilized agglomerant with recoveries ranging from 81% to 89%.

13. Oxygen and polar content of agglomerants could be used to screen candidates for LRC agglomeration.

14. Autoclave deoiling has proven to be an ineffective method for agglomerate deoiling due to the poor recovery of material and inadequate temperature control of the system.

15. Fluidized-bed deoiling is an effective method for deoiling the agglomerates using superheated nitrogen as the deoiling and fluidizing medium because of its simplicity, uniform temperature control, and minimal agglomerate degradation.

16. Fluidized-bed deoiling using superheated steam is an inadequate deoiling method because of poor condensate recovery and difficulty in accurately determining oil content of the condensate.

17. Residue and humic acid ash contents following colloidal coal cleaning were 50.7 and 22.2 wt%, respectively, indicating that additional extraction on the residue and additional washing of the humic acid may be needed.

18. Air- and oven-dried agglomerates are able to withstand moderate Ro-Tap abrasion testing with minimal size degradation.

19. Equilibrium moisture contents of oven-dried agglomerates are approximately 10 wt% compared to 27 wt% for raw Beulah coal.

12.0 FUTURE RESEARCH EFFORTS

Testing for the next year will concentrate on improving the fluid-bed thermal deoiling system to achieve near 100% balance closure. Sufficient deoiling tests will be performed to provide agglomerates for strength and stability testing. The strength and stability of the deoiled agglomerates will be correlated to the analytically determined moisture and oil contents. Successful investigation of these tasks will determine if a transportable solid fuel can be produced and whether deoiling is necessary to produce a transportable fuel. A plant design and economic analysis will be prepared based on the consumption of acid and agglomerating oil.

1. Agglomeration design scale-up of recovery processes have to be optimized in order to eliminate labor-intensive steps that result in loss of product.

2. Further investigation of ion-exchange using carbonic acid is needed for comparison with nitric acid for removal of ion-exchangeable species.

3. Analysis for trace metals in used crankcase oil should be performed if this oil is chosen as an agglomerant.

4. Perform ash fusion analysis on agglomerates that are ion-exchanged with nitric and carbonic acid and on those that are not ion-exchanged and determine which has the most promising combustion properties.

5. Do further analysis on neutralizing filtrate using hydrated lime, and determine the feasibility of its use in the agglomeration process versus the reuse of the acid.

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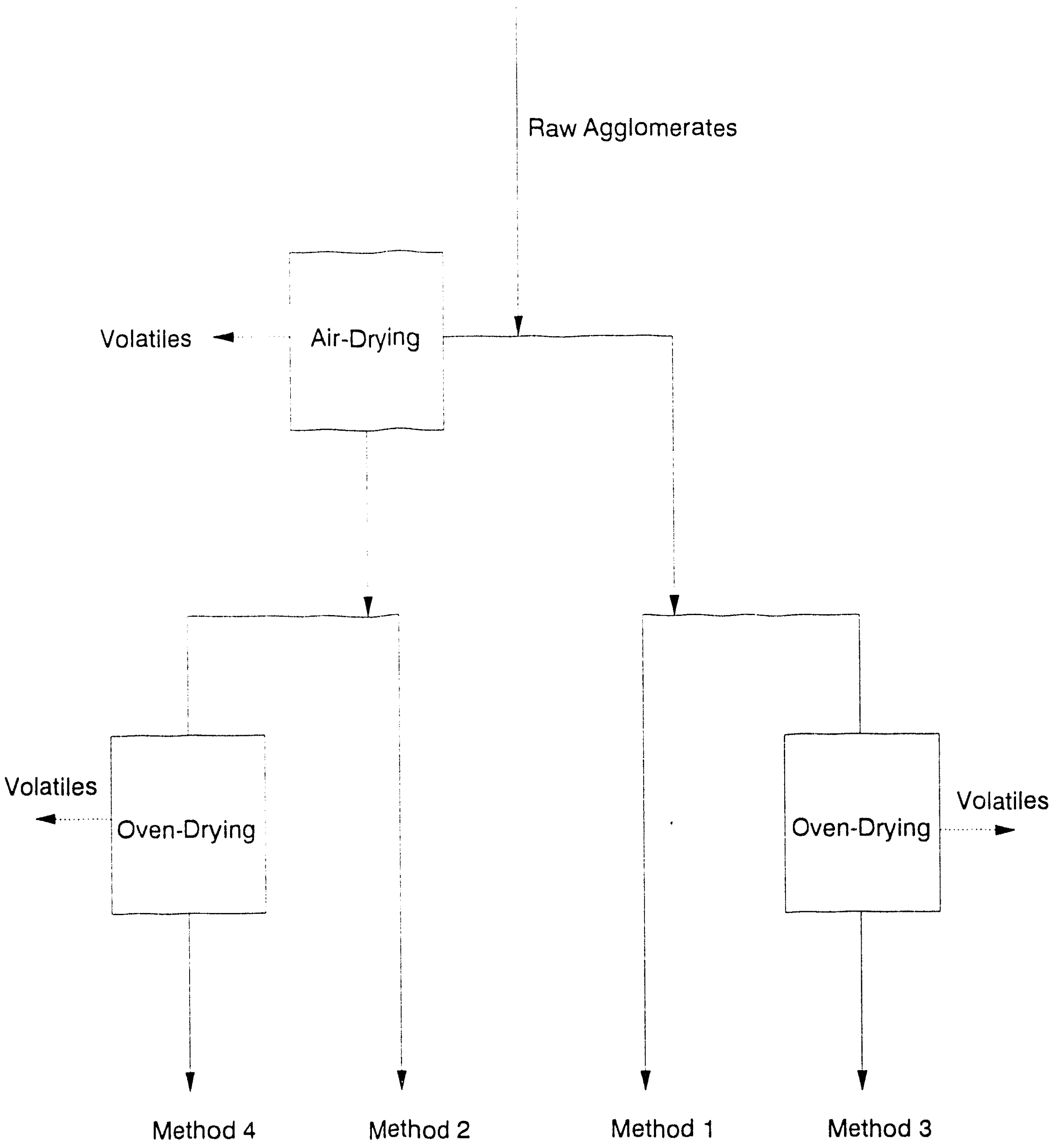
APPENDIX A

Sequence to Determine Moisture and Oil Contents of Raw Agglomerates

Appendix A


Raw Agglomerates
Volatiles
Air-Drying
Volatiles
Oven-Drying
Oven-Drying
Volatiles
Method 4
Method 2
Method 1
Method 3

APPENDIX B

Moisture, Oil, and Solids Balances

TABLE B-1

Beulah Agglomeration Balance Test 1
Raw Agglomerate Oil Calculation Method 2

Component	HNO_3	Water	Oil	Solids	Total
Influents, g					
Coal	0.0	61.0	0.0	139.0	200.0
Acid Solution	12.0	388.0	0.0	0.0	400.0
Agglomerant	0.0	10.8	189.3	0.0	200.1
Wash/Rinse Water	0.0	1308.8	0.0	0.0	1308.8
Total	12.0	1768.6	189.3	139.0	2108.9
Effluents, g					
Raw Agglomerates	0.0	129.0	149.5	129.8	408.3
Raw Tailings	0.0	14.5	16.0	15.4	45.9
Filtrate/Rinse Water	12.0	1607.9	27.2	0.0	1647.1
Total	12.0	1751.4	192.7	145.2	2101.3
Closure, wt%	100.0	99.0	101.8	104.5	99.6

TABLE B-2

Beulah Agglomeration Balance Test 1
Raw Agglomerate Oil Calculation Method 3

Component	HNO_3	Water	Oil	Solids	Total
Influents, g					
Coal	0.0	61.0	0.0	139.0	200.0
Acid Solution	12.0	388.0	0.0	0.0	400.0
Agglomerant	0.0	10.8	189.3	0.0	200.1
Wash/Rinse Water	0.0	1308.8	0.0	0.0	1308.8
Total	12.0	1768.6	189.3	139.0	2108.9
Effluents, g					
Raw Agglomerates	0.0	129.0	120.4	158.9	408.3
Raw Tailings	0.0	14.5	13.5	17.9	45.9
Filtrate/Rinse Water	12.0	1607.9	27.2	0.0	1647.1
Total	12.0	1751.4	161.1	176.8	2101.3
Closure, wt%	100.0	99.0	85.1	127.2	99.6

TABLE B-3

Beulah Agglomeration Balance Test 1
Raw Agglomerate Oil Calculation Method 4

Component	HNO_3	Water	Oil	Solids	Total
Influents, g					
Coal	0.0	61.0	0.0	139.0	200.0
Acid Solution	12.0	388.0	0.0	0.0	400.0
Agglomerant	0.0	10.8	189.3	0.0	200.1
Wash/Rinse Water	0.0	1308.8	0.0	0.0	1308.8
Total	12.0	1768.6	189.3	139.0	2108.9
Effluents, g					
Raw Agglomerates	0.0	129.0	132.7	146.6	408.3
Raw Tailings	0.0	14.5	14.9	16.5	45.9
Filtrate/Rinse Water	12.0	1607.9	27.2	0.0	1647.1
Total	12.0	1751.4	174.8	163.1	2101.3
Closure, wt%	100.0	99.0	92.3	117.3	99.6

TABLE B-4

Beulah Agglomeration Balance Test 2
Raw Agglomerate Oil Calculation Method 1

Component	HNO_3	Water	Oil	Solids	Total
Influents, g					
Coal	0.0	34.1	0.0	65.9	100.0
Acid	8.6	192.0	0.0	0.0	200.6
Agglomerant	0.0	5.7	100.2	0.0	105.9
Wash/Rinse Water	0.0	1364.1	0.0	0.0	1364.1
Total	8.6	1595.9	100.2	65.9	1770.6
Effluents, g					
Raw Agglomerates	0.0	64.6	48.4	91.3	204.3
Raw Tailings	0.0	5.9	4.5	8.4	18.8
Filtrate	8.6	362.9	7.4	0.0	378.9
Rinse Water	0.0	1129.7	9.9	0.0	1139.6
Total	8.6	1563.1	70.2	99.7	1741.6
Closure, wt%	100.0	97.9	70.1	151.3	98.4

TABLE B-5

Beulah Agglomeration Balance Test 2
Raw Agglomerate Oil Calculation Method 2

Component	HNO_3	Water	Oil	Solids	Total
Influents, g					
Coal	0.0	34.1	0.0	65.9	100.0
Acid	8.6	192.0	0.0	0.0	200.6
Agglomerant	0.0	5.7	100.2	0.0	105.9
Wash/Rinse Water	0.0	1364.1	0.0	0.0	1364.1
Total	8.6	1595.9	100.2	65.9	1770.6
Effluents, g					
Raw Agglomerates	0.0	64.6	75.4	64.3	204.3
Raw Tailings	0.0	5.9	7.8	5.1	18.8
Filtrate	8.6	362.9	7.4	0.0	378.9
Rinse Water	0.0	1129.7	9.9	0.0	1139.6
Total	8.6	1563.1	100.5	69.4	1741.6
Closure, wt%	100.0	97.9	100.3	105.3	98.4

TABLE B-6

Spring Creek Agglomeration Balance Test 1
Raw Agglomerate Oil Calculation Method 1

Component	HNO_3	Water	Oil	Solids	Total
Influents, g					
Coal	0.0	24.6	0.0	75.4	100.0
Acid Solution	16.7	190.5	0.0	0.0	207.2
Agglomerant	0.0	5.7	100.5	0.0	106.2
Wash/Rinse Water	0.0	1195.5	0.0	0.0	1195.5
Total	16.7	1416.3	100.5	75.4	1608.9
Effluents, g					
Raw Agglomerates	0.0	51.9	68.0	63.0	182.9
Raw Tailings	0.0	6.1	6.3	14.0	26.4
Filtrate/Rinse Water	16.7	1345.6	16.9	0.0	1379.2
Total	16.7	1403.6	91.2	77.0	1588.5
Closure, wt%	100.0	99.1	90.7	102.1	98.7

TABLE B-7

Spring Creek Agglomeration Balance Test 1
Raw Agglomerate Oil Calculation Method 2

Component	HNO_3	Water	Oil	Solids	Total
Influents, g					
Coal	0.0	24.6	0.0	75.4	100.0
Acid Solution	16.7	190.5	0.0	0.0	207.2
Agglomerant	0.0	5.7	100.5	0.0	106.2
Wash/Rinse Water	0.0	1195.5	0.0	0.0	1195.5
Total	16.7	1416.3	100.5	75.4	1608.9
Effluents, g					
Raw Agglomerates	0.0	51.9	66.8	64.2	182.9
Raw Tailings	0.0	6.1	6.3	14.0	26.4
Filtrate/Rinse Water	16.7	1345.6	16.9	0.0	1379.2
Total	16.7	1403.6	90.0	78.2	1588.5
Closure, wt%	100.0	99.1	89.6	103.7	98.7

FORM EIA-460E
(10/80)

U.S. DEPARTMENT OF ENERGY

FEDERAL ASSISTANCE MANAGEMENT SUMMARY REPORT

FORM APPROVED
OMB NO 1900-0127
Page 1 of 2

1. Program/Project Identification No. DE-FC21-86MC10637
2. Program/Project Title Beneficiation of Low-Rank Coal (4.2)
3. Reporting Period 4-1-92 through 6-30-92
4. Name and Address Energy and Environmental Research Center
University of North Dakota
Box 8213, University Station, Grand Forks, ND 58202 (701) 777-5000
5. Program/Project Start Date 4-1-86
6. Completion Date 9-30-92

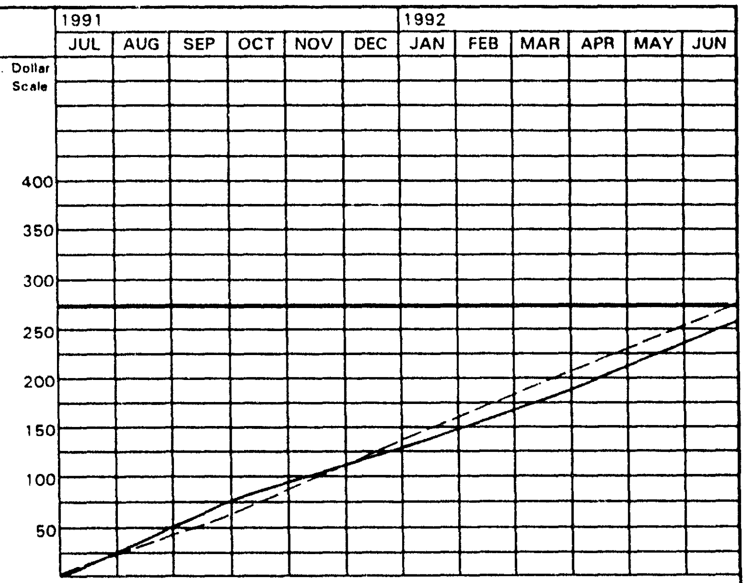
7. FY 91/92
8. Months or Quarters Quarters
9. Cost Status a. Dollars Expressed in Thousands b. Dollar Scale

10. Cost Chart

Fund Source		Quarter 1st	2nd	3rd	4th	Cum. to Date	Tot Plan
DOE	P	68	69	69	69	275	275
	A	76	51	56	75	258	
	P						
	A						
	P						
	A						
	P						
	A						
Total P		68	69	69	69	275	275
Total A		76	51	56	75	258	
Variance		(8)	18	13	(6)	17	

P = Planned A = Actual

Total Planned Costs for Program/Project $275

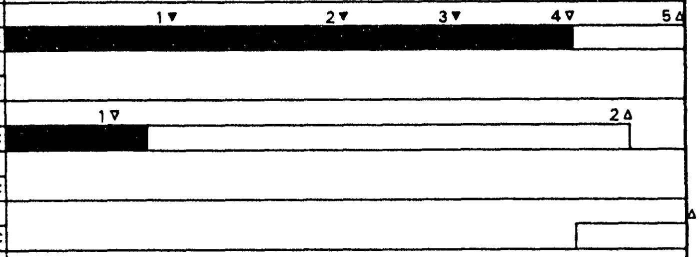


c. Cumulative Accrued Costs

	JUL	AUG	SEP	OCT	NOV	DEC	JAN	FEB	MAR	APR	MAY	JUN
Planned			68			137			206			275
Actual			76			127			183			258
Variance			(8)			10			23			17

11. Major Milestone Status

	Units Planned / Units Complete
A. Oil Agglomeration	P C
B. Additional Cleaning Techniques	P C
C. Reports and Briefings--Final Report	P C



12. Remarks

13. Signature of Recipient and Date
Raymond DeWall 7/30/92

14. Signature of DOE Reviewing Representative and Date

FORM E1A-459E
(10/80)

U.S. DEPARTMENT OF ENERGY

FEDERAL ASSISTANCE MANAGEMENT SUMMARY REPORT

FORM APPROVED
OMB NO. 1900 0127

Page 2 of 2

1. Program/Project Identification No. DE-FC21-86MC10637	2. Program/Project Title Beneficiation of Low-Rank Coals (4.2)	3. Reporting Period 1-1-92 through 3-31-92
4. Name and Address	Energy and Environmental Research Center University of North Dakota Box 8213, University Station Grand Forks, ND 58202 (701) 777-5000	5. Program/Project Start Date 4-1-86 6. Completion Date 6-30-92

Milestone ID. No.	Description	Planned Completion Date	Actual Completion Date	Comments
Task A	Oil Agglomeration:			
a.1	Agglomeration with polar oil and carrier solvent	9-30-91	9-30-91	
a.2	Recycle and makeup requirements	12-31-91	1-31-92	
a.3	Process modifications	2-29-92	4-15-92	
a.4	Mass agglomerate formation and deoiling techniques	4-30-92		
a.5	Development of agglomerate stability analysis techniques	6-30-92		
Task B	Additional Cleaning Techniques:			
b.1	Froth flotation	8-31-91		
b.2	Colloidal coal cleaning	5-31-92		
Task C	Reports and Briefings--Final Report	7-31-92		