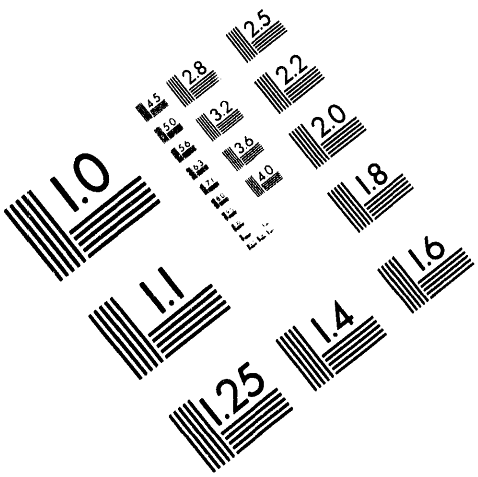

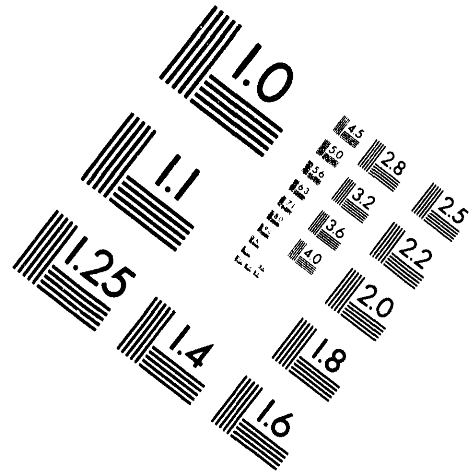

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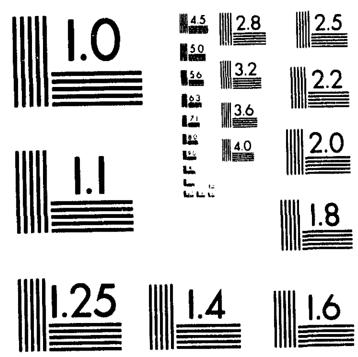

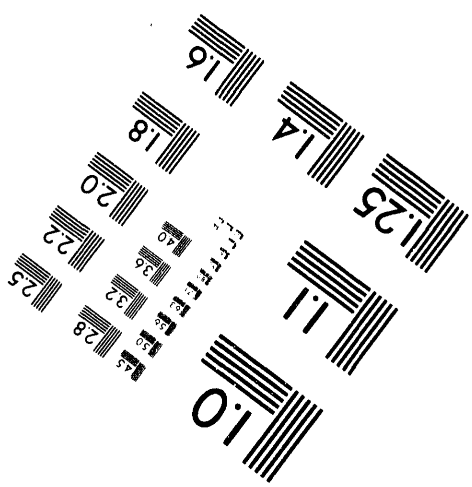

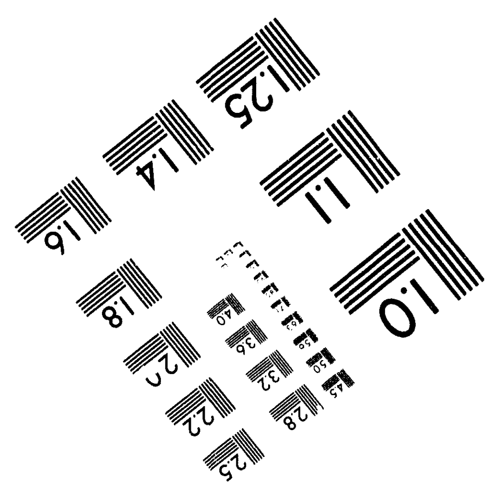

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DOE/PC/92521--T178

TECHNICAL REPORT
March 1 through May 31, 1994

Project Title: **BENCH-SCALE DEVELOPMENT OF MILD GASIFICATION CHAR DESULFURIZATION**

DOE Grant Number:	DE-FC22-92PC92521 (Year 2)
ICCI Project Number:	93-1/1.2B-3M
Principal Investigator:	Richard A. Knight, Institute of Gas Technology
Project Manager:	Dan Banerjee, Illinois Clean Coal Institute

ABSTRACT

The goal of this project is to scale up a process, developed under a previous ICCI grant, for desulfurization of mild gasification char by treatment with hydrogen-rich process-derived fuel gas at 650°-760°C and 7-15 atm. The char can be converted into a low-sulfur metallurgical form coke. In the prior study, IBC-105 coal with 4.0 wt% sulfur was converted to chars with less than 1.0 wt% sulfur in a laboratory-scale batch reactor. The susceptibility of the char to desulfurization was correlated with physicochemical char properties and mild gasification conditions. Acid pretreatment of the coal prior to mild gasification was also shown to significantly enhance subsequent sulfur removal.

In this study, IGT is conducting continuous bench-scale tests in a 1-lb/h fluidized-bed reactor to determine the preferred process conditions and obtain steady-state data necessary for process design and scale-up. The desulfurized chars are to be used to produce low-sulfur form coke, which will be evaluated for density, reactivity, and strength properties relevant to utilization in blast furnaces.

This quarter, 2500 g of mild gasification char was produced from untreated IBC-105 coal in the bench-scale reactor. Half of this char will be subjected to sulfuric acid treatment to enhance subsequent desulfurization. Char-producing runs were also initiated with acid-pretreated coal, which will produce about 1250 g of char.

During the next quarter, the char production runs will be completed, char desulfurization will be performed, form coke briquettes will be produced and evaluated, and a conceptual process flow diagram will be produced based on the bench-scale test data.

EXECUTIVE SUMMARY

The Institute of Gas Technology (IGT) is conducting a bench-scale project to develop the conversion of high-sulfur Illinois No. 6 coal to a low-sulfur mild gasification char, which in turn is converted to low-sulfur form coke. Mild gasification produces solid, liquid, and gaseous co-products at conditions of low severity (540°-700°C, <4.5 atm). The MILDGAS process, developed by IGT under U.S. DOE sponsorship (DOE Contract DE-AC21-87MC24266), uses a coaxial fluidized-bed/entrained-bed reactor system.

Possible uses for mild gasification char include: form coke for steel-making, form coke for foundries, carbon-based adsorbents, smokeless fuel, and boiler fuel for power generation. Form coke is particularly attractive in price and marketability. The value of coke for blast furnaces and foundries increases as sulfur content decreases, and a sulfur level of about 1 wt% or less is typical for conventional coke.

In the MILDGAS development project, a conventionally washed Illinois coal with about 3.5 wt% sulfur produced char ranging from 2.1 wt% to 2.8 wt% sulfur, which was 20-40% lower than that of the coal. Further desulfurization of the char required to yield an attractive product for form coke production can be accomplished by desulfurization with process-derived reducing gases. The desulfurization can be performed on the char particles exiting the mild gasifier and, if desired, again in the carbonization step that converts the cured briquettes into form coke.

Research performed in the 1970's at IGT,[1] U.S. Steel,[2,3] Garrett Research,[4] and elsewhere, has shown that coal chars from various types of gasifiers can be effectively desulfurized by exposure to reducing gases at temperatures of 1100°-1500°F (590°-815°C). Mixtures of H_2 and CH_4 are effective for this purpose, and the presence of CH_4 can inhibit carbon hydrogasification, thus allowing desulfurization to proceed with minimal carbon losses, compared to treatment with pure H_2. The product gas from

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1 Fleming, D.K., R.D. Smith, and M.R.Y. Aquino, *ACS Div. of Fuel Chem. Prepr.* **22:2**, 45-49 (1977)

2 Boodman, N.S., T. F. Johnson, and K.C. Krupinski, *ACS Div. of Fuel Chem. Prepr.* **22:2**, 28-44 (1977).

3 Kor, G.J.W., *ACS Div. of Fuel Chem. Prepr.* **22:2**, 1-27 (1977)

4 Robinson, L., *Fuel* **55:3**, 193-201 (1976)

mild gasification, using enriched-air partial combustion for heat supply, contains 10-20 vol% CH_4 and 16 to 49% H_2, depending on gasifier temperature and off-gas conditioning methods.

In September 1991, IGT began an ICCI-funded study of char desulfurization on a laboratory scale. Early tests showed that the sulfur content of chars could be reduced by exposure to CH_4/H_2 mixtures. However, char preparation conditions strongly affected the susceptibility of the char to desulfurization. Also, data from Garrett Research showed that acid-washing of char to remove Ca- and Fe-containing minerals strongly increased the susceptibility of char to subsequent desulfurization.

Chars were then produced in the laboratory from a single coal (IBC-105) under controlled conditions in the IFFR and a mini-bench fluidized-bed reactor (MBFBR), and acid pretreatment of the coal and/or char were investigated. By a combination of acid pretreatment, mild gasification, and CH_4/H_2 treatment at 1400°F (760°C) and 200 psig (14.6 atm), we converted IBC-105 coal to low-sulfur chars. The original sulfur content of the parent coal was 4.1-4.3 wt%, and the mild gasification chars prior to desulfurization contained 2.1-3.6 wt% sulfur. Desulfurization with CH_4/H_2 mixtures resulted in sulfur removal of 28-95%, with char sulfur contents of 0.1-2.7 wt% and carbon losses of 5-29%.

The findings of the previous study can be summarized as follows:

- Acid-washing of either the coal prior to mild gasification *or* of the char prior to desulfurization increases desulfurization susceptibility by 50%-84%
- Micropore collapse occurs during entrained (IFFR) mild gasification, but does not occur in the fluidized bed (MBFBR), and therefore the microporous surface area plays a significantly greater role in the desulfurization of fluidized-bed chars, increasing sulfur removal.
- Lower mild gasification temperature (540°C versus 620°-700°C) generally favors the subsequent desulfurization, but also results in greater carbon consumption.
- The data suggest that an optimum combination of H_2:CH_4 ratio and system pressure to obtain maximum sulfur removal with minimum carbon loss can be achieved.
- Fluidized-bed (MBFBR) chars are more easily desulfurized than entrained (IFFR) chars

- Form coke briquettes prepared from IFFR chars using a mild gasification pitch binder displayed tensile strengths similar to commercial cokes
- Form coke briquettes carbonized in an H_2:CH_4 atmosphere lost 22-48% more sulfur than those carbonized in nitrogen

The current study builds on the positive results of the laboratory-scale work. The objectives are to conduct bench-scale tests to confirm the effectiveness of the process under steady-state conditions, identify preferred conditions for the desulfurization step, produce and test low-sulfur form coke, and develop data for possible scale-up of the desulfurization step in coordination with ongoing mild gasification technology development programs. Fluidized-bed mild gasification char will be produced from an IBCSP coal, IBC-105, with and without acid pretreatment, in the MBFBR.

The 2-inch-ID screw-fed MBFBR will then be used for the continuous char desulfurization tests. The mild gasification chars will be treated with simulated recycled fuel gas mixtures containing H_2, CH_4, CO, CO_2, and N_2 at 620°-760°C, 4.4-14.6 atm, and residence times of 30-120 minutes. The desulfurized chars will be evaluated for sulfur and carbon conversion to gas. Key microstructural properties (internal surface area, pore volume, and helium density, and mean crystallite size) will also be evaluated for correlations with desulfurization susceptibility. Form coke briquettes will then be produced from the desulfurized chars and evaluated for density, diametral tensile strength, and CO_2 reactivity. The data obtained from the program will be used to develop a desulfurization block flow diagram.

In the third quarter, mild gasification of untreated IBC-105 coal was completed in the MBFBR. The char was produced in four passes through the reactor at 1100°F (590°C) and 20 psig (2.3 atm), using a 1:4 mass ratio of fresh coal to char. The starting char diluent was produced in a fixed-bed carbonizer, and the product from each pass was used as a diluent for the subsequent pass. In this manner, 2500 g of mild gasification char entirely derived from the parent coal was produced.

Char production runs with acid-pretreated coal were also begun at the end of the quarter. Approximately 1250 g of char will be produced from this feedstock after four passes through the MBFBR at conditions identical to those used for the untreated coal.

Half (1250 g) of the mild gasification char produced from untreated coal will be subjected to acid treatment to remove

a portion of alkaline minerals which inhibit subsequent desulfurization. As was done with a portion of the parent coal, the char will be leached with 1.0*N* sulfuric acid at 200°F (93°C) for five minutes, rinsed with deionized water, and vacuum-dried.

During the final quarter, mild gasification of acid-treated IBC-105 coal and acid treatment of half of the char from untreated coal will be completed. Char desulfurization tests will be performed on all three of these chars. Selected samples of the desulfurized chars will be briquetted into form coke, which will then be evaluated for strength, density, and reactivity. Finally, a conceptual process flow diagram will be produced, based on the bench-scale test data.

OBJECTIVES

The ultimate goal of this project is to develop a method for desulfurization of mild gasification char using process-derived gases. The objectives of the 24-month program are to conduct laboratory studies that assess the technical viability of the process, develop a preliminary process flow scheme, and make recommendations for integration of the desulfurization step into an ongoing mild gasification development program.

The task structure of the current project year is as follows:

- Task 1. Coal Preparation and Characterization
- Task 2. Coal Pretreatment
- Task 3. Mild Gasification
  - Subtask 3.1. Equipment Preparation and Shakedown
  - Subtask 3.2. Char Production Runs
- Task 4. Char Desulfurization
  - Subtask 4.1. Equipment Preparation and Shakedown
  - Subtask 4.2. Bench-Scale Desulfurization Tests
- Task 5. Form Coking
  - Subtask 5.1. Briquetting
  - Subtask 5.2. Briquette Carbonization
- Task 6. Data Analysis and Interpretation
- Task 7. Process Scale-Up Design

INTRODUCTION AND BACKGROUND

Mild gasification is an advanced coal carbonization process emphasizing simple reactor and process design and low-severity processing conditions to produce value-added co-products. The U.S. DOE has been supporting the development of this technology since 1987, including the MILDGAS process, developed by a project team consisting of Peabody Holding Company, Bechtel National, and IGT. Under that project, a 100-lb/h (45-kg/h) PRU was built and operated for 47 tests.[5,6,7,8] A subsequent team led by Kerr-McGee Coal Corporation has designed a 24-ton/day (22-tonne/day) MILDGAS PDU which is slated to be built at SIUC in the near future. The MILDGAS reactor consists of a coaxial fluidized-bed/entrained-bed vessel which can process all types of coals.

A major target market for mild gasification char is metallurgical form coke for blast furnaces and foundries. Although there are no formalized guidelines for form coke sulfur content, a target sulfur limit of 1.0 wt% has been selected for this project.

In the 1970's, U.S. Steel developed the Clean Coke process, in which desulfurization of the char with recycled product gas played an important role.[2] The recycle gas used for fluidization contained H_2, CO, CH_4, and higher hydrocarbons. At 1400°F (760°C), char was reduced in sulfur content from 1.7 wt% to 0.3 wt%, which is an 82% reduction. The H_2S concentration in the recycle gas was identified as a critical factor in achieving effective desulfurization. An associated study of char-sulfur chemistry[3] with an Illinois No. 6 hydrogasification char determined that, while H_2 is more effective than CH_4 for char desulfurization, mixtures of CH_4 and H_2 were also effective and, furthermore, the presence of CH_4 inhibited carbon gasification while allowing desulfurization to continue.

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5 Wootten, *et al.*, Task 1 Topical Report to DOE/METC, Contract DE-AC21-87MC24266, (1988).

6 Knight, R.A., *et al.*, Task 2 Topical Report to DOE/METC, Contract DE-AC21-87MC24266, (1990).

7 Carty, R.H., *et al.*, Task 3 Topical Report to DOE/METC, Contract DE-AC21-87MC24266, (1990).

8 Knight, R.A., *et al.*, Task 4 Topical Report to DOE/METC, Contract DE-AC21-87MC24266, (1990).

In similar work, IGT studied the hydrodesulfurization of four bituminous coals at 1300°-1500°F (700°-815°C) for 30 minutes in hydrogen.[1] A reduction of 74% in the sulfur content per unit energy was achieved with Illinois No. 6 coal. This study emphasized the importance of maintaining a low H_2S concentration in the treatment gas.

Research on char hydrodesulfurization was also performed by Garrett Research and Development (later Occidental Petroleum). A 1976 paper[4] discussed the effectiveness of subjecting char to acid-washing to remove Ca- and Fe-containing compounds, which appear to contribute to the severity of H_2S inhibition. Although the mechanism responsible for this effect was not fully determined, the H_2 requirement to desulfurize an acid-washed char was 88% lower than that required for an untreated char.

The present study is evaluating the technical feasibility of desulfurizing mild gasification char and/or form coke briquettes with process-derived reducing gases. The product gas from mild gasification, on an inert-free basis, contains 20 to 28 vol% CH_4 and 28 to 50% H_2, where the H_2:CH_4 mole ratio increases monotonically from about 0.4 at 1100°F (590°C) to 1.0 at 1300°F (700°C).[6] The application of this technique to mild gasification char and/or briquettes, and the integration of the technique with form coking, had not previously been studied until IGT began an ICCI-sponsored laboratory-scale project two years ago. Early tests showed that the sulfur content of chars could be reduced by exposure to CH_4/H_2 mixtures. However, char preparation conditions strongly affected the susceptibility of the char to desulfurization. Chars were then produced in the laboratory from a single coal (IBC-105) under controlled conditions in the IFFR and a mini-bench fluidized-bed reactor (MBFBR), and acid pretreatment of the coal and/or char, based on the Garrett Research results,[4] were investigated. By a combination of acid pretreatment, mild gasification, and CH_4/H_2 treatment at 1400°F (760°C) and 14.6 atm, we converted IBC-105 coal to low-sulfur chars. The original sulfur content of the parent coal was 4.1-4.3 wt%, and the mild gasification chars prior to desulfurization contained 2.1-3.6 wt% sulfur. Desulfurization with CH_4/H_2 mixtures resulted in sulfur removal of 28-95%, with char sulfur contents of 0.1-2.7 wt% and carbon losses of 5-29%.

In form coke production, char is blended either with fresh coal which supplies an *in-situ* binder for briquetting (binderless briquetting), or with a coal-derived pitch binder. The blend is heated to a plastic mass and formed

into briquettes in a hot roller press. The resulting "green" briquettes are then calcined in a shaft furnace at about 1800°F (980°C). The relative feasibility and impact on overall process efficiency of desulfurization before and/or after briquetting must be evaluated.

The findings of the previous ICCI-sponsored study can be summarized as follows:

- Acid-washing of either the coal prior to mild gasification *or* of the char prior to desulfurization increases desulfurization susceptibility by 50%-84%.
- Micropore collapse occurs during entrained (IFFR) mild gasification, but does not occur in the fluidized bed (MBFBR), and therefore the microporous surface area plays a significantly greater role in the desulfurization of fluidized-bed chars.
- Lower mild gasification temperature (540°C versus 620°-700°C) generally favors the subsequent desulfurization, but also results in greater carbon conversion.
- The data suggest that an optimum combination of H_2:CH_4 ratio and system pressure to obtain maximum sulfur removal with minimum carbon loss can be achieved.
- Fluidized-bed (MBFBR) chars are more easily desulfurized than entrained (IFFR) chars.
- Form coke briquettes prepared from IFFR chars using a mild gasification pitch binder displayed tensile strengths similar to commercial cokes.
- Form coke briquettes carbonized in an H_2:CH_4 atmosphere lost 22-48% more sulfur than those carbonized in nitrogen.

EXPERIMENTAL PROCEDURES

Task 1. Coal Preparation and Characterization

The test coal for this program is IBC-105, an Illinois No. 6 channel lot collected and processed under inert gases. The coal contains about 4.5% sulfur, about evenly divided between pyritic and organic. This is the same coal studied in the previous laboratory-scale char desulfurization project.

Approximately 180 lb (82 kg) of IBC-105 coal is to be obtained from the IBCSP and crushed, dried, and sized to 40×80-mesh. Based on past experience, it is expected that the processing will yield about 50-60 lb (23-27 kg) of this size fraction. Initial sizing is done on an 18" Sweco rotary screener, and the sample is then screened further on a Rotap shaker to obtain a tight cut.

The test coal sample is then riffled and subjected to proximate and ultimate analyses, heating value determination, mineral matter analysis, and determination of physical properties (connected pore volume, internal surface area, helium density, and mean crystallite size).

Task 2. Coal Pretreatment

The test coal is washed with 1*N* sulfuric acid to enhance desulfurization susceptibility. This method has been successfully employed in the current laboratory study upon which this proposal is based. Half of the 40×80-mesh coal prepared in Task 1 is to be pretreated in this way, yielding about 12 lb (5.4 kg) of 40×80-mesh acid-washed coal. The pretreated sample is to be analyzed in the same way as the untreated 40×80-mesh coal fraction.

Based on discussions during the last quarter between the Principal Investigator and the ICCI Project Manager, it was decided that acid treatment of the mild gasification char from untreated coal will be also performed prior to char desulfurization.

Task 3. Mild Gasification

This task is divided into two subtasks as follows.

Subtask 3.1. Equipment Preparation and Shakedown

Under this Subtask, an acid-washing setup is to be constructed to efficiently treat 13 lb (5.9 kg) of the test coal 40×80-mesh fraction.

The existing MBFBR system, shown in Figure 1, is to be modified to accommodate mild gasification of larger batches of coarse-fraction caking coal than have been used in the past. The present configuration is limited to processing about 0.44 lb (200 g) of coal per run at a rate of 0.18 lb/h (80 g/hr), with the limitations being related to the buildup of agglomerated material at the end of the feed screw and around the feed inlet.

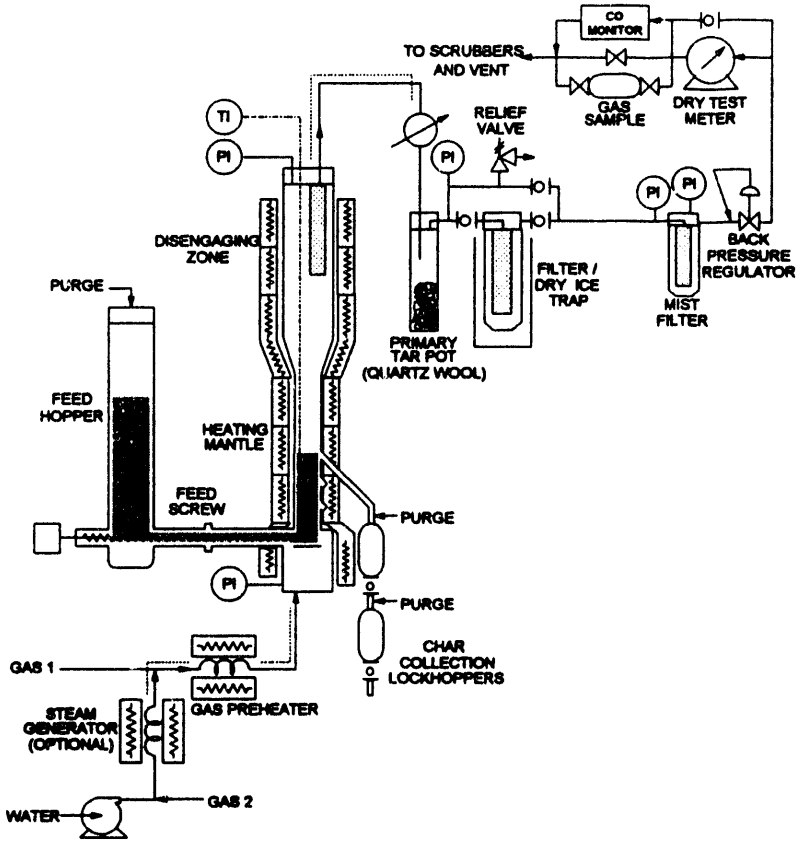


Figure 1. SCHEMATIC DIAGRAM OF MINI-BENCH FLUIDIZED-BED REACTOR (MBFBR)

The desired modifications involve:

- Replacement of a sintered metal fluidizing plate with a perforated fluidizing plate
- Installation of sweep gas purges near the end of the feed screw
- Installation of a cooling jacket around the feed screw, and
- Modification of the feed screw to prevent agglomeration at the screw tip

Routine recalibration of instruments and shakedown are also to be performed under this task.

Subtask 3.2. Char Production Runs

Mild gasification chars from untreated and acid-washed 40×80-mesh coal are to be produced in the MBFBR. The initial run segment uses an IBC-105 char produced in a fixed-bed carbonizer as a diluent. The "starter" char from the fixed-bed carbonizer is produced at 1200°F (650°C) and atmospheric pressure. The -80 mesh rejects (unders) from test coal sizing is to be carbonized for this purpose. The coal is to be loaded into the carbonizer, which is then heated slowly (approximately 5°F/min) to 1200°F (650°C), and then soaked for 60 minutes at constant temperature. The resulting agglomerated char is then manually crushed and screened to obtain a 40×80-mesh fraction.

For char production, the test coal, mixed in a 1:4 weight ratio with "starter" char, is to be fed into the MBFBR at 1.8-2.0 lb/h (0.82-0.91 kg/h). Each run segment processes 1.5-1.6 lb (0.68-0.73 kg) of fresh coal. The char from each subsequent run segment uses the recovered char from the previous run segment as a bed diluent. This approach produces an unadulterated mild gasification char from 100% IBC-105 coal. About 6.0-6.5 lb (2.7-2.9 kg) of each feed (untreated and acid-treated) is to be processed to produce about 4.0-4.2 lb (1.8-1.9 kg) of char. The MBFBR char production requires about 16 h of total operation in four four-hour segments for each feed. The mild gasification char production conditions are nominally 1100°F (593°C) and 20 psig (0.24 MPa).

Task 4. Char Desulfurization

This task is divided into two subtasks as shown below.

Subtask 4.1. Equipment Preparation and Shakedown

For char desulfurization tests, the MBFBR requires replacement of the 1200°F (650°C) heating mantle used for mild gasification with an existing 2000°F- (1090°C)-rated clamshell furnace. The 2-inch-ID section of the reactor containing the char overflow ports will be removed and replaced with a straight pipe section. A central char overflow standpipe which was used prior to modifications for mild gasification will be reinstalled, along with a bed distributor plate with a center hole to accommodate the standpipe. Routine instrument calibrations for the char feedstocks and the higher pressure operation are to be completed under this Subtask. The unit is to be pressure-tested to 15 atm for each test, and the proper operation of the two-zone clamshell furnace is verified. The existing gas delivery equipment necessary to feed H_2 and a CH_4:CO:N_2:CO_2 mixture at controlled rates is to be tested and calibrated.

Subtask 4.2. Bench-Scale Desulfurization Tests

Char desulfurization experiments are to be conducted in the MBFBR to determine the effects of temperature, pressure, residence time, and gas composition on the char sulfur content. A total of 12 char desulfurization experiments are planned for the 12-month program. The planned ranges of parameters are shown in Table 1.

Table 1. PARAMETERS FOR PLANNED CHAR DESULFURIZATION TESTS

Coal pretreatment	None, Acid-wash (1*N*, 5 min, 80°C)
Temperature	1200 - 1400°F (649 - 760°C)
H_2 partial pressure	40 - 60 psi (0.28 - 0.41 MPa)
Residence time	30 - 60 minutes

Task 5. Form Coking

This task is divided into two subtasks as shown below.

Subtask 5.1. Briquetting

Briquettes are to be produced from desulfurized char by techniques which have been developed for mild gasification char in previous programs. Char is mixed with a crude pitch derived from Illinois No. 6 PRU mild gasification liquids. The mixture is then compressed into a 1.0×0.25-inch (2.54×0.64-cm) disk at 600°-650°F (315°-345°C) to form the "green" briquette. It is anticipated that briquettes will be prepared from each char produced in Task 4 with a sulfur content of 1% or less, and that a total of about 24 briquettes will be prepared.

Subtask 5.2. Briquette Carbonization

Briquettes prepared in Subtask 5.1 are to be carbonized at 1700°-2000°F (930°-1090°C) to fully devolatilize and develop tensile strength, producing form coke. Selected briquettes are carbonized under various conditions of temperature and gas atmosphere. Approximately eight tests are to be conducted. Form coke briquettes are then tested for chemical composition, diametral tensile strength, density, and CO_2 reactivity, and the values obtained compared to those for a conventional coke sample.

Task 6. Data Analysis and Interpretation

Samples are analyzed and tested under this Task. The analyses to be performed are summarized as follows:

Untreated coal and acid-washed coal --

Proximate and ultimate analysis
Sulfur by type
Particle size distribution (sieve analysis)
Connected pore volume (Hg porosimetry)
CO_2 surface area
Helium density
Mean crystallite size (XRD)

Raw and desulfurized mild gasification char --

Proximate and ultimate analysis
Particle size distribution (sieve analysis)
Connected pore volume (Hg porosimetry)
CO_2 surface area
Helium density
Mean crystallite size (XRD)

Cured briquettes --

Proximate and ultimate analysis
Briquette density

Carbonized form coke briquettes --

Proximate and ultimate analysis
Briquette density
Diametral tensile strength
CO_2 reactivity (Bethlehem Steel test)

Desulfurization exit gases --

GC/TCD/FID syngas analysis (H_2, CO_x, N_2, O_2/Ar, C_1-C_6)
GC/FPD sulfur gas analysis (H_2S, COS)

Data Interpretation will be in the form of material balances, assessment of sulfur removal and carbon loss during desulfurization, physical property correlations with desulfurization, and briquette performance properties.

Task 7. Process Scale-Up Design

The data from the bench-scale testing are to be used to develop a material- and energy-balanced block flow diagram (BFD). The BFD will also be reviewed by process engineers at Kerr-McGee Coal Corporation (KM Coal), which is the team leader for the DOE-sponsored MILDGAS PDU program. Recommendations will be made, in concert with KM Coal, for possible scale-up of the desulfurization process and coordination of scale-up operations with activities in the PDU program.

RESULTS AND DISCUSSION

Task 1. Coal Preparation and Characterization

This task was completed in a previous quarter.

Task 2. Coal Pretreatment

This task was completed in a previous quarter.

Task 3. Mild Gasification

Subtask 3.1. Equipment Preparation and Shakedown

The planned MBFBR modifications to accommodate extended caking coal operation were completed during previous quarters. However, additional modifications were found necessary to allow a steady discharge of char from the reactor. Even with the use of a diluent char, the presence of very soft agglomerates in the bed periodically obstructed the char overflow, eventually leading to increased bed height and, ultimately, to defulidization and failure of the feed screw coupling. This was addressed by enlarging the 45° discharge port to a size of one inch (2.5 cm) and modifying the piping to eliminate bridging. The problem was reduced to a manageable level by this approach.

Several modifications will be needed for the desulfurization tests. These will include cutting out the 2-inch reactor pipe with side discharge used for char preparation, welding a 2-inch pipe with a central overflow tube in place for desulfurization tests, and replacement of the heating mantle with a clamshell-type electric furnace capable of reaching temperatures above 1400°F (760°C). Changes needed for the char desulfurization tests will be completed after the acid-washed coal char has been prepared.

Subtask 3.2. Char Production Runs

Operation of the MBFBR for production of two test batches of mild gasification char continued this quarter. Char production from untreated 40×80-mesh untreated IBC-105 coal was completed with approximately 2500 g of mild gasification char generated. A smaller batch of char (approximately 1250 g) is being produced from acid-washed IBC-105 coal at the same mild gasification conditions. These char

production conditions are 1100°F (590°C) with 20 psig (2.3 atm) nitrogen fluidizing gas at 0.49 ft/sec (0.19 m/s). The coal:char mixture feed rate of 6 g/min results in a 40-minute solids residence time.

Mild gasification char was generated from untreated IBC-105 coal though four passes in the MBFBR. Feed for the first pass was composed of 80 wt% starter char, which was previously prepared by slow heating in a fixed-bed carbonizer, and 20 wt% coal. In each subsequent pass, the feed consisted of 80 wt% char from the previous pass and 20 wt% coal. The first pass feed was comprised of 1506 g of starter char and 377 g of coal. By the end of the fourth pass, 2500 g of char had been produced. In the four passes, 1989 g of coal was fed along with the initial 1506 g of starter char.

Mild gasification of the acid-pretreated coal has just begun. No data is yet available on the results of these runs.

Task 4. Char Desulfurization

No char desulfurization tests were performed in this quarter. This task will be activated next quarter when all of the char from untreated and acid-washed coal prepared in Task 3 are both available. Char preparation and desulfurization are conducted in different configurations of the MBFBR reactor. The minor changes needed to perform desulfurization tests will be quickly completed prior to beginning the tests.

Task 5. Form Coking

No form coking tests were conducted this quarter. Form coking work will use desulfurized char produced in the Task 4 desulfurization tests. Form coke tests will be performed next quarter.

Task 6. Data Analysis and Interpretation

Each mild gasification pass in the MBFBR produced, on average, 13.5 wt% more product char than feed (diluent) char. Since 20 wt% of the feed was untreated coal and diluent char can be assumed to be unreactive, the average weight loss was estimated as 46% of the feed coal. This weight loss corresponds to a char yield from the coal of

approximately 54 wt% on a total weight basis. This char yield will also be verified by char analysis, using the total ash balance.

Mild gasification char analyses are not yet available. These analyses will be reported in the annual report.

Task 7. Process Scale-Up Design

No work was done on this task during the current quarter.

CONCLUSIONS AND RECOMMENDATIONS

Production of mild gasification char from untreated coal was completed this quarter. This char was produced in a continuous fluidized-bed reactor at 1100°F (590°C) and 20 psig (2.3 atm) in nitrogen.

In the next quarter, char will be produced from acid-washed coal, and a portion of the char from untreated coal will also be acid-washed to enhance desulfurization susceptibility. Bench-scale desulfurization tests will also proceed next quarter along with form coke preparation and testing.

This work was prepared with the support, in part, by grants made possible by the Illinois Department of Energy and Natural Resources through its Coal Development Board and Illinois Clean Coal Institute, and by the U.S. Department of Energy. However, any opinions, findings, conclusions, or recommendations expressed herein are those of the author(s) and do not necessarily reflect the views of IDENR, ICCI, and the DOE.

PROJECT MANAGEMENT REPORT
March 1 through May 31, 1994

Project Title: **BENCH-SCALE DEVELOPMENT OF MILD GASIFICATION CHAR DESULFURIZATION**

DOE Grant Number:	DE-FC22-92PC92521 (Year 2)
ICCI Project Number:	93-1/1.2B-3M
Principal Investigator:	Richard A. Knight, Institute of Gas Technology
Project Manager:	Dan Banerjee, Illinois Clean Coal Institute

COMMENTS

No changes were made to budget and management in the third quarter.

Projected and Estimated Actual Expenditures by Quarter

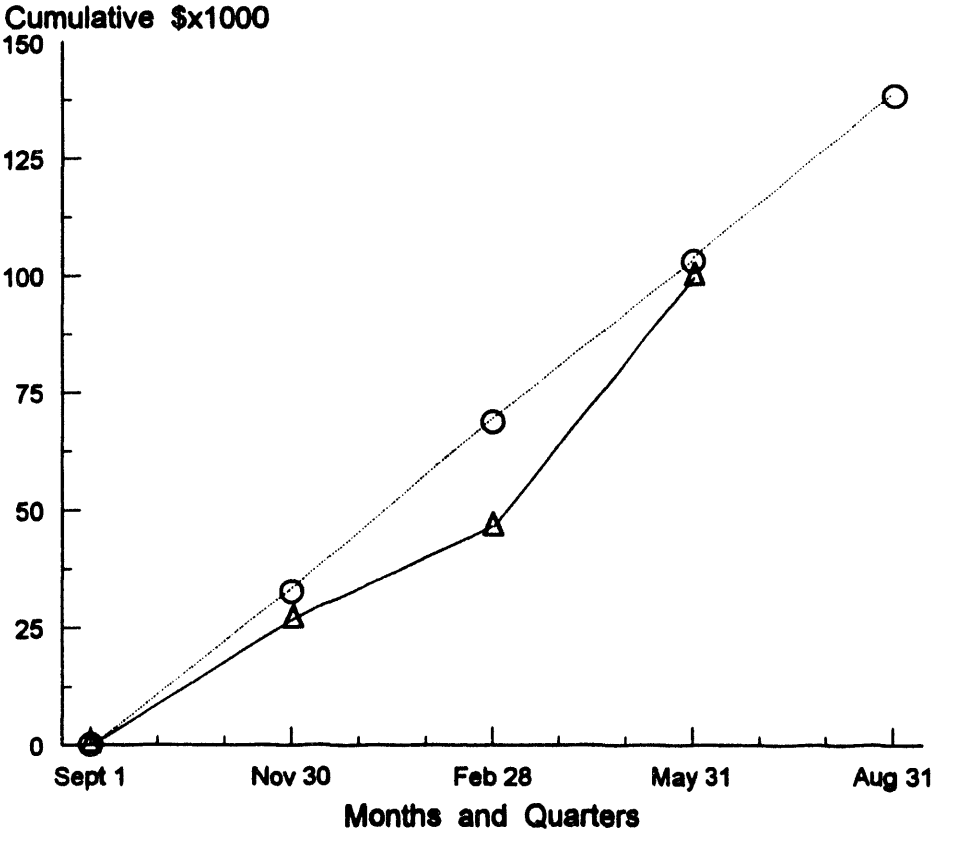
BENCH-SCALE DEVELOPMENT OF MILD GASIFICATION CHAR DESULFURIZATION

Quarter*	Types of Cost	Direct Labor	Fringe Benefits	Materials & Supplies	Travel	Major Equipment	Other Direct Costs	Indirect Costs	Total
Sept 1, 1993 to Nov 30, 1993	Projected	10,480		400				18,511	29,391
	Estimated	7,919		924				14,118	22,961
Sept 1, 1993 to Feb 28, 1994	Projected	24,290		2,400		1,300		43,505	71,495
	Estimated	16,285		1,521		0		28,951	46,757
Sept 1, 1993 to May 31, 1994	Projected	35,447		4,150		1,300		63,500	104,397
	Estimated	34,667		5,711		109		62,197	102,684
Sept 1, 1993 to Aug 31, 1994	Projected	46,680		5,505	260	1,300		83,581	137,326
	Estimated								

* Cumulative by Quarter

COSTS BY QUARTER

BENCH-SCALE DEVELOPMENT OF MILD GASIFICATION
CHAR DESULFURIZATION

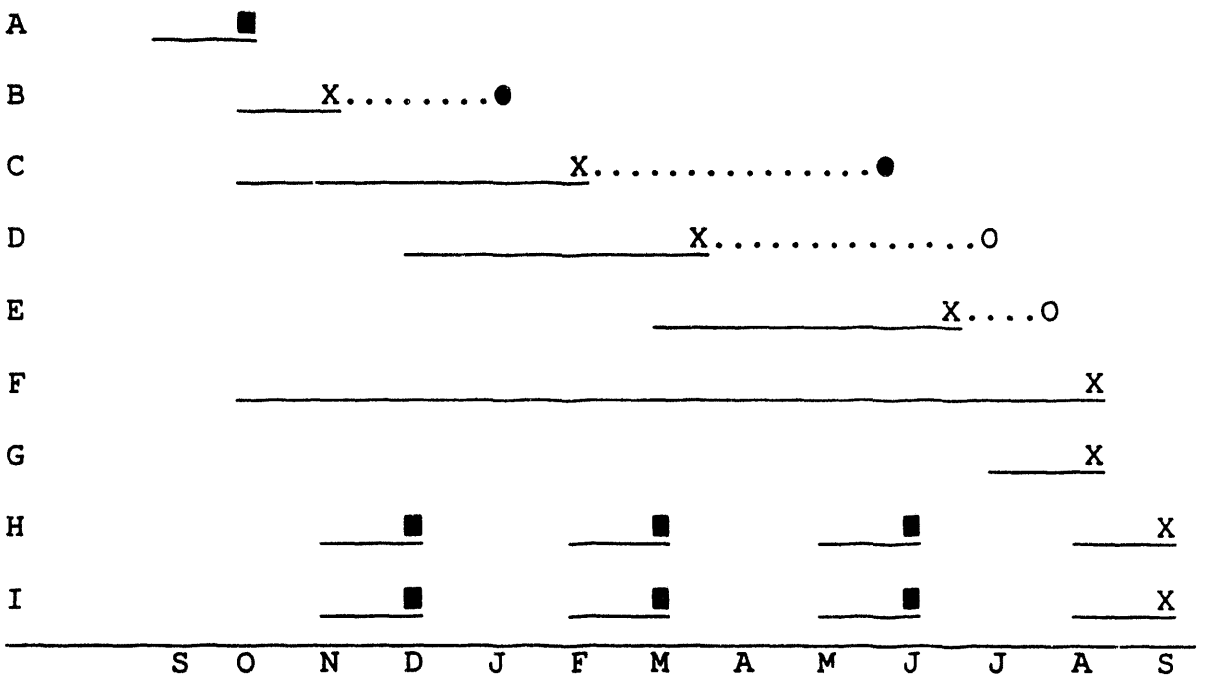


Projected Expenditures

Estimated Actual Expenditures

Illinois Clean Coal Institute Award $137,326

SCHEDULE OF PROJECT MILESTONES



Begin
Sept. 1
1993

Hypothetical Milestones:

A. IBC-105 coal sample prepared and characterized (Task 1)
B. Acid-washing of coal completed (Task 2)
C. Mild gasification char produced (Task 3)
D. Char desulfurization tests completed (Task 4)
E. Form coke briquettes produced and evaluated (Task 5)
F. Bench-scale test data analyzed and interpreted (Task 6)
G. Process block flow diagram updated (Task 7)
H. Quarterly and annual technical reports prepared and submitted
I. Quarterly project management reports prepared and submitted

Comments:

Tasks 3 and 4 have been delayed due to operational difficulties. All of the proposed work will be completed within the grant period.

DATE
FILMED
10/17/94
END