

TECHNICAL REPORT  
September 1 through November 30, 1993Project Title: **BENCH-SCALE DEVELOPMENT OF MILD GASIFICATION  
CHAR DESULFURIZATION**

DOE Grant Number: DE-FC22-92PC92521 (Year 2)  
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Technology  
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Institute

## ABSTRACT

This 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.

During the first quarter, 180 lb (82 kg) of IBC-105 coal was obtained and subjected to crushing, and sizing to prepare 49 lb (22 kg) of material for test operation. A portion of the coal was also carbonized in a fixed-bed reactor to produce 13 lb (6 kg) of diluent char required for bench-scale simulation of the mild gasification reactor conditions. The analysis of the test coal is reported.

During the next quarter, necessary modifications of the bench-scale reactor will be completed, a portion of the test coal will be subjected to acid pretreatment, and char production runs will begin.

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## 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°F, <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 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,<sup>1</sup> U.S. Steel,<sup>2,3</sup> Garrett Research,<sup>4</sup> 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<sub>2</sub> and CH<sub>4</sub> are effective for this purpose, and the presence of CH<sub>4</sub> can inhibit carbon hydrogasification, thus allowing desulfurization to proceed with minimal carbon losses, compared to treatment with pure H<sub>2</sub>. The product gas from

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

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

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

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

mild gasification, using enriched-air partial combustion for heat supply, contains 10-20 vol%  $\text{CH}_4$  and 16 to 49%  $\text{H}_2$ , 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  $\text{CH}_4/\text{H}_2$  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  $\text{CH}_4/\text{H}_2$  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  $\text{CH}_4/\text{H}_2$  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.
- 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  $\text{H}_2:\text{CH}_4$  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, 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 first quarter, 180 lb (82 kg) of IBC-105 test coal were obtained from the IBCSP. The coal was screened to 40×80 mesh on a rotary screener, the overs (+40 mesh) were crushed and re-screened to obtain more 40×80-mesh coal, and the resultant test sample was riffled for analysis. Whenever possible, the coal was protected from air exposure to minimize weathering. Analysis of the test coal showed a sulfur content of 4.05 wt% dry (4.77 wt% maf).

The unders (-80 mesh) from the coal sizing operation and a portion of the test sample (40×80 mesh) were then carbonized in an atmospheric-pressure fixed-bed reactor at 1200°F (650°C) to produce a "starter" char for subsequent fluidized-bed mild gasification. The char, which was lightly to moderately agglomerated, was crushed by hand and screened to 40×80 mesh on the rotary screener. About 13 lb (5.9 kg) of char was obtained in this manner. This char, containing 3.23 wt% (dry) sulfur will be used as a bed diluent in mild gasification char production runs.

The proposed modifications to the MBFBR have been finalized, and the reactor has been delivered to a machine shop along with design drawings. The modifications include:

- Installation of 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

During the next quarter, modification of the MBFBR will be completed, a portion of the IBC-105 test sample will be subjected to acid pretreatment, and mild gasification char production runs will proceed with both untreated and acid-treated coal.

## **DISCLAIMER**

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

## 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.<sup>5,6,7,8</sup> 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.<sup>2</sup> The recycle gas used for fluidization contained H<sub>2</sub>, CO, CH<sub>4</sub>, 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<sub>2</sub>S concentration in the recycle gas was identified as a critical factor in achieving effective desulfurization. An associated study of char-sulfur chemistry<sup>3</sup> with an Illinois No. 6 hydrogasification char determined that, while H<sub>2</sub> is more effective than CH<sub>4</sub> for char desulfurization, mixtures of CH<sub>4</sub> and H<sub>2</sub> were also effective and, furthermore, the presence of CH<sub>4</sub> 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.<sup>1</sup> 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<sub>2</sub>S concentration in the treatment gas.

Research on char hydrodesulfurization was also performed by Garrett Research and Development (later Occidental Petroleum). A 1976 paper<sup>4</sup> 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<sub>2</sub>S inhibition. Although the mechanism responsible for this effect was not fully determined, the H<sub>2</sub> 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<sub>4</sub> and 28 to 50% H<sub>2</sub>, where the H<sub>2</sub>:CH<sub>4</sub> mole ratio increases monotonically from about 0.4 at 1100°F (590°C) to 1.0 at 1300°F (700°C).<sup>6</sup> 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<sub>4</sub>/H<sub>2</sub> 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,<sup>4</sup> were investigated. By a combination of acid pretreatment, mild gasification, and CH<sub>4</sub>/H<sub>2</sub> 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<sub>4</sub>/H<sub>2</sub> 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 1N 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.

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.

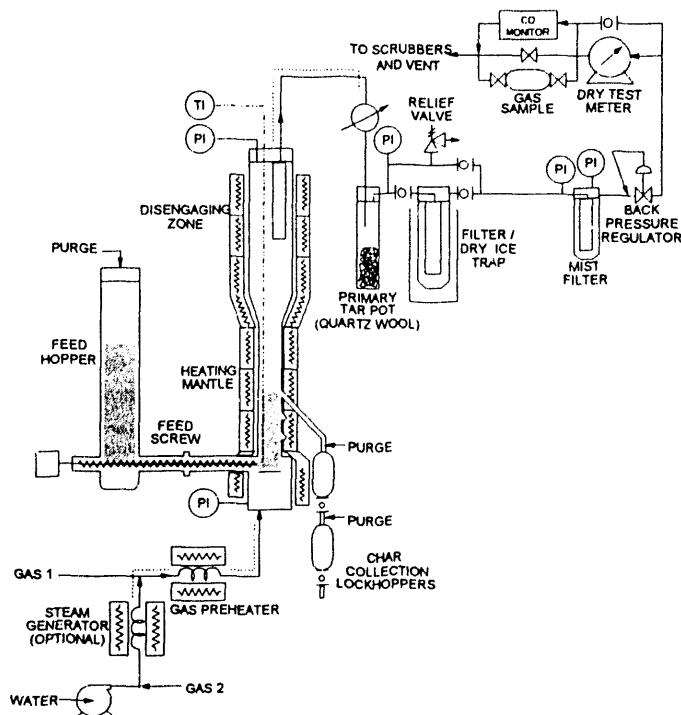


Figure 1. SCHEMATIC DIAGRAM OF MINI-BENCH FLUIDIZED-BED REACTOR (MBFBR)

The desired modifications will involve:

- Installation of 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 will be produced in the MBFBR. The initial run segment will use an IBC-105 char produced in a fixed-bed carbonizer as a diluent. The "starter" char from the fixed-bed carbonizer will be produced at 1200°F (650°C) and atmospheric pressure. The -80 mesh rejects (unders) from test coal sizing will 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 will process 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.

#### 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. Routine instrument calibrations for the char feedstocks and the higher pressure operation are to be completed under this Subtask. The unit is 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<sub>2</sub> and a CH<sub>4</sub>:CO:N<sub>2</sub>:CO<sub>2</sub> 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.

### 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  $1.0 \times 0.25$ -inch ( $2.54 \times 0.64$ -cm) disk at  $600^{\circ}$ - $650^{\circ}$ F ( $315^{\circ}$ - $345^{\circ}$ C) to form the "green" briquette. It is anticipated that briquettes will be prepared from each char produced in Task 4 which has 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  $1700^{\circ}$ - $2000^{\circ}$ F ( $930^{\circ}$ - $1090^{\circ}$ 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  $\text{CO}_2$  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<sub>2</sub> 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<sub>2</sub> 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<sub>2</sub> reactivity (Bethlehem Steel test)

*Desulfurization exit gases --*

GC/TCD/FID syngas analysis (H<sub>2</sub>, CO<sub>x</sub>, N<sub>2</sub>, O<sub>2</sub>/Ar, C<sub>1</sub>-C<sub>6</sub>)  
GC/FPD sulfur gas analysis (H<sub>2</sub>S, 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

IBC-105 coal was obtained from the IBCSP and processed to produce 49 lb (22 kg) of 40×80-mesh test sample. Six five-gallon pails, comprising about 180 lb (82 kg) of the as-received coal was required to produce the necessary quantity of the 40×80-mesh fraction.

The available analyses of the 40×80-mesh IBC-105 coal are given in Table 1. The *Forms of Sulfur* and *Mean Crystallite Size* analyses have not yet been performed.

Table 1. ANALYSIS OF UNTREATED TEST COAL

	IBC-105 (40×80)
<u>Proximate, wt% as rec'd</u>	
Moisture	8.80
Volatile Matter	34.16
Ash	13.63
Fixed Carbon	43.41
<u>Ultimate, wt% dry</u>	
Ash	14.95
Carbon	64.65
Hydrogen	4.62
Nitrogen	1.32
Sulfur	4.05
Pyritic	NA
Organic	NA
Sulfate	NA
Oxygen (by difference)	10.41
<u>Heating Value, kJ/g (dry)</u>	27.49
<u>Physical Properties</u>	
V <sub>cp</sub> , cm <sup>3</sup> /g	0.505
ρ <sub>He</sub> , g/cm <sup>3</sup>	1.48
L <sub>C</sub> , Å	NA
A <sub>S(CO<sub>2</sub>)</sub> , m <sup>2</sup> /g	294
<u>Particle Size Analysis (wt% retained on screen)</u>	
6-40 mesh	0.00
60	42.30
80	47.90
100	9.83
140-pan	0.00

Task 2. Coal Pretreatment

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

Task 3. Mild GasificationSubtask 3.1. Equipment Preparation and Shakedown

The anticipated modifications to the MBFBR were reviewed, and the detailed drawings were prepared for a machine shop to complete the work. The drawings were delivered to the shop, and the modifications are currently being done.

The fixed-bed carbonizer for preparation of "starter" char was examined and shaken down for use in Subtask 3.2.

Subtask 3.2. Char Production Runs

The oversize (+40 mesh) coal from the coal preparation activities was converted to "starter" char in the fixed-bed carbonizer. In addition, a portion of the 40×80 test coal was also carbonized to obtain an adequate amount of "starter" char for char production tests. A total of 28.5 lb (12.9 kg) of coal was processed to obtain 13.1 lb (5.97 kg) of 40×80-mesh char. The carbonized char was lightly to moderately agglomerated, required hand-crushing prior to screening out the 40×80-mesh fraction.

Task 4. Char Desulfurization

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

Task 5. Form Coking

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

Task 6. Data Analysis and Interpretation

No data analysis was required for the work performed during the current quarter.

Task 7. Process Scale-Up Design

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

## CONCLUSIONS AND RECOMMENDATIONS

No conclusions about bench-scale char desulfurization can be drawn at this time. Char production runs and char desulfurization tests will begin in the next quarter.

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  
September 1 through November 30, 1993

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 first quarter.

Projected and Estimated Actual Expenditures by Quarter

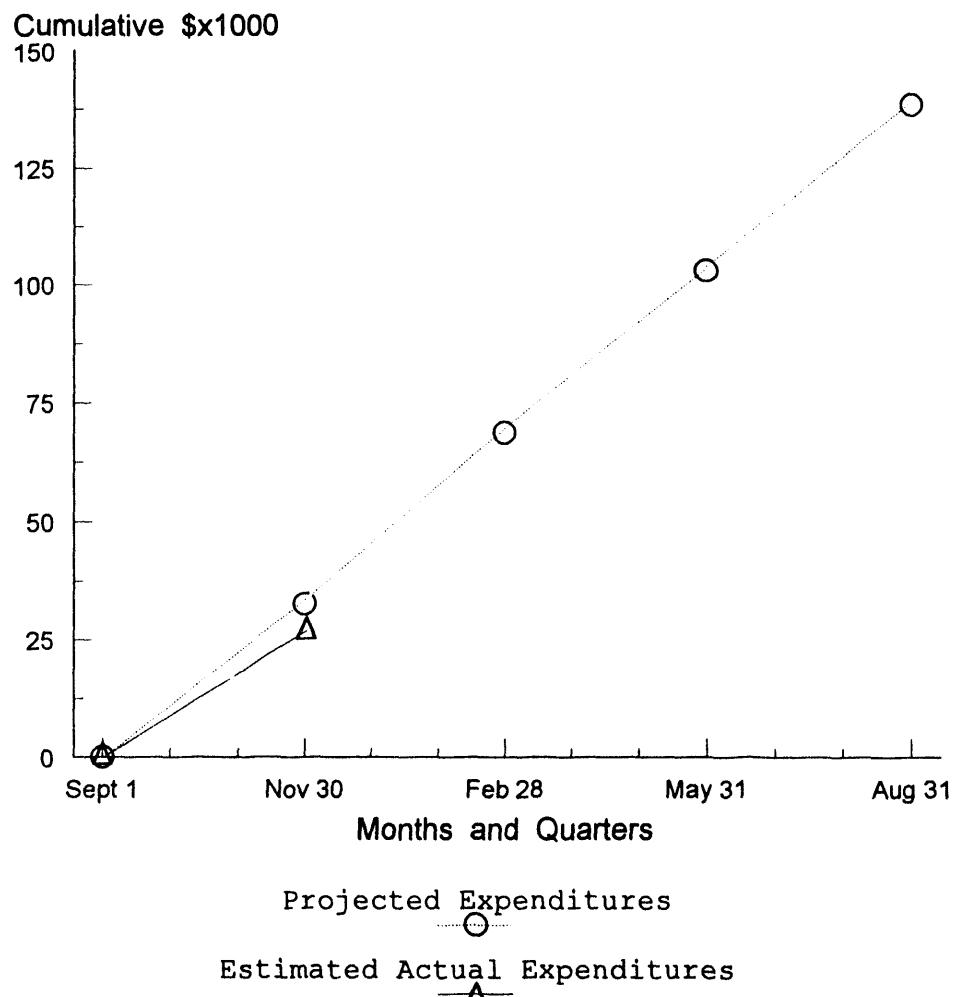
**BENCH-SCALE DEVELOPMENT OF MILD GASIFICATION CHAR DESULFURIZATION**

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	7,843		391				13,867	22,101
Sept 1, 1993 to Feb 28, 1994	Projected	24,290		2,400		1,300		43,505	71,495
	Estimated								
Sept 1, 1993 to May 31, 1994	Projected	35,447		4,150		1,300		63,500	104,397
	Estimated								
Sept 1, 1993 to Aug 31, 1994	Projected	46,680		5,505	260	1,300		83,581	137,326
	Estimated								

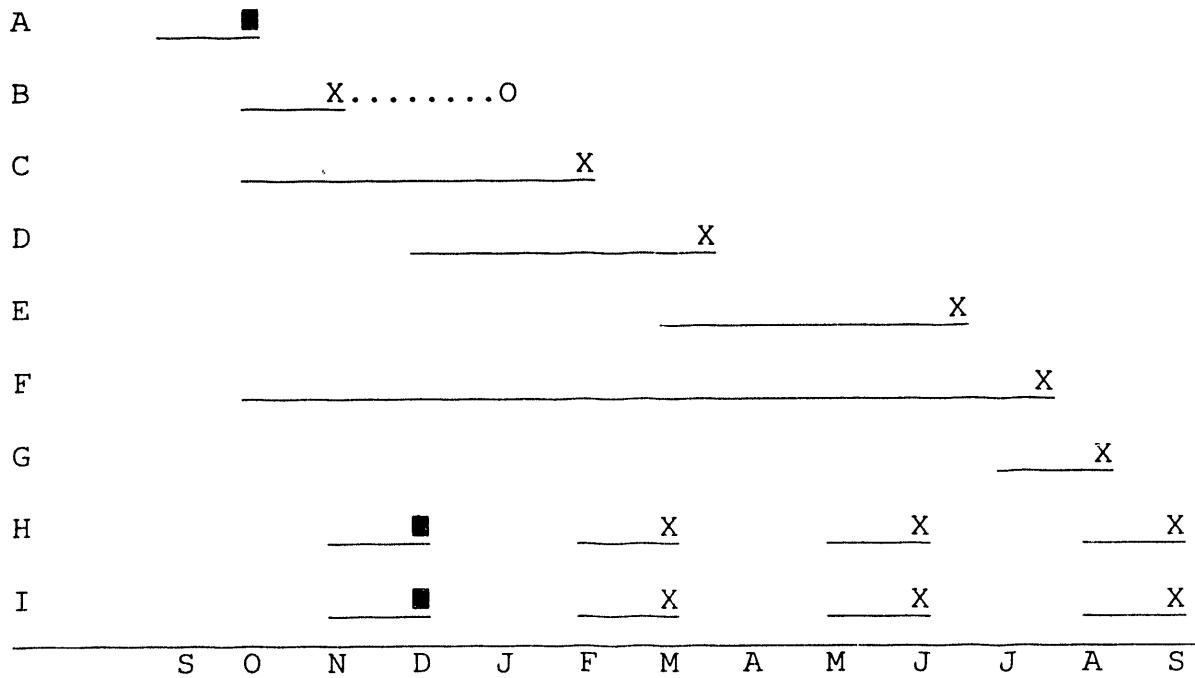
\* Cumulative by Quarter

## COSTS BY QUARTER

BENCH-SCALE DEVELOPMENT OF MILD GASIFICATION  
CHAR DESULFURIZATION



## 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:

Completion of Task 2 has been rescheduled for January 1994.



