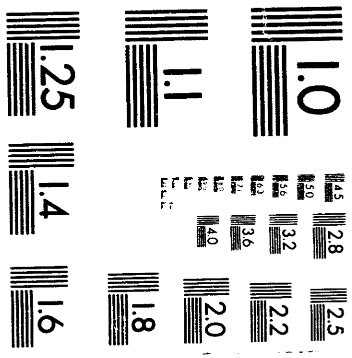
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DOE/PC/92521--T88

FINAL TECHNICAL REPORT
September 1, 1992 through August 31, 1993

Project Title: **INVESTIGATION OF A SULFUR REDUCTION TECHNIQUE FOR MILD GASIFICATION CHAR**

DE-FC22-92PC92521

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## ABSTRACT

The objective of this program is to investigate the desulfurization of mild gasification char using $H_2$:$CH_4$ mixtures. Mild gasification of coal produces char, liquids, and gases at 1000°-1500°F and near-ambient pressure. Char, comprising 60-70% of the product, can be used to make high-value form coke for steel making and foundries. However, a sulfur content below 1 wt% is desirable, and char from high-sulfur Illinois coals must be upgraded to meet this criterion.

In the first year of the two-year program, Illinois No. 6 chars were treated in a batch fluidized bed with $H_2$:$CH_4$ blends containing 9-24 vol% $CH_4$ at 1100°-1600°F and 50-200 psig. Sulfur removal up to 92.5 wt% were obtained, and the char desulfurization susceptibility was related to porosity, density, and carbon crystallite size.

During the second year, the relationships among mild gasification parameters, char properties, char desulfurization susceptibility, and form coke properties were studied. Acid washing of coal to remove Ca and Fe was explored for its effect on subsequent sulfur removal, and secondary desulfurization of form coke produced from the desulfurized chars was also studied.

In the second year, desulfurization tests of entrained and fluidized-bed reactor chars from IBC-105 coal (4.1-4.3 wt% sulfur) were completed. Desulfurization conditions were 1400°F, 100-200 psig and reactant gas compositions of 15-49 vol% $CH_4$ in $H_2$. Sulfur removal ranged from 28 to 95%, with carbon losses from 5 to 29%. Acid-washing of the coal prior to mild gasification or the char prior to desulfurization increased its susceptibility to desulfurization, with sulfur content reduced to as low as 0.10 wt% dry char. Fluidized-bed chars were easier to desulfurize than entrained chars, with or without acid-washing.

Form coke briquettes were prepared from selected chars and mild gasification pitch binder, and then carbonized under both $N_2$ and $H_2$:$CH_4$ atmospheres. Carbonization in $H_2$:$CH_4$ removed additional sulfur from the form coke. Adequate tensile strength (837 psi) was achieved in briquettes with 1.2 wt% sulfur made from desulfurized char.

## EXECUTIVE SUMMARY

The objective of this program is to investigate the desulfurization of mild gasification char using hydrogen/methane mixtures in a laboratory-scale experimental study. Mild gasification is a coal conversion technique which produces a slate of solid, liquid, and gaseous co-products at conditions of low severity (1000°-1500°F, <50 psig). A mild gasification process (MILDGAS) which uses a coaxial fluidized-bed/entrained-bed reactor system has been developed by IGT under U.S. DOE sponsorship. Char is the major co-product, comprising about 60% to 70% of the yield from the dry coal feed.

A major target market for mild gasification char is metallurgical form coke for blast furnaces and foundries, which has a market value inversely related to its sulfur content. With caking bituminous coals, binders (tar-derived pitch) for briquetting can be generated within the process.

Conventional cokes generally contain less than 1 wt% sulfur. Excess sulfur must be removed in the blast furnace by adding more limestone, which increases slag production and consumes energy. Mild gasification chars from high-sulfur Illinois coals contain about 3 wt% sulfur, and must therefore be upgraded. One method to accomplish this is desulfurization with reducing gases, which can be derived from the mild gasification co-product gases. The desulfurization can be performed on the char particles exiting the mild gasifier, and also on the formed briquettes in combination with calcining.

Research performed in the 1970's at IGT, U.S. Steel, Garrett Research, and elsewhere has shown that coal chars from various types of gasifiers can be effectively desulfurized by exposure to reducing gases at temperatures from 1100°F to 1500°F. Mixtures of hydrogen and methane are effective for this purpose, and the presence of methane inhibits carbon hydrogasification, thus allowing desulfurization to proceed with reduced carbon losses. Also, the use of a process-derived gas mixture reduces the separation costs associated with the use of pure $H_2$. The product gas from mild gasification, on an inert-free basis, contains 20-28 vol% $CH_4$ and 28-50% $H_2$, depending primarily on temperature. Estimates show that the amount of $H_2$ gas produced in mild gasification is two to three times the amount required to stoichiometrically react with all of the sulfur in the char.

In the first year of this project, particulate mild gasification chars were treated in a batch fluidized-bed reactor with mixtures of $H_2$ and $CH_4$ at various temperatures, pressures, residence times, $CH_4$ content, and fluidization gas velocity. The experiments were performed on chars produced by the IGT MILDGAS process research unit (PRU) in the

previous DOE/METC-sponsored technology development program, and by an isothermal free-fall reactor (IFFR) in a related ICCI-sponsored program. These chars contained 1.16 to 4.58 wt% sulfur. Twenty-three desulfurization tests were conducted on ten chars.

Sulfur conversions ranged from 6.0% to 92.5%, and carbon conversion ranged from 0.0% to 25.6%. The data showed that sulfur conversion correlated strongly with total connected pore volume and helium density. Correlations were also found between connected pore volume and volatile matter content: the more extensively devolatilized chars with reduced pore volume were less reactive toward desulfurization.

In the second year, the project activities included the following:

- Mild gasification chars were produced from a uniform coal feedstock (IBC-105) in a laboratory IFFR and a continuous mini-bench fluidized-bed reactor (MBFBR) which simulated, respectively, the entrained and fluidized sections of the MILDGAS reactor.
- These chars were desulfurized in the batch fluidized-bed reactor, and char desulfurization susceptibility was related to physicochemical properties of the char.
- Chars and parent coal were washed with dilute $H_2SO_4$ (prior to desulfurization) to remove Ca and Fe species, enhancing desulfurization susceptibility.
- The effects of desulfurization temperature, pressure, and $H_2$ partial pressure on sulfur removal, carbon conversion, and physical properties of char were investigated.
- Form coke briquettes were made from desulfurized mild gasification chars, and some briquettes were carbonized under reducing gas atmosphere for further sulfur removal.
- The effects of process parameters on sulfur removal, carbon gasification, and the strength and density of the resultant form coke, which are important to their use in blast furnaces, were investigated.
- The data obtained from both years of the program were used to define a desulfurization process which can be integrated with the IGT MILDGAS process.

Desulfurization tests of IFFR and MBFBR mild gasification chars from IBC-105 coal were completed. Fifteen desulfurization tests were performed on eight IFFR chars and four MBFBR chars at 1400°F, 100-200 psig, and residence times of 135-240 minutes. The desulfurizing gas was 15-49 vol% $CH_4$ in $H_2$.

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. Sulfur removal was 28-95%, resulting in char sulfur contents of 0.1-2.7 wt%, with carbon losses of 5-29%. Acid-washing of either the coal prior to mild gasification *or* of the char prior to desulfurization increased its susceptibility to desulfurization. Compared with unwashed chars, acid-washed IFFR chars released 50%-84% more of the original char sulfur, with final sulfur contents as low as 0.20 wt%. For the MBFBR chars, acid-washing of the parent coal before mild gasification resulted in 71%-74% greater sulfur release in the subsequent desulfurization, with final sulfur contents as low as 0.10 wt% dry char.

The relationships between char preparation conditions, char properties, and desulfurization susceptibility were also investigated. The data show that the desulfurization susceptibility of IFFR chars is adequately correlated to char helium density and mean crystallite size. However, these correlations did not hold true for the MBFBR chars. The data suggest that micropore collapse which occurs during entrained mild gasification does not occur in the fluidized bed, and therefore the microporous surface area plays a significantly greater role in the desulfurization of fluidized-bed chars.

It was shown that lower mild gasification temperature (1000°F versus 1150°-1300°F) generally favors the subsequent desulfurization, but also results in greater carbon conversion. Hydrogen partial pressure affected both desulfurization and carbon conversion, but, significantly, the data indicate that there may be an optimum combination of $H_2$:$CH_4$ ratio and system pressure to obtain maximum sulfur removal with minimum carbon loss. For example, with 1150°-1200°F acid-washed IFFR chars, reducing the system pressure from 200 to 100 psig at 75% $H_2$ decreased the carbon loss from 25% to 17%, while the sulfur removal remained essentially constant at 76%-78%. A lower system pressure also reduced carbon conversion for a 1000°F MBFBR char, from 12% to 5%, while sulfur removal only dropped slightly, from 83% to 78%.

For the chars prepared in this program, the fluidized-bed (MBFBR) chars were more easily desulfurized than the entrained (IFFR) chars. Acid-washing, while effective in enhancing desulfurization susceptibility, had a less dramatic effect than for the IFFR chars. The sulfur content of IBC-105 coal was reduced from 4.1 wt% to 0.54 wt% by fluidized-bed mild gasification at 1000°F and desulfurization with 25% $CH_4$:75% $H_2$ at 1400°F and 100 psig. Acid-washing of the coal prior to mild gasification resulted in a char with 0.10 wt% sulfur. Carbon losses were also less for the MBFBR chars than for the IFFR chars.

Form coke briquettes were prepared from selected IFFR chars, carbonized under both $N_2$ and $H_2$:$CH_4$ atmosphere, and tested. A total of 15 briquettes were prepared from four selected IFFR chars, using as binder either the parent coal or a mild gasification pitch derived from Illinois No. 6 coal. The pitch binder contained 2.4 wt% sulfur. The form coke briquettes carbonized in the reducing atmosphere lost 22-48% more sulfur than those carbonized in nitrogen As with the particulate chars, the form coke samples made from acid-washed chars were more susceptible to sulfur release during carbonization under $H_2$:$CH_4$ atmosphere than were briquettes made from unwashed chars. For example, an acid-washed IFFR char with 0.70 wt% sulfur produced form coke with 0.48 wt% sulfur.

Form coke tensile strengths ranged from 80-837 lb/in$^2$, with two briquettes reporting in a range similar to commercial cokes tested (300-800 lb/in$^2$).

## 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.

## 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 PRU was built and operated for 47 tests at temperatures ranging from 1034° to 1390°F. A subsequent team led by Kerr-McGee Coal Corporation has designed a 24-ton/day MILDGAS PDU which will be built at SIUC in 1993-94.[1,2,3,4] 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. In a blast furnace, coke acts as both a fuel and as a reductant for iron ore. Excessive coke sulfur requires large limestone input, which adversely affect the economics of the process. 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. With Illinois No. 6 coal containing 3-5 wt% sulfur, the char sulfur content from the PRU was 2.1-2.8 wt%.[2]

In the 1970's, U.S. Steel developed the Clean Coke process, in which desulfurization of the char with recycled product

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

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

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

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

gas played an important role.[5] The recycle gas used for fluidization contained $H_2$, CO, $CH_4$, and higher hydrocarbons. At 1400°F, 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[6] 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 at longer residence times while allowing desulfurization to continue. The same study also found that, for a given char and gas composition, the relationship between sulfur removal and carbon loss was fairly constant regardless of the temperature or pressure. Based on this relationship, a 50% to 65% sulfur reduction, such as may be required for mild gasification char to meet the selected form coke requirement of 1 wt% sulfur, could be achieved with an accompanying 10% to 13% carbon conversion.

In similar work, IGT studied the hydrodesulfurization of four bituminous coals at 1300° to 1500°F for 30 minutes in hydrogen.[7] 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[8] discussed the effectiveness of subjecting char to acid-washing to remove Ca and Fe 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 consisting primarily of $H_2$ and $CH_4$. The application of this technique to mild gasification char and/or briquettes, and the

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

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

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

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

integration of the technique with form coking, have not previously been studied. In the IGT MILDGAS process, bituminous coal is not pre-oxidized to prevent caking as has been done in other processes.[5,7] The intrinsic caking tendency of the coal is instead overcome by reactor geometry and feed conditions which achieve rapid mixing. MILDGAS char thus differs in physical and chemical properties from coal and from gasification or hydrogasification chars that have been examined in other programs.

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. The relative feasibility and impact on overall process efficiency of desulfurization before and/or after briquetting must be evaluated.

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 to 1.0 at 1300°F.[2] In addition to the presence of $CH_4$ inhibiting carbon gasification during desulfurization, the use of a process-derived gas mixture, even if it required $H_2$ enrichment, would be more economical than using pure $H_2$. If necessary, the $H_2$ content of the desulfurizing gas can be adjusted via the shift reaction or partial steam reforming of $CH_4$ and other hydrocarbon gases.

## EXPERIMENTAL PROCEDURES

In the first year, Illinois No. 6 mild gasification chars from PRU tests conducted under a DOE/METC contract were tested, in addition to chars made from IBCSP coals in previous ICCI projects. Although this work yielded valuable insights into the factors affecting char desulfurization, PRU chars tested in the first year contained up to 64% residual coke breeze, used in the PRU as a bed diluent to simulate char recycle. This introduced difficulties in correlating char desulfurization with certain physical properties, such as crystallite size and surface area.

In the present (second-year) program, particulate chars were prepared in laboratory reactors from IBC-105 coal, without adulteration from coke breeze. This allowed a more direct correlation of properties with desulfurization data. Acid washing of coal and char prior to desulfurization was also studied. Form coke briquettes were prepared with desulfurized chars, and briquette carbonization in a reducing atmosphere for additional sulfur removal was examined.

Task 1. Sample Preparation and Characterization

IBC-105 coal was dried under nitrogen, crushed, and screened to two size fractions: -40+80 mesh for mild gasification in a mini-bench fluidized-bed reactor (MBFBR), and -80+100 mesh for mild gasification in an isothermal free-fall reactor (IFFR).

Mild gasification chars simulating the rapid-heating dilute-phase conditions of the entrained section of the MILDGAS reactor were prepared in the continuous IFFR at temperatures of 1000°-1300°F, near-ambient pressure, and particle residence times of 1.5-2.5 seconds. Fluidized-bed char simulating the product from the fluidized section of the MILDGAS reactor was prepared in the continuous MBFBR at temperatures of 1000° to 1200°F, 10-30 psig, and particle residence times of about 40 minutes. In MBFBR char production runs, coal was fed as a 1:4 (w/w) blend with 40×70-mesh alumina beads to control agglomeration. The recovered chars were vacuum-dried and pneumatically separated from alumina. The density differential between the char (ca. 1.4 g/mL) and alumina spheres (ca. 3.6 g/mL) provided the necessary difference in fluidization properties. Two passes through a fluidization column were used, and the recovered low-density fraction from each run was analyzed to determine residual $Al_2O_3$. Following separation, the chars were re-screened to 40×70 mesh.

Acid-washed coals or chars were produced by stirring the coal or char with 1*N* $H_2SO_4$ at 176°F for 5 minutes, followed by water washing until a $BaCl_2$ test of the filtrate determined that residual acid had been removed from the char. The coal or char was then vacuum-dried at 104°F to constant weight.

The coal and particulate char samples were analyzed in IGT laboratories for proximate and ultimate analyses, connected pore volume, helium density, $N_2$ surface area, $CO_2$ surface area, and mean crystallite size by x-ray diffraction.

Form coke briquettes were prepared by both conventional and binderless techniques developed in the recently completed DOE-sponsored mild gasification development program. Char was mixed either with a fluxing oil and a small amount of coal to generate an *in-situ* binder from the plastic state of the coal (binderless briquetting) or with a pitch derived from mild gasification of Illinois No. 6 coal in the PRU. The mixture was then compressed at 850°F (binderless method) or at 600°-650°F (pitch method) to form the "green" briquette, then carbonized at 1800°F to devolatilize the briquette and develop maximum mechanical strength.

Task 2. Equipment Preparation and Shakedown

The IFFR, shown in Figure 1, did not require any extensive modifications for char production. The continuous MBFBR, however, was modified for production of char under mild gasification conditions with caking Illinois coals. The coaxial char overflow tube was eliminated in favor of a side discharge, as shown in Figure 2, to increase the effective bed cross-section and reduce wall effects. Three selectable ports were incorporated at 4-, 6-, and 8-inch bed height locations. Also, an internal thermocouple port was included in the feed screw housing to provide more reliable monitoring of the screw temperature.

A batch fluidized-bed reactor, shown in Figure 3, was used for char desulfurization tests. The reactor vessel was fabricated in the first year from Sch40 316 stainless steel to safely operate up to 1800°F at atmospheric pressure and up to 1400°F at 200 psig. The desulfurization reactor did not require any significant modifications in the current year.

The equipment for producing form coke briquettes, existing from previous work, did not require any significant modifications.

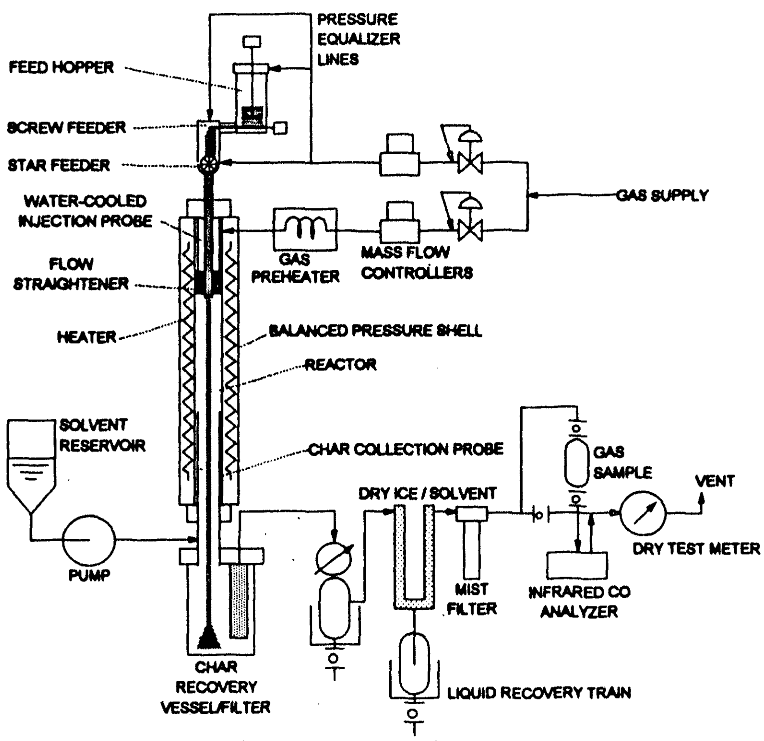



Figure 1. SCHEMATIC DIAGRAM OF ISOTHERMAL FREE-FALL REACTOR

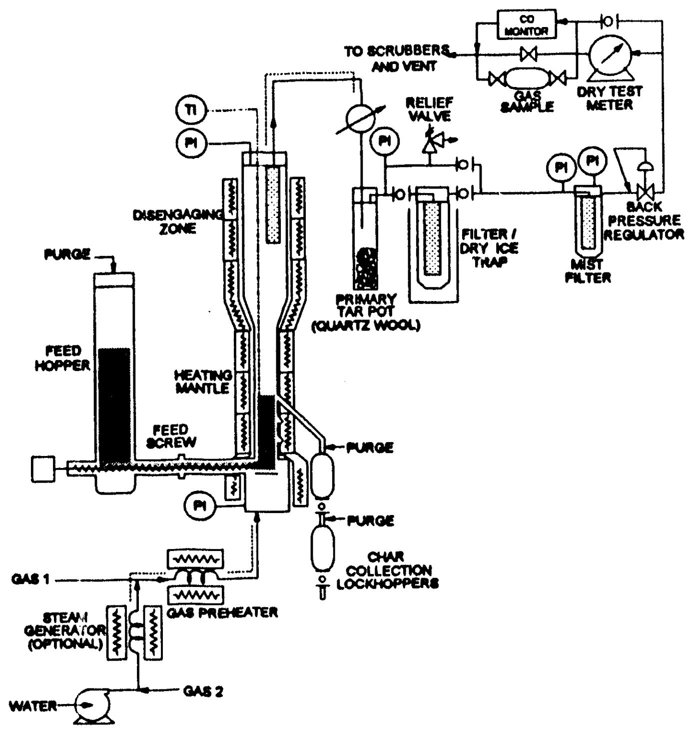



Figure 2. SCHEMATIC DIAGRAM OF MINI-BENCH FLUIDIZED-BED REACTOR

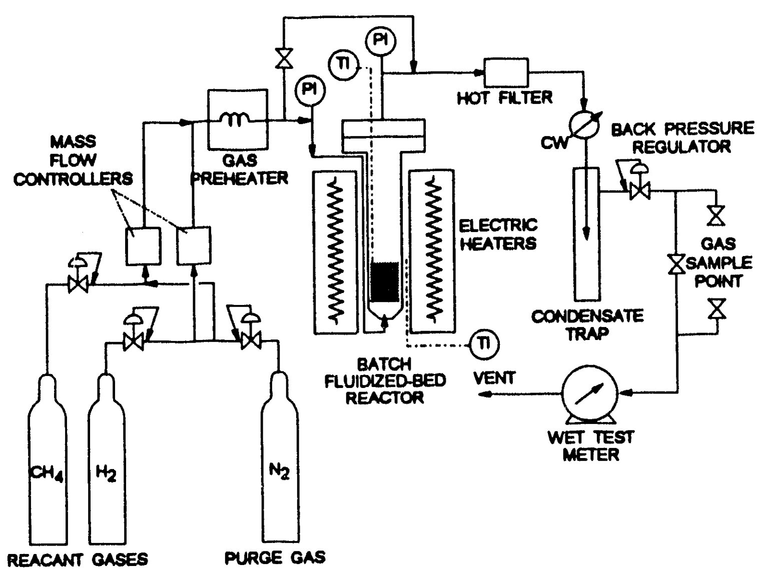



Figure 3. EXPERIMENTAL CHAR DESULFURIZATION REACTOR

## Task 3. Desulfurization

### Subtask 3.1. Parametric Experiments

Particulate Char: Particulate char desulfurization experiments were performed to determine the relationship among: char preparation conditions (reactor type, mild gasification temperature, acid-washing); char physicochemical properties (volatile matter, helium density, connected pore volume, surface area, mean crystallite size); desulfurization conditions (temperature, pressure); and desulfurization performance (sulfur conversion, carbon conversion). Table 1 shows the specific nominal ranges of these parameters that were tested.

Table 1. EXPERIMENTAL PARAMETERS FOR PARTICULATE CHARS

| | |
|---|---|
| Mild gasification reactor type | Entrained (IFFR), Fluidized (MBFBR) |
| Mild gasification temperature | 1000 - 1300 °F |
| Acid-washing | None, coal, char |
| Desulfurization temperature | 1400 °F |
| Desulfurization pressure | 100 - 200 psig |
| Desulfurization residence time | 120 - 240 min |
| Gas composition | 25 - 50 vol% $CH_4$ in $H_2$ |
| Superficial gas velocity | 300% of $u_{mf}$ |

In a typical desulfurization experiment with particulate char, 50 to 100 grams of char were charged to the reactor, which was then sealed and purged with inert gas. The reactor was heated to the desired desulfurization temperature while continuing the flow of inert gas. When the desired temperature was attained, the desired premixed reaction gas was introduced. Gas samples were retrieved at 10- to 30-minute intervals until the end of the desired residence time. Inert gas flow then replaced the reactive gas mixture, and the reactor was rapidly cooled to retrieve the desulfurized sample (spent char).

Form Coke: Briquette desulfurization tests were performed on cured (uncarbonized) briquettes in combination with carbonization (calcination). Briquettes were produced from five IFFR chars, one prior to desulfurization and four after desulfurization. The parent (IBC-105) coal was used as the binder in seven tests, and a mild gasification-derived pitch (IGT MILDGAS PRU Test IST-9)[4] was used as the binder in the remaining 12 tests, six of which were performed in $H_2$:$CH_4$.

The conditions of form coke carbonization-desulfurization are given in Table 2.

Table 2. EXPERIMENTAL PARAMETERS FOR FORM COKE BRIQUETTES

| | |
|---|---|
| Particulate char type | Entrained (IFFR), |
| Acid-washing | None, char |
| Briquette desulfurization temperature | 1800 °F |
| Briquette desulfurization pressure | Ambient |
| Briquette desulfurization res time | 120 min |
| Gas composition | 100% $N_2$, 25 vol% $CH_4$:$H_2$ |

<u>Subtask 3.2. Product Characterization and Evaluation</u>

The desulfurized sample from each experiment was evaluated by proximate and ultimate analyses. The amount of sulfur removal was calculated based on the weight percent of sulfur in the parent sample. Other particulate char properties examined in the study were: connected pore volume, helium density, surface area, and mean (graphitic) crystallite size.

*Connected pore volume*, $V_{cp}$, was computed from a plot of cumulative pore volume per gram vs pore diameter, determined on a Micromeritics 9310 mercury porosimeter, corrected for interparticle void volume.

*Helium density*, $\rho_{He}$, was measured in a helium pycnometer.

*$N_2$ Surface area*, $A_s(N_2)$, was determined by the BET method.

*$CO_2$ Surface area*, $A_s(CO_2)$, was determined by the DP method.

*Mean crystallite size*, $L_c$, was determined from x-ray diffraction spectra using the line broadening at half height of the 002 reflection using the Scherrer equation. $L_c$ is the mean dimension of the crystallite in the direction which is perpendicular to the layer plane.

For form coke briquettes, proximate and ultimate analyses were performed to determine the extent of carbonization and sulfur removal. Also, the effects of sulfur reduction during carbonization on the density and strength of the form coke were assessed.

*Briquette density*, $\rho$, was calculated from weight and volume measurements

*Briquette diametral tensile strength, S,* was measured on an Instron Model 101 compression-tension device following the ASTM B485-76 procedure.

Task 4. Data Analysis and Interpretation

The experimental data were analyzed and interpreted to determine the effects of the selected parameters and physicochemical properties on the following properties of the mild gasification char and form coke briquettes:

- Percent sulfur reduction, $100\left[1-\frac{(\%S_p/100)}{(\%S_f/100)}\right]$

  where $\%S_f$ = feed char S content and $\%S_p$ = product S content.

- Percent sulfur conversion, $100\left[1-\frac{m_p(\%S_p/100)}{m_f(\%S_f/100)}\right]$

  where $m_f$ = feed char mass, $m_p$ = product mass, $\%S_f$ = feed char S content, and $\%S_p$ = product S content.

- Percent carbon conversion, $100\left[1-\frac{m_p(\%C_p/100)}{m_f(\%C_f/100)}\right]$

  where $m_f$ = feed char mass, $m_p$ = product mass, $\%C_f$ = feed char C content, and $\%C_p$ = product C content.

For form coke, the results of density and strength testing were compared with data on commercial coke.

## RESULTS AND DISCUSSION

Task 1. Sample Preparation and Characterization

Coal Preparation: A fresh sample of IBC-105 coal was obtained from the IBCSP. The 20-lb sample was dried under nitrogen, crushed, and screened to two size fractions: -40+80 for MBFBR char preparation, and -80+170 for IFFR char preparation. A portion of the coal feed sized for the MBFBR was acid-washed with 1*N* $H_2SO_4$ as previously described.

The analyses of these coal samples are given in Table 3. In addition to the data in Table 3, both parent coal samples were found to have a free-swelling index of 1.

Table 3. COAL ANALYSES

| | MBFBR feed (-40+80) | Acid-washed MBFBR feed (-40+80) | IFFR feed (-80+170) |
|---|---|---|---|
| Proximate, wt% as rec'd | | | |
| Moisture | 8.90 | 1.40 | 9.00 |
| Volatile Matter | 33.50 | 38.60 | 33.70 |
| Ash | 13.80 | 11.30 | 13.00 |
| Fixed Carbon | 43.60 | 48.50 | 44.20 |
| Ultimate, wt% dry | | | |
| Ash | 15.17 | 11.50 | 14.29 |
| Carbon | 64.75 | 67.33 | 64.23 |
| Hydrogen | 4.47 | 4.76 | 4.40 |
| Nitrogen | 1.26 | 1.36 | 1.19 |
| Sulfur | 4.06 | 3.50 | 4.34 |
| Oxygen (by difference) | 10.29 | 11.55 | 11.55 |
| Physical Properties | | | |
| $V_{cp}$, $cm^3/g$ | 0.103 | 0.0807 | 0.0994 |
| $\rho_{He}$, $g/cm^3$ | 1.48 | 1.43 | 1.47 |
| $A_s(N_2)$, $m^2/g$ | 15.2 | 25.5 | 15.7 |
| $A_s(CO_2)$, $m^2/g$ | 126 | 159 | 118 |
| $L_c$, Å | 9 | 7 | 10 |

Acid-washing of the -40+80 coal removed 29.8 wt% of the ash and 20.1 wt% of the sulfur. The carbon loss during acid-washing was estimated at only 3.7 wt%. The sulfur content of the coal was reduced from 4.06 wt% to 3.50 wt% of dry coal by acid-washing. The physical properties of the coal changed significantly, with both $N_2$ and $CO_2$ surface areas increasing and the mean crystallite size decreasing with the acid washing.

According to Johnson,[9] the $CO_2$ desorption surface area, $A_s(CO_2)$, is a more realistic estimate of the true internal surface of a coal or coal char than the $N_2$ surface area, $A_s(N_2)$, which does not generally account for the micropores below 12Å. As expected, the $A_s(CO_2)$ values for untreated coal and acid-washed coal exceeded the $A_s(N_2)$ values by an order of magnitude, as shown in Table 3.

Mild Gasification (IFFR): In the IFFR, four mild gasification chars were produced from the 80×170-mesh coal at average hot zone temperatures of 1000°, 1150°, 1200°, and 1300°F. The conditions of the IFFR mild gasification runs are given in Table 4. With particle residence times of 2.0

9 Johnson, J.L., *Kinetics of Coal Gasification*, John Wiley & Sons, New York, N.Y., 51-59, (1979).

Table 4. IFFR MILD GASIFICATION RUN CONDITIONS

| Run No. | ICP1002 | ICP1008 | ICP1014 | ICP1021 |
|---|---|---|---|---|
| Temperature, °F | 1000 | 1150 | 1300 | 1200 |
| Pressure, psig | 3.1 | 2.6 | 2.9 | 2.3 |
| Solid res time, s | 2.0 | 2.5 | 2.5 | 2.5 |
| Gas res time, s | 38.1 | 35.9 | 34.0 | 35.2 |
| Gas atmosphere | $N_2$ | $N_2$ | $N_2$ | $N_2$ |
| Coal feed rate, g/min | 1.3 | 1.3 | 1.4 | 1.4 |
| Gas feed rate, sL/min | 1.27 | 1.22 | 1.18 | 1.21 |
| Char yield, wt% as rec'd | 77.6 | 67.3 | 62.6 | 65.9 |

to 2.5 seconds, the as-received char yields were in the range of 62 to 78 wt%, decreasing with higher reactor temperature. The recovered chars were vacuum-dried, agglomerates were broken up where necessary, and the entire samples were re-screened to their original size fraction of -80+170 mesh prior to desulfurization tests.

Mild Gasification (MBFBR): In the MBFBR, four mild gasification chars were produced: two from the unwashed 40×80-mesh coal at 1000° and 1200°F, and two from the acid-washed 40×80-mesh coal at the same two temperatures. The unwashed coal, fed as a 1:4 (w/w) blend with 40×70-mesh alumina beads, agglomerated lightly during the tests. The agglomerates were quite soft and easily crushed by hand, except for approximately 10 grams of hard sinter that formed around the feed inlet. In the first (1000°F) run with acid-washed coal, however, agglomeration was more severe, which resulted in binding of the feed screw by the end of the run. The gas velocity was subsequently increased, and cooling of the screw tip was improved to reduce caking at the reactor inlet. These steps improved performance to the extent that production of 1200°F MBFBR char could be completed.

The conditions of the MBFBR mild gasification runs are given in Table 5. The recovered chars were vacuum-dried, agglomerates were broken up where necessary, and the entire sample from each run was subjected to pneumatic separation and re-screening as previously described.

Form Coke Production: Form coke briquettes were produced from mild gasification chars before and after desulfurization. Both binderless briquetting, using the parent coal, and conventional briquetting, using mild gasification "crude pitch" as binding agent, were studied.

Table 5. MBFBR MILD GASIFICATION RUN CONDITIONS

| Run No. | FCP0106 | FCP0112 | FCP0120 | FCP0202 |
|---|---|---|---|---|
| Temperature, °F | 1000 | 1185 | 1000 | 1180 |
| Pressure, psig | 20 | 14 | 23 | 5 |
| Gas atmosphere | $N_2$ | $N_2$ | $N_2$ | $N_2$ |
| Sup gas velocity, ft/s | 0.24 | 0.30 | 0.23 | 0.49 |
| Coal feed rate, g/min | 1.1 | 1.3 | 1.1 | 1.0 |
| Gas feed rate, sL/min | 6.9 | 6.3 | 7.1 | 7.2 |
| Gas res time in bed, s | 1.7 | 1.4 | 1.8 | 0.9 |
| Solid res time, min | 103 | 88 | 103 | 114 |

Fifteen briquetting tests were performed, and the briquettes were subjected to carbonization under both neutral ($N_2$) and reducing ($H_2$:$CH_4$) atmospheres. The results of the carbonization and combined carbonization-desulfurization steps will be discussed under Task 4.

Mild Gasification Char Properties (IFFR): Table 6 shows the chemical and physical property data for the four unwashed IFFR chars. Compared to the parent coal, the chars showed typical temperature-dependent decreases in volatile matter and increases in ash content. The IFFR char sulfur content was lower than that of the parent coal by 16% to 22%, with higher IFFR temperature promoting more sulfur reduction. This yielded mild gasification chars with sulfur contents of 3.37 to 3.63 wt% sulfur on a dry basis. Figure 6 shows the effects of IFFR temperature on char volatile matter, ash, and sulfur content.

As for physical properties of IFFR chars, the connected pore volume increased very significantly compared to the parent coal, with higher IFFR temperature resulting in a further increase in connected pore volume. Helium density also followed the same trend. However, BET surface area decreased from 15.2 $m^2/g$ in the parent coal to much lower values of 0.4 to 0.6 $m^2/g$ in the IFFR chars, and the $CO_2$ surface areas also decreased dramatically, from 118 to <2 $m^2/g$. This is consistent with a model of micropore closing or collapse upon thermolysis, together with the enlargement of the macropores which are the major contributor to overall pore volume, but only a minor contributor to surface area. The mean crystallite size by XRD increased moderately from 9Å in the coal to 11-13Å in the chars. The effect of IFFR temperature over the 1000°-1300°F range on crystallinity was not significant in these tests.

Figure 7 shows the effects of IFFR mild gasification temperature on key physical properties of the char.

Table 6. ANALYSES OF UNWASHED IFFR MILD GASIFICATION CHARS

| Run No. | ICP1002 | ICP1008 | ICP1014 | ICP1021 |
|---|---|---|---|---|
| Proximate, wt% as rec'd | | | | |
| Moisture | 2.40 | 1.60 | 1.90 | 1.50 |
| Volatile Matter | 27.70 | 20.60 | 16.50 | 19.50 |
| Ash | 17.80 | 20.30 | 21.10 | 20.30 |
| Fixed Carbon | 52.00 | 57.30 | 60.20 | 58.50 |
| Ultimate, wt% dry | | | | |
| Ash | 18.25 | 20.68 | 21.59 | 20.70 |
| Carbon | 64.94 | 63.41 | 65.28 | 64.32 |
| Hydrogen | 3.76 | 2.91 | 2.32 | 2.79 |
| Nitrogen | 1.24 | 1.28 | 1.29 | 1.29 |
| Sulfur | 3.63 | 3.49 | 3.37 | 3.51 |
| Oxygen | 8.18 | 8.23 | 6.15 | 7.39 |
| Physical Property Data | | | | |
| $V_{cp}$, $cm^3/g$ | 0.559 | 0.594 | 0.693 | 0.581 |
| $\rho_{He}$, $g/cm^3$ | 1.53 | 1.60 | 1.62 | 1.59 |
| $A_s(N_2)$, $m^2/g$ | 0.4 | 0.4 | 0.6 | <0.5 |
| $A_s(CO_2)$, $m^2/g$ | <2.0 | <2.0 | -- | -- |
| $L_c$, Å | 11 | 13 | 11 | 13 |

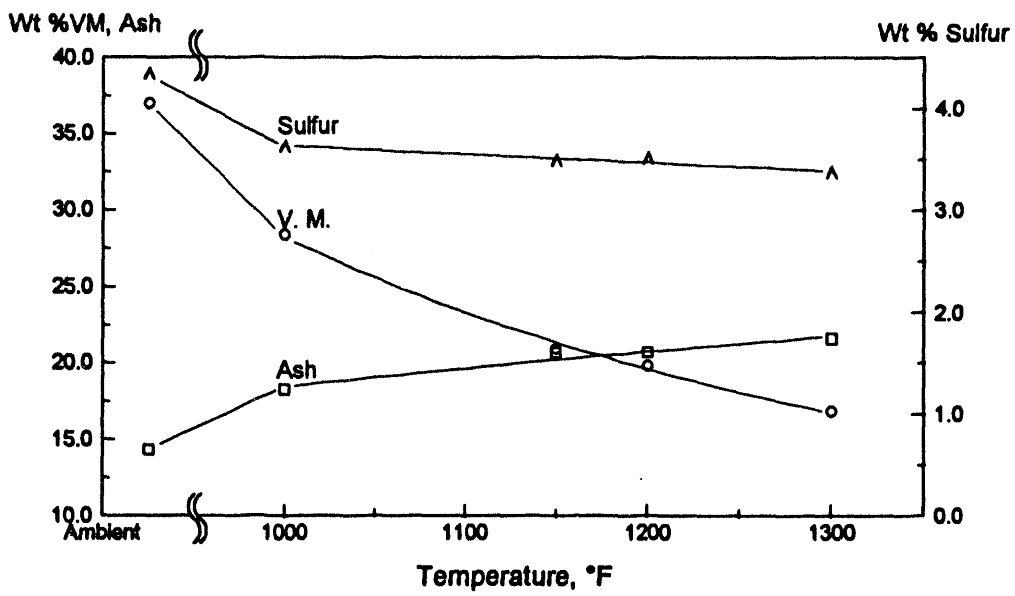



Figure 6. EFFECTS OF IFFR MILD GASIFICATION TEMPERATURE ON CHAR VOLATILE MATTER, ASH, AND SULFUR CONTENT

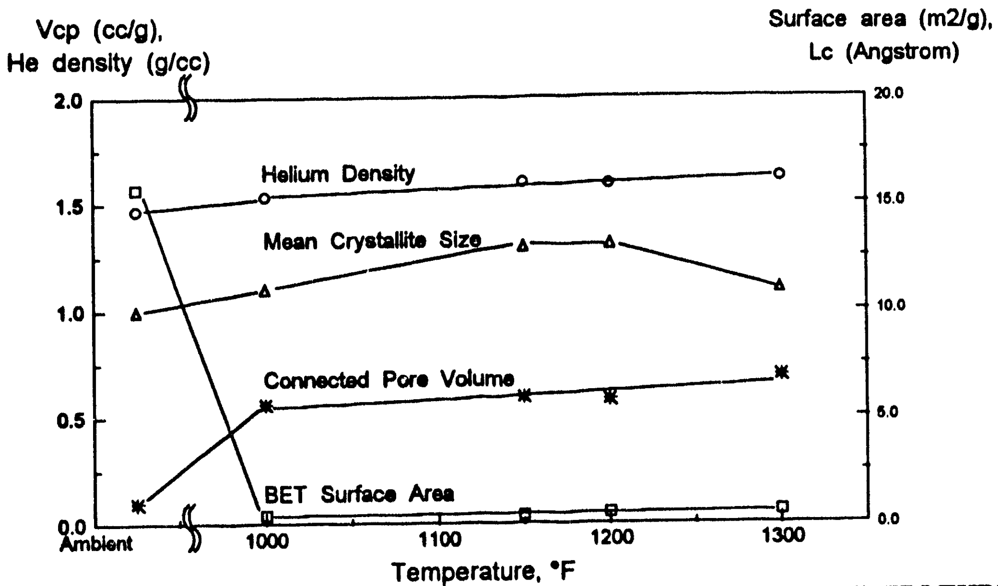



Figure 7. EFFECTS OF IFFR MILD GASIFICATION TEMPERATURE ON PORE, SURFACE, AND CRYSTALLINITY PROPERTIES OF THE CHAR

A portion of each of the IFFR mild gasification chars was subjected to acid-washing. This was done to compare the effect on the desulfurization susceptibility of acid-washing mild gasification *char* versus acid-washing of the parent *coal*. The analyses of the acid-washed IFFR chars are given in Table 7. Based on normalization to constant carbon (carbon is not removed by acid-washing), the treatment removed 9% to 15% of the ash and 10% to 22% of the sulfur from the chars. More ash and sulfur were removed from the lower-temperature chars than the higher-temperature chars. The effects of acid-washing on hydrogen, nitrogen, and oxygen contents did not appear to be significant (less than 5% relative change). Acid-washing increased the $A_s(N_2)$ and $A_s(CO_2)$ surface areas moderately, indicating a minor degree of pore opening by dissolution of mineral matter.

Figure 8 shows the relationship between mild gasification temperature and the effects of acid-washing on chemical composition of IFFR chars.

<u>Char Properties (MBFBR)</u>: Pneumatic separations of the MBFBR chars from alumina beads were performed, and the residual alumina in the chars ranged from 0.0 to 8.9 wt%. The physical and chemical properties of the four MBFBR chars, corrected for residual alumina, are shown in Table 8.

The MBFBR char property data in Table 10 show that the MBFBR chars are, as expected, denser and less porous (1.6-1.8 g/mL, 0.21-0.27 $cm^3/g$) than the IFFR chars (1.5-1.6 g/mL, 0.55-0.69 $cm^3/g$). However, the MBFBR chars possessed much

Table 7. ANALYSES OF *ACID-WASHED* IFFR MILD GASIFICATION CHARS

| Run No. | ICP1002 | ICP1008 | ICP1014 | ICP1021 |
|---|---|---|---|---|
| Proximate, wt% as rec'd | | | | |
| Moisture | 1.80 | 1.20 | 1.40 | 0.90 |
| Volatile Matter | 27.70 | 20.20 | 15.70 | 18.80 |
| Ash | 15.70 | 18.10 | 19.80 | 18.80 |
| Fixed Carbon | 54.50 | 60.30 | 62.90 | 61.30 |
| Ultimate, wt% dry | | | | |
| Ash | 16.06 | 18.39 | 20.14 | 19.00 |
| Carbon | 67.26 | 65.68 | 66.98 | 65.57 |
| Hydrogen | 3.69 | 3.05 | 2.48 | 2.96 |
| Nitrogen | 1.31 | 1.29 | 1.26 | 1.32 |
| Sulfur | 2.95 | 3.04 | 3.08 | 3.22 |
| Oxygen | 8.73 | 8.55 | 6.06 | 7.93 |
| Physical Property Data | | | | |
| $V_{cp}$, $cm^3/g$ | 0.562 | 0.584 | 0.679 | 0.554 |
| $\rho_{He}$, $g/cm^3$ | 1.48 | 1.57 | 1.62 | 1.60 |
| $A_s(N_2)$, $m^2/g$ | 0.8 | 0.7 | 1.0 | 1.4 |
| $L_c$, Å | 10 | 13 | 12 | 13 |

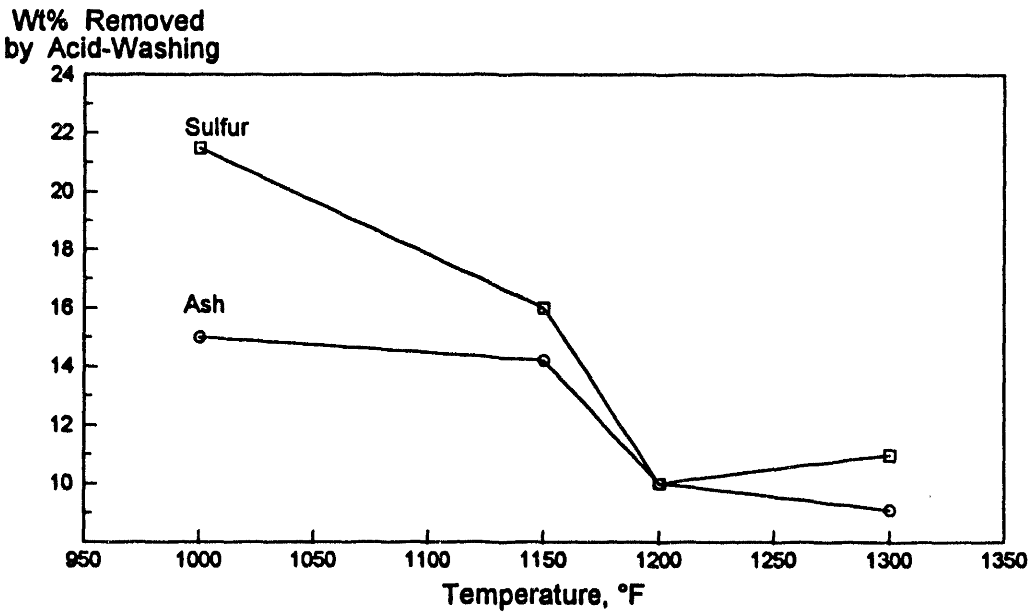



Figure 8. EFFECTS OF ACID-WASHING AND MILD GASIFICATION TEMPERATURE ON IFFR CHAR COMPOSITION

Table 8. PROPERTIES OF MBFBR MILD GASIFICATION CHARS

| MBFBR Run | FCP0106 | FCP0112 | FCP0120 | FCP0202 |
|---|---|---|---|---|
| Acid-Washed Coal | No | No | Yes | Yes |
| Run Temperature, °F | 1000 | 1200 | 1000 | 1200 |
| Proximate, wt% as rec'd | | | | |
| Moisture | 0.66 | 0.64 | 0.90 | 0.77 |
| Volatile Matter | 14.90 | 9.22 | 13.89 | 9.33 |
| Ash | 20.97 | 20.39 | 13.16 | 22.25 |
| Fixed Carbon | 63.47 | 69.75 | 72.05 | 67.65 |
| Ultimate, wt% dry | | | | |
| Ash | 21.11 | 20.52 | 13.28 | 22.42 |
| Carbon | 66.81 | 71.81 | 73.46 | 68.70 |
| Hydrogen | 2.37 | 1.46 | 2.58 | 1.55 |
| Nitrogen | 1.63 | 1.64 | 1.80 | 1.61 |
| Sulfur | 2.43 | 2.75 | 2.11 | 2.06 |
| Oxygen (by difference) | 5.65 | 1.81 | 6.77 | 3.65 |
| Physical Property Data | | | | |
| $V_{cp}$, $cm^3/g$ | 0.206 | 0.205 | 0.266 | 0.229 |
| $\rho_{He}$, $g/cm^3$ | 1.59 | 1.79 | 1.62 | 1.81 |
| $A_s(N_2)$, $m^2/g$ | 0.4 | 5.0 | <0.5 | 2.0 |
| $A_s(CO_2)$, $m^2/g$ | 189 | 299 | 267 | 261 |
| $L_c$, Å | 13 | 11 | 10 | 11 |

higher $CO_2$ surface areas (189-299 $m^2/g$) than the IFFR chars prior to desulfurization (<2 $m^2/g$), indicating that micropore collapse did not occur. These facts point out profound differences in char microstructure related to the mode of pyrolysis, which impact the desulfurization susceptibility.

## Task 2. Equipment Preparation and Shakedown

The MBFBR reactor was modified as described in EXPERIMENTAL PROCEDURES during the program. A new heating mantle with openings for char discharge ports was installed on the MBFBR. Modifications of the other units (IFFR, desulfurization reactor, and form coke briquetting apparatus) were minor.

## Task 3. Desulfurization

### Subtask 3.1. Parametric Experiments

Desulfurization tests were performed on particulate (IFFR and MBFBR) chars and on form coke briquettes in conjunction with briquette carbonization.

<u>IFFR Chars</u>: 10 desulfurization tests were performed on four IFFR chars. These tests were conducted at 1400°F, 100-200 psig, and residence times of 135-240 minutes. The desulfurizing gas compositions included $CH_4$ levels of 15-49 vol% in $H_2$. Tests D1113, D1117, D1123, D1208, D0317, and D0322 were performed on unwashed IFFR chars; tests D1215, D1217, D0222, and D0315 were performed on acid-washed IFFR chars. Table 9 shows the operating conditions and results of the desulfurization tests on IFFR chars.

<u>MBFBR Chars</u>: Five desulfurization tests were performed on four MBFBR chars. The tests were conducted at 1400°F, 100-200 psig, and residence times of 135-240 minutes. The desulfurizing gas composition was the same in all tests with MBFBR chars (21 vol% $CH_4$ in $H_2$). Tests D0224, D0301, and D0310 were performed on MBFBR chars from unwashed coal; and tests D0303 and D0308 were performed on MBFBR chars from acid-washed coal. Table 10 shows the operating conditions and results of the desulfurization tests on MBFBR chars.

<u>Form Coke</u>: 15 form coke briquettes were carbonized in neutral and reducing atmospheres to develop full strength and remove additional sulfur. Three of the briquettes were produced by binderless briquetting using IBC-105 coal as an *in-situ* binder generator. These form coking runs failed to produce briquettes of adequate strength for further characterization. Subsequent form coke samples were produced using a mild gasification pitch binder, as previously described. Of the remaining twelve samples made, seven of the briquettes were of adequate strength for further testing and characterization. The data on these form coke samples are shown in Table 11. Depending on the quantity of char available, some of the briquettes were made as duplicates, and the data in Table 11 show these in combined form. Seven of the form coking tests used an undesulfurized IFFR char (ICP1002), and the remaining eight tests used desulfurized chars D1113, D1215, and D1217. In nine of the tests, the briquettes were calcined under $N_2$ atmosphere, and the remaining six briquettes were calcined under a 3:1 (v/v) $H_2$:$CH_4$ atmosphere. In all form coking runs, the pressing force was 14,000 lb/in$^2$ and the hold time was 30 seconds. The briquetting temperature was 650°F in all runs but the last, R20, in which the briquetting temperature was 600°F. The carbonization temperature in all cases was 1800°F at atmospheric pressure.

Table 9, part 1. DESULFURIZATION TESTS WITH IFFR CHARS

| Desulfurization Test | D1113 | | D1117 | | D1123 | | D1208 | |
|---|---|---|---|---|---|---|---|---|
| **TEST CONDITIONS** | | | | | | | | |
| Test char | ICP1002 | | ICP1014 | | ICP1008 | | ICP1021 | |
| Acid-washed | No | | No | | No | | No | |
| Temperature, °F | 1400 | | 1400 | | 1400 | | 1400 | |
| Pressure, psig | 200 | | 200 | | 200 | | 200 | |
| Res Time, min | 120 | | 120 | | 120 | | 120 | |
| Vol% $CH_4$ | 23 | | 14 | | 20 | | 21 | |
| Vol% $H_2$ | 77 | | 86 | | 80 | | 79 | |
| **CHAR ANALYSES** | Feed | Spent | Feed | Spent | Feed | Spent | Feed | Spent |
| Proximate, wt% as rec'd | | | | | | | | |
| Moisture | 2.40 | 0.90 | 1.90 | 0.80 | 1.60 | 0.60 | 1.50 | 0.50 |
| Volatile Matter | 27.70 | 3.50 | 16.50 | 3.30 | 20.60 | 3.90 | 19.50 | 3.50 |
| Ash | 17.80 | 26.00 | 21.10 | 27.50 | 20.30 | 28.70 | 20.30 | 28.70 |
| Fixed Carbon | 52.00 | 69.50 | 60.20 | 68.20 | 57.30 | 66.60 | 58.50 | 67.20 |
| Ultimate, wt% dry | | | | | | | | |
| Ash | 18.25 | 26.25 | 21.59 | 27.74 | 20.68 | 28.98 | 20.70 | 28.86 |
| Carbon | 64.94 | 70.48 | 65.28 | 68.81 | 63.41 | 66.54 | 64.32 | 68.05 |
| Hydrogen | 3.76 | 0.72 | 2.32 | 0.76 | 2.91 | 0.66 | 2.79 | 0.78 |
| Nitrogen | 1.24 | 0.69 | 1.29 | 0.95 | 1.28 | 1.14 | 1.29 | 0.87 |
| Sulfur | 3.63 | 1.25 | 3.37 | 1.51 | 3.49 | 2.04 | 3.51 | 2.16 |
| Oxygen (diff) | 8.18 | 0.61 | 6.15 | 0.23 | 8.23 | 0.64 | 7.39 | -0.72 |
| **PHYSICAL PROPERTY DATA** | | | | | | | | |
| $V_{cp}$, $cm^3/g$ | 0.559 | -- | 0.693 | -- | 0.594 | -- | 0.581 | -- |
| $\rho_{He}$, $g/cm^3$ | 1.53 | -- | 1.62 | -- | 1.60 | -- | 1.59 | -- |
| $A_s(N_2)$, $m^2/g$ | 0.4 | -- | 0.6 | -- | 0.4 | -- | <0.5 | -- |
| $A_s(CO_2)$, $m^2/g$ | <2.0 | 384 | -- | 78 | <2.0 | -- | -- | -- |
| $L_c$, Å | 11 | -- | 11 | -- | 13 | -- | 13 | -- |
| **CONVERSION DATA** | | | | | | | | |
| S reduction, % | 65.6 | | 55.2 | | 41.6 | | 38.5 | |
| S conversion, % | 76.1 | | 65.1 | | 58.3 | | 55.9 | |
| C conversion, % | 24.6 | | 18.0 | | 25.1 | | 24.1 | |
| HHV conversion, % | 34.4 | | 23.2 | | 32.2 | | 30.0 | |
| S/C conv ratio | 3.1 | | 3.6 | | 2.3 | | 2.3 | |

Table 9, part 2. DESULFURIZATION TESTS WITH IFFR CHARS

| Desulfurization Test | D0317 | | D0322 | | D1215 | |
|---|---|---|---|---|---|---|
| **TEST CONDITIONS** | | | | | | |
| Test char | ICP1008 | | ICP1014 | | ICP1002AW | |
| Acid-washing | No | | No | | Yes | |
| Temperature, °F | 1400 | | 1400 | | 1400 | |
| Pressure, psig | 100 | | 100 | | 200 | |
| Res Time, min | 135 | | 135 | | 120 | |
| Vol% $CH_4$ | 44 | | 49 | | 23 | |
| Vol% $H_2$ | 56 | | 51 | | 77 | |
| **CHAR ANALYSES** | Feed | Spent | Feed | Spent | Feed | Spent |
| Proximate, wt% as rec'd | | | | | | |
| Moisture | 1.60 | 0.20 | 1.90 | 0.20 | 1.80 | 0.60 |
| Volatile Matter | 20.60 | 3.30 | 16.50 | 26.20 | 27.70 | 4.50 |
| Ash | 20.30 | 27.30 | 21.10 | 26.20 | 15.70 | 24.00 |
| Fixed Carbon | 57.50 | 69.20 | 60.20 | 70.80 | 54.50 | 70.70 |
| Ultimate, wt% dry | | | | | | |
| Ash | 20.68 | 27.37 | 21.59 | 26.32 | 16.06 | 24.24 |
| Carbon | 63.41 | 69.05 | 65.28 | 70.47 | 67.26 | 71.80 |
| Hydrogen | 2.91 | 0.79 | 2.32 | 0.85 | 3.69 | 0.94 |
| Nitrogen | 1.28 | 1.12 | 1.29 | 0.78 | 1.31 | 0.97 |
| Sulfur | 3.49 | 2.52 | 3.37 | 2.26 | 2.95 | 0.20 |
| Oxygen (diff) | 8.23 | -0.85 | 6.15 | -0.68 | 8.73 | 1.85 |
| **PHYSICAL PROPERTY DATA** | | | | | | |
| $V_{cp}$, $cm^3/g$ | 0.594 | -- | 0.693 | -- | 0.562 | -- |
| $\rho_{He}$, $g/cm^3$ | 1.60 | -- | 1.62 | -- | 1.48 | -- |
| $A_s(N_2)$, $m^2/g$ | 0.4 | -- | 0.6 | -- | 0.8 | -- |
| $A_s(CO_2)$, $m^2/g$ | <2.0 | -- | -- | -- | <2.0 | 435 |
| $L_c$, Å | 13 | -- | 11 | -- | 10 | -- |
| **CONVERSION DATA** | | | | | | |
| S reduction, % | 27.8 | | 32.9 | | 93.2 | |
| S conversion, % | 45.4 | | 45.0 | | 95.5 | |
| C conversion, % | 17.7 | | 11.5 | | 29.3 | |
| HHV conversion, % | 24.3 | | 16.1 | | 37.6 | |
| S/C conv ratio | 2.56 | | 3.93 | | 3.3 | |

Table 9, part 3. DESULFURIZATION TESTS WITH IFFR CHARS

| Desulfurization Test | D1217 | | D1222 | | D0315 | |
|---|---|---|---|---|---|---|
| TEST CONDITIONS | | | | | | |
| Test char | ICP1021AW | | ICP1014AW | | ICP1008AW | |
| Acid-washed | Yes | | Yes | | Yes | |
| Temperature, °F | 1400 | | 1400 | | 1400 | |
| Pressure, psig | 200 | | 200 | | 100 | |
| Res Time, min | 120 | | 120 | | 135 | |
| Vol% $CH_4$ | 14 | | 81 | | 21 | |
| Vol% $H_2$ | 86 | | 19 | | 79 | |
| CHAR ANALYSES | Feed | Spent | Feed | Spent | Feed | Spent |
| Proximate, wt% as rec'd | | | | | | |
| Moisture | 0.90 | 0.40 | 1.40 | 0.40 | 1.20 | 0.50 |
| Volatile Matter | 18.80 | 3.90 | 15.70 | 3.70 | 20.20 | 3.40 |
| Ash | 18.80 | 25.70 | 19.80 | 25.00 | 18.10 | 24.30 |
| Fixed Carbon | 61.30 | 69.80 | 62.90 | 70.70 | 60.50 | 71.80 |
| Ultimate, wt% dry | | | | | | |
| Ash | 19.00 | 25.87 | 20.14 | 25.13 | 18.39 | 24.51 |
| Carbon | 65.57 | 69.14 | 66.98 | 71.52 | 65.68 | 73.06 |
| Hydrogen | 2.96 | 0.88 | 2.48 | 0.97 | 3.05 | 0.92 |
| Nitrogen | 1.32 | 0.91 | 1.26 | 1.03 | 1.29 | 1.26 |
| Sulfur | 3.22 | 0.70 | 3.08 | 0.76 | 3.04 | 0.72 |
| Oxygen (diff) | 7.93 | 2.50 | 6.06 | 0.59 | 8.55 | -0.47 |
| PHYSICAL PROPERTY DATA | | | | | | |
| $V_{cp}$, $cm^3/g$ | 0.554 | -- | 0.679 | -- | -- | -- |
| $\rho_{He}$, $g/cm^3$ | 1.60 | -- | 1.62 | -- | -- | -- |
| $A_s(N_2)$, $m^2/g$ | 1.4 | -- | 1.0 | -- | -- | -- |
| $A_s(CO_2)$, $m^2/g$ | -- | -- | <2.0 | -- | -- | -- |
| $L_c$, Å | 13 | -- | 12 | -- | -- | -- |
| CONVERSION DATA | | | | | | |
| S reduction, % | 78.3 | | 75.3 | | 76.3 | |
| S conversion, % | 84.0 | | 80.2 | | 82.2 | |
| C conversion, % | 22.6 | | 14.4 | | 16.5 | |
| HHV conversion, % | 29.8 | | 19.9 | | 23.4 | |
| S/C conv ratio | 3.7 | | 5.6 | | 4.97 | |

## Table 10, part 1. DESULFURIZATION TESTS WITH MBFBR CHARS

| Desulfurization Test | D0224 | | D0301 | | D0308 | |
|---|---|---|---|---|---|---|
| **TEST CONDITIONS** | | | | | | |
| Test char | FCP0106 | | FCP0112 | | FCP0120 | |
| Acid-washed | No | | No | | Yes (coal) | |
| Temperature, °F | 1400 | | 1400 | | 1400 | |
| Pressure, psig | 200 | | 200 | | 200 | |
| Res Time, min | 240 | | 240 | | 240 | |
| Vol% $CH_4$ | 21 | | 21 | | 21 | |
| Vol% $H_2$ | 79 | | 79 | | 79 | |
| **CHAR ANALYSES** | Feed | Spent | Feed | Spent | Feed | Spent |
| Proximate, wt% as rec'd | | | | | | |
| Moisture | 0.66 | 0.20 | 0.64 | 0.30 | 0.90 | 0.30 |
| Volatile Matter | 14.90 | 2.90 | 9.22 | 3.40 | 13.89 | 3.60 |
| Ash | 20.97 | 33.20 | 20.39 | 28.10 | 13.16 | 16.20 |
| Fixed Carbon | 63.47 | 63.70 | 69.75 | 68.20 | 72.05 | 79.90 |
| Ultimate, wt% dry | | | | | | |
| Ash | 18.25 | 26.25 | 21.59 | 27.74 | 20.68 | 28.98 |
| Carbon | 66.81 | 71.03 | 71.81 | 74.53 | 73.46 | 80.52 |
| Hydrogen | 2.37 | 0.89 | 1.46 | 0.98 | 2.58 | 1.06 |
| Nitrogen | 1.63 | 0.89 | 1.64 | 1.03 | 1.80 | 1.12 |
| Sulfur | 2.43 | 0.41 | 2.75 | 0.51 | 2.11 | 0.10 |
| Oxygen (diff) | 5.65 | 1.20 | 1.82 | -0.41 | 6.77 | 0.80 |
| **PHYSICAL PROPERTY DATA** | | | | | | |
| $V_{cp}$, $cm^3/g$ | 0.206 | -- | 0.205 | -- | 0.266 | -- |
| $\rho_{He}$, $g/cm^3$ | 1.59 | -- | 1.79 | -- | 1.62 | -- |
| $A_s(N_2)$, $m^2/g$ | 0.4 | -- | 5.0 | -- | <0.5 | -- |
| $A_s(CO_2)$, $m^2/g$ | 189 | -- | 299 | -- | 267 | -- |
| $L_c$, Å | 13 | -- | 11 | -- | 10 | -- |
| **CONVERSION DATA** | | | | | | |
| S reduction, % | 83.1 | | 81.5 | | 95.3 | |
| S conversion, % | 86.1 | | 83.7 | | 96.2 | |
| C conversion, % | 12.3 | | 8.8 | | 11.2 | |
| HHV conversion, % | 17.8 | | 10.7 | | 16.0 | |
| S/C conv ratio | 7.0 | | 9.5 | | 8.6 | |

## Table 10, part 2. DESULFURIZATION TESTS WITH MBFBR CHARS

| Desulfurization Test | D0308 | | D0310 | |
|---|---|---|---|---|
| **TEST CONDITIONS** | | | | |
| Test char | FCP0202 | | FCP0106 | |
| Acid-washed | Yes (coal) | | No | |
| Temperature, °F | 1400 | | 1400 | |
| Pressure, psig | 200 | | 100 | |
| Res Time, min | 240 | | 135 | |
| Vol% $CH_4$ | 21 | | 21 | |
| Vol% $H_2$ | 79 | | 79 | |
| **CHAR ANALYSES** | Feed | Spent | Feed | Spent |
| Proximate, wt% as rec'd | | | | |
| Moisture | 0.77 | 0.50 | 0.66 | 0.40 |
| Volatile Matter | 9.33 | 2.90 | 14.90 | 3.50 |
| Ash | 22.25 | 33.30 | 20.97 | 31.80 |
| Fixed Carbon | 67.65 | 63.30 | 63.47 | 64.30 |
| Ultimate, wt% dry | | | | |
| Ash | 20.70 | 28.86 | | |
| Carbon | 68.70 | 71.96 | 66.81 | 73.14 |
| Hydrogen | 1.55 | 0.97 | 2.37 | 0.88 |
| Nitrogen | 1.61 | 1.11 | 1.63 | 1.17 |
| Sulfur | 2.06 | 0.10 | 2.43 | 0.54 |
| Oxygen (diff) | 3.66 | -0.13 | 2.43 | -0.15 |
| **PHYSICAL PROPERTY DATA** | | | | |
| $V_{cp}$, $cm^3/g$ | 0.229 | -- | 0.206 | -- |
| $\rho_{He}$, $g/cm^3$ | 1.81 | -- | 1.59 | -- |
| $A_s(N_2)$, $m^2/g$ | 2.0 | -- | 0.4 | -- |
| $A_s(CO_2)$, $m^2/g$ | 261 | -- | 189 | -- |
| $L_c$, Å | 11 | -- | 13 | -- |
| **CONVERSION DATA** | | | | |
| S reduction, % | 95.2 | | 77.8 | |
| S conversion, % | 95.8 | | 80.8 | |
| C conversion, % | 9.6 | | 5.36 | |
| HHV conversion, % | 11.4 | | 10.9 | |
| S/C conv ratio | 9.9 | | 15.1 | |

Table 11, part 1. CARBONIZATION-DESULFURIZATION TESTS WITH FORM COKE BRIQUETTES

| Form Coking Test | R9/R10 | | R11/R12 | | R13 | | R14/R15 | |
|---|---|---|---|---|---|---|---|---|
| **TEST CONDITIONS** | | | | | | | | |
| Test char | ICP1002 | | ICP1002 | | D1113 | | D1113 | |
| Acid-washed | No | | No | | No | | No | |
| Desulfurized | No | | No | | Yes | | Yes | |
| Pitch binder, wt% | 18 | | 18 | | 18 | | 18 | |
| Carbonization atmosphere | $N_2$ | | $H_2/CH_4$ | | $N_2$ | | $H_2/CH_4$ | |
| **ANALYSES** | Feed char | Form coke | Feed char | Form coke | Feed char | Form coke | Feed char | Form coke |
| Proximate, wt% as rec'd | | | | | | | | |
| Moisture | 2.4 | 6.3 | 2.4 | 2.2 | 0.9 | 5.2 | 0.9 | 2.2 |
| Volatile Matter | 27.7 | 4.4 | 27.7 | 2.6 | 3.5 | 3.4 | 3.5 | 2.6 |
| Ash | 17.8 | 18.1 | 17.8 | 21.1 | 26.0 | 22.2 | 26.0 | 24.1 |
| Fixed Carbon | 52.0 | 71.0 | 52.0 | 73.9 | 69.5 | 69.0 | 69.5 | 70.9 |
| Ultimate, wt% dry | | | | | | | | |
| Ash | 18.25 | 19.41 | 18.25 | 21.65 | 26.25 | 23.46 | 26.25 | 24.74 |
| Carbon | 64.94 | 76.04 | 64.94 | 75.91 | 70.48 | 74.88 | 70.48 | 74.68 |
| Hydrogen | 3.76 | 0.23 | 3.76 | 0.28 | 0.72 | 0.17 | 0.72 | 0.25 |
| Nitrogen | 1.24 | 0.91 | 1.24 | 0.54 | 0.69 | 0.57 | 0.69 | 0.33 |
| Sulfur | 3.63 | 3.03 | 3.63 | 2.29 | 1.25 | 1.58 | 1.25 | 1.24 |
| Oxygen (diff) | 8.18 | 0.38 | 8.18 | -0.67 | 0.61 | -0.66 | 0.61 | -1.24 |
| **PHYSICAL PROPERTY DATA** | | | | | | | | |
| Tensile strength, $lb/in^2$ | -- | frac* | -- | 172 | -- | frac | -- | 837 |
| $\rho_b$, $g/cm^3$ | -- | 1.41 | -- | 1.35 | -- | -- | -- | 1.14 |

* "frac" = Briquette fractured during handling

Table 11, part 2. CARBONIZATION-DESULFURIZATION TESTS WITH FORM COKE BRIQUETTES

| Form Coking Test | R16 | | R17 | | R18/19 | | R20 | |
|---|---|---|---|---|---|---|---|---|
| TEST CONDITIONS | | | | | | | | |
| Test char | D1215 | | D1217 | | D1217 | | D1113 | |
| Acid-washed | Yes | | Yes | | Yes | | No | |
| Desulfurized | Yes | | Yes | | Yes | | Yes | |
| Pitch binder, wt% | 18 | | 18 | | 18 | | 51 | |
| Carbonization atmosphere | $N_2$ | | $N_2$ | | $H_2/CH_4$ | | $N_2$ | |
| ANALYSES | Feed char | Form coke | Feed char | Form coke | Feed char | Form coke | Feed char | Form coke |
| Proximate, wt% as rec'd | | | | | | | | |
| Moisture | 0.6 | 5.8 | 0.4 | 5.1 | 0.4 | 3.7 | 0.9 | -- |
| Volatile Matter | 4.5 | 4.1 | 3.9 | 3.5 | 3.9 | 3.0 | 3.5 | -- |
| Ash | 24.0 | 20.5 | 25.7 | 22.3 | 25.7 | 21.5 | 26.0 | -- |
| Fixed Carbon | 70.7 | 69.4 | 69.8 | 68.9 | 69.8 | 71.6 | 69.5 | -- |
| Ultimate, wt% dry | | | | | | | | |
| Ash | 24.24 | 21.80 | 25.87 | 23.57 | 25.87 | 22.40 | 26.25 | -- |
| Carbon | 71.80 | 75.78 | 69.14 | 73.80 | 69.14 | 75.61 | 70.48 | -- |
| Hydrogen | 0.94 | 0.16 | 0.88 | 0.24 | 0.88 | 0.35 | 0.72 | -- |
| Nitrogen | 0.97 | 0.58 | 0.91 | 0.63 | 0.91 | 0.45 | 0.69 | -- |
| Sulfur | 0.20 | 0.51 | 0.70 | 0.93 | 0.70 | 0.48 | 1.25 | -- |
| Oxygen (diff) | 1.85 | 1.17 | 2.50 | 0.83 | 2.50 | 0.71 | 0.61 | -- |
| PHYSICAL PROPERTY DATA | | | | | | | | |
| Tensile strength, $lb/in^2$ | -- | frac* | -- | 80 | -- | 294 | -- | 84 |
| $\rho_b$, $g/cm^3$ | -- | -- | -- | 1.01 | -- | 1.08 | -- | 1.01 |

* "frac" = Briquette fractured during handling

## Task 4. Data Analysis and Interpretation

IFFR Chars: Desulfurization of the IFFR chars was significant in all cases. Acid-washing of the IFFR chars dramatically improved sulfur release from the char, regardless of the temperature at which the char was produced. The data in Table 9 show that sulfur reduction exceeding 95% can be achieved for IFFR chars by combining acid-washing with desulfurization in $H_2$:$CH_4$ atmosphere.

Figure 9 shows the sulfur content of desulfurized IFFR chars as a function of the mild gasification (IFFR) temperature used to produce the char. The original sulfur content of the parent coal was 4.3 wt%, and the IFFR chars prior to desulfurization contained 3.0-3.6 wt% sulfur. For the desulfurization conditions used in these tests, acid-washing of the IFFR char was necessary to achieve a final sulfur content below 1.0 wt% after desulfurization. For either unwashed or acid-washed chars, the char susceptibility to desulfurization was greatest at the lowest mild gasification temperature of 1000°F, but an apparent minimum was observed for the unwashed char in the 1100°-1200°F region. Compared with unwashed chars, acid-washed IFFR chars released 50%-84% more of the original char sulfur, with final sulfur contents as low as 0.20 wt%. However, dropping the system pressure from 200 psig ($P_{H2}$ = 170 psi) to 100 psig ($P_{H2}$ = 91 psi) had little effect on the degree of sulfur removal from acid-washed IFFR char. Also, in two tests on unwashed IFFR chars at 100 psig and 50 vol% $H_2$, the sulfur removal decreased by 33-40% compared to tests at the same $P_{CH4}$ (54-58 psi), but at a higher $P_{H2}$ (170 psi versus 58 psi).

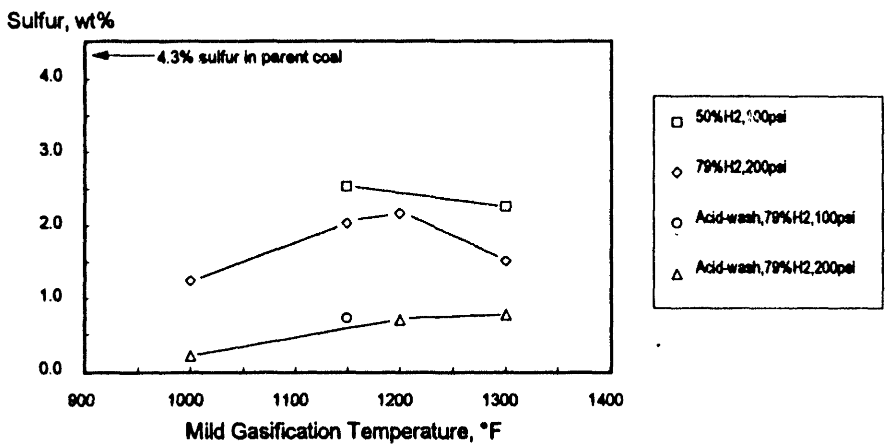



Figure 9. SULFUR CONTENT OF DESULFURIZED IFFR CHARS VERSUS MILD GASIFICATION TEMPERATURE

MBFBR Chars: From the data in Table 10, it can be seen that MBFBR chars can be reduced in sulfur below the target level of 1.0 wt%. Indeed, the MBFBR chars were more easily desulfurized than the less dense IFFR chars, which reported higher total pore volumes but much lower $CO_2$ surface areas. The residual sulfur contents of MBFBR chars after desulfurization, related to the mild gasification temperatures used to produce the char, are shown in Figure 10.

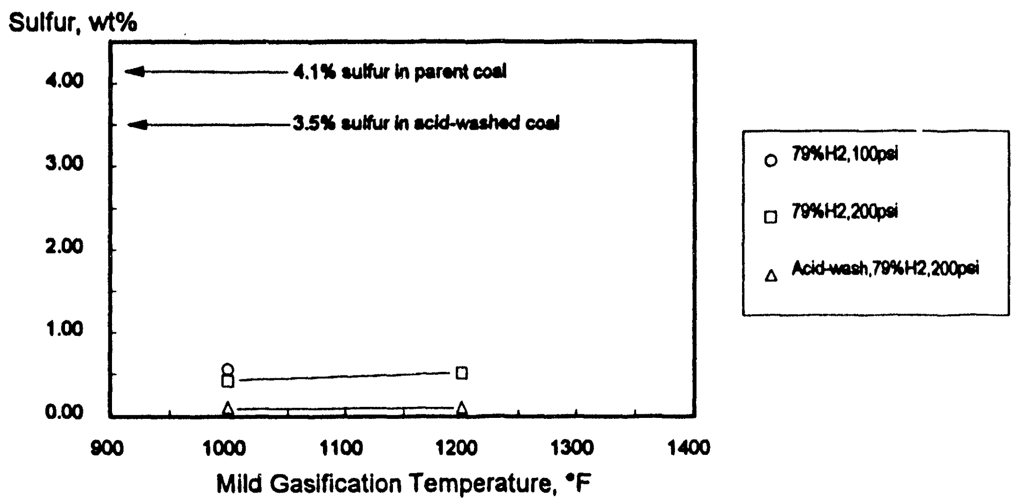



Figure 10. SULFUR CONTENT OF DESULFURIZED MBFBR CHARS VERSUS MILD GASIFICATION TEMPERATURE

For the MBFBR chars, acid-washing did not have as dramatic an effect as for the IFFR chars, mainly because sulfur removal was already higher for MBFBR chars from unwashed coal than for unwashed IFFR chars. Even so, desulfurization of MBFBR chars from acid-washed coal produced the lowest-sulfur chars in this program, with 0.10 wt% sulfur. The sulfur content of the parent coal was 4.3 wt%, and the MBFBR chars contained 2.1-2.8 wt% sulfur. Even at 100 psig system pressure ($P_{H2}$ = 91 psi), the sulfur removal was over 70%, and the spent char, with 0.54 wt% sulfur, easily exceeded the target sulfur level of 1.0 wt%. The influence of mild gasification temperature on desulfurization of MBFBR chars was negligible.

Carbon Conversion: The data on carbon gasification also show interesting effects of mild gasification temperature and reactor type. Figure 11 shows the carbon conversion data for the IFFR chars, and Figure 12 shows analogous data for the MBFBR chars.

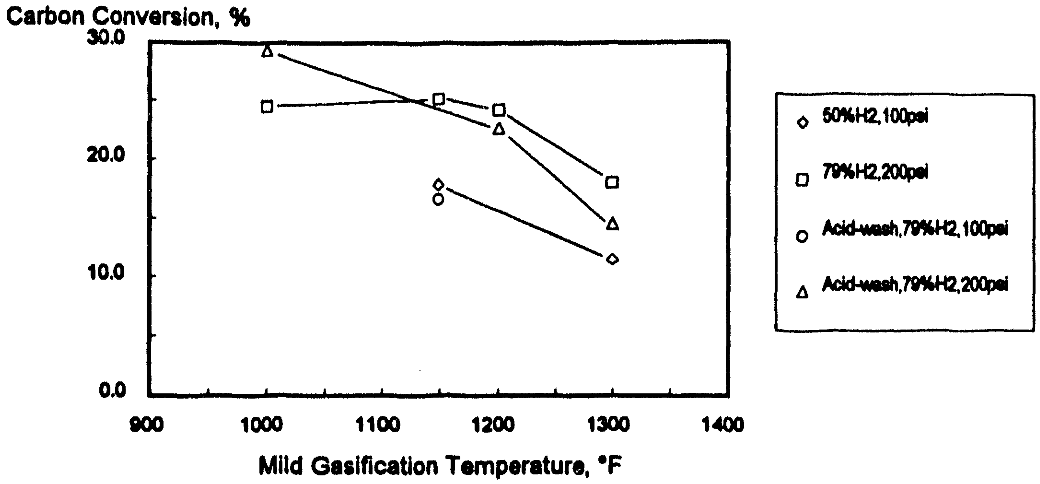



Figure 11. CARBON CONVERSION FROM IFFR CHARS VERSUS MILD GASIFICATION TEMPERATURE

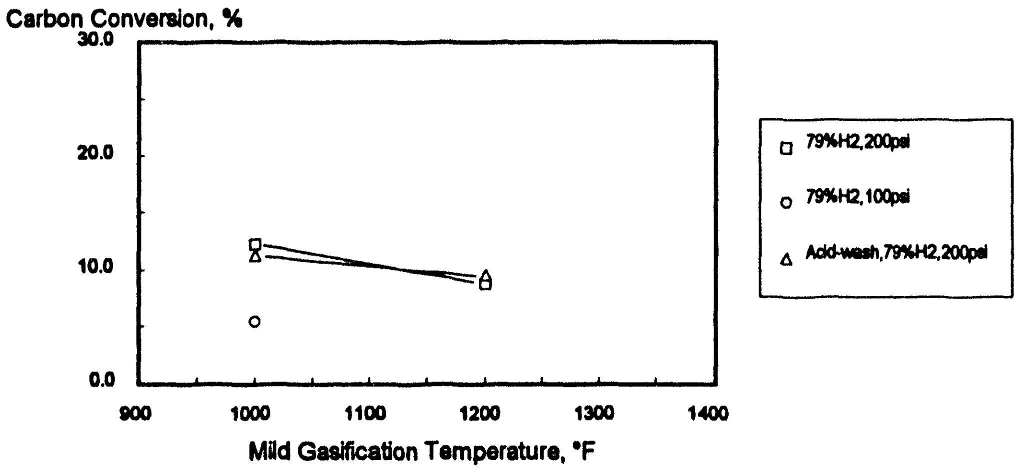



Figure 12. CARBON CONVERSION FROM MBFBR CHARS VERSUS MILD GASIFICATION TEMPERATURE

As with sulfur conversion, carbon conversion for IFFR chars generally decreased as the temperature at which the char was produced increased. However the data suggest that there are optimum conditions of $H_2$:$CH_4$ ratio and system pressure which can maximize the selectivity of sulfur conversion at minimal carbon loss. For example, with 1150°-1200°F acid-washed IFFR chars, reducing the system pressure from 200 to 100 psig at 75% $H_2$ decreased the carbon loss from 25% to 17%, while the sulfur removal remained essentially constant at 76%-78%. A lower system pressure also reduced carbon conversion for a 1000°F MBFBR char, from 12% to 5%, while sulfur removal only dropped slightly, from 83% to 78%.

Physical Properties: It was previously noted that IFFR mild gasification appeared to result in nearly complete micropore collapse, evidenced by the very low $CO_2$ surface areas measured in IFFR chars prior to desulfurization. This does not appear to be true of the MBFBR chars, which increased in $CO_2$ surface area by 50-137% after mild gasification. With MBFBR chars, the desulfurization data did not correlate with density or crystallite size as did the IFFR char data. It can be stated qualitatively that, for fluidized-bed chars, the skeletal density, crystallinity, micropore surface area, and mineral matter composition all contribute in a complex manner to determine the desulfurization susceptibility of fluidized-bed chars.

The correlations between desulfurization susceptibility of IFFR chars and helium density and crystallinity are shown in Figures 13 and 14. The data represented on these plots include data from the first year, using MILDGAS PRU chars, as well as the current data with IFFR chars.

Form Coke: Form coke briquettes were produced from several IFFR chars and tested. The form coke data shown in Table 11 indicate that carbonization of the green briquette in a reducing atmosphere, even at low pressure, can remove additional sulfur. Figure 17 compares the tensile strength

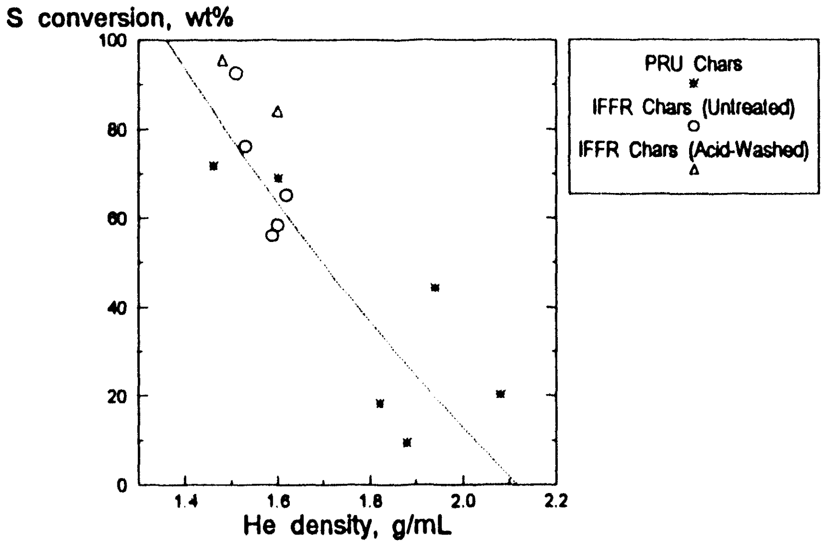



Figure 13. SULFUR CONVERSION VERSUS HELIUM DENSITY FOR IFFR MILD GASIFICATION CHARS

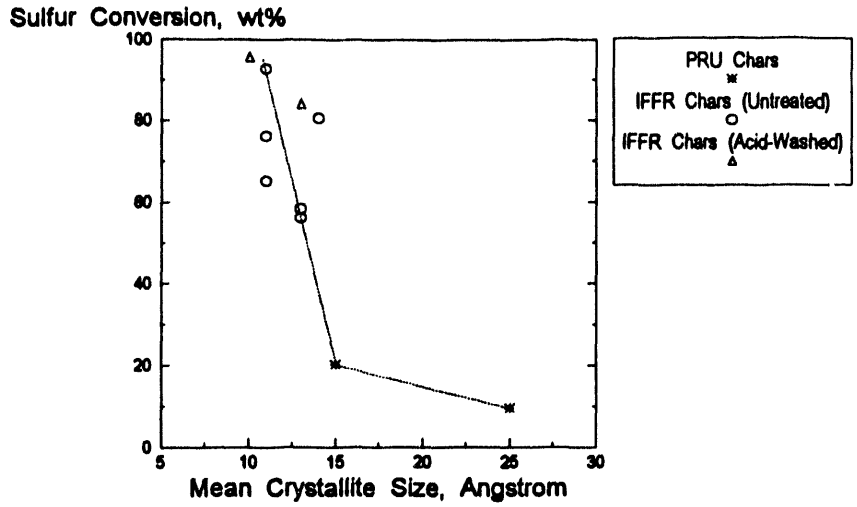



Figure 16. SULFUR CONVERSION VERSUS MEAN CRYSTALLITE SIZE FOR IFFR MILD GASIFICATION CHARS

of test briquettes made in this program with commercial cokes and form coke samples produced in the DOE-sponsored MILDGAS development program. Two of the briquettes produced here showed strength values comparable to those of commercial cokes, and one (R14/15) had a tensile strength comparable to the strongest coke previously tested. Although these diametral tensile strength data cannot be considered definitive measurements of coke strength performance in a blast furnace, it is valuable as a screening tool to show that the desulfurized IBC-105 char can produce a strong briquette under the proper conditions, and that therefore further development is warranted.

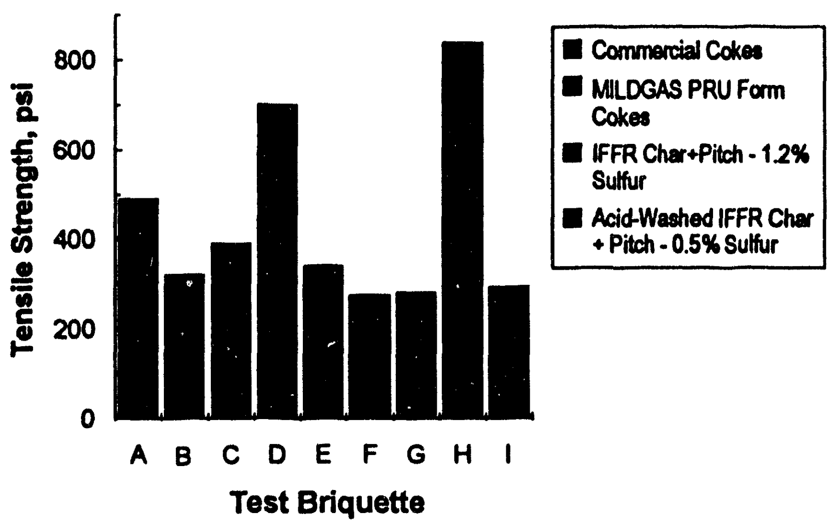



Figure 18. FORM COKE DIAMETRAL TENSILE STRENGTH

## CONCLUSIONS AND RECOMMENDATIONS

- Mild gasification of IBC-105 coal results in 16% to 22% reduction in sulfur content, with more sulfur removed as mild gasification temperature increases; subsequent char desulfurization with $H_2:CH_4$ mixtures can remove up to 79% additional sulfur.
- Lower mild gasification temperatures favor the subsequent desulfurization by $CH_4:H_2$ mixtures.
- Fluidized-bed chars are easier to desulfurize than entrained chars.
- Acid-washing of the coal or the mild gasification char greatly enhances the desulfurization susceptibility of the char, in addition to removing 9-30% of the ash and 10-22% of the sulfur from the coal or char.
- At 1400°F, $H_2$ partial pressures of about 70 psig appear to be adequate to obtain a char with 1 wt% sulfur, and even lower $H_2$ pressures may be sufficient if acid-washing of the coal is performed prior to mild gasification.
- Entrained mild gasification increases the connected (macro-)pore volume and decreases density, but reduces surface area by essentially eliminating micropores.
- Fluidized-bed mild gasification produces denser, less porous chars, but does not result in micropore collapse, and thus internal surface area actually increases, resulting in greater susceptibility to desulfurization.
- Desulfurization of entrained chars correlates well with the helium density and mean crystallite size, but fluidized-bed chars appear to follow more complicated correlations involving microporosity.

A follow-on study will begin in FY1994 to obtain data in a continuous bench-scale unit. In a batch reactor, the $H_2S$ content of the gas atmosphere in the bed continuously decreases as sulfur is removed from the char, which is not the case in a continuous reactor, where a steady-state $H_2S$ level is reached. The follow-on program will address this aspect of the process. Studies of form coke from desulfurized Illinois No. 6 chars, especially focusing on fluidized-bed chars, will also continue.

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**
June 1 through August 31, 1993

Project Title: **INVESTIGATION OF A SULFUR REDUCTION TECHNIQUE FOR MILD GASIFICATION CHAR**

Principal Investigator: Richard Knight, Institute of Gas Technology

Project Monitor: Dan Banerjee, ICCI

COMMENTS

No changes to budget or management were made during this quarter.

## Projected and Estimated Actual Expenditures by Quarter

**INVESTIGATION OF A SULFUR REDUCTION TECHNIQUE FOR MILD GASIFICATION CHAR**

| Quarter* | Types of Cost | Direct Labor | Fringe Benefits | Materials & Supplies | Travel | Major Equipment | Other Direct Costs | Indirect Costs | Total |
|---|---|---|---|---|---|---|---|---|---|
| Sept 1, 1992 to Nov 30, 1992 | Projected | 8,500 | | 1,000 | | 3,630 | | 16,334 | 29,464 |
| | Estimated | 14,561 | | 2,532 | | | | 26,143 | 43,236 |
| Sept 1, 1992 to Feb 28, 1993 | Projected | 20,000 | | 4,220 | | 1,580 | | 37,221 | 62,351 |
| | Estimated | 33,121 | | 4,510 | | 931 | | 59,397 | 97,959 |
| Sept 1, 1992 to May 31, 1993 | Projected | 32,000 | | 4,220 | 260 | 1,580 | | 59,015 | 96,655 |
| | Estimated | 40,422 | | 4,492 | | 1,206 | | 72,283 | 118,403 |
| Sept 1, 1992 to Aug 31, 1993 | Projected | 41,435 | | 4,220 | 260 | 1,580 | | 74,143 | 121,638 |
| | Estimated | 41,602 | | 4,385 | 92 | 1,206 | | 74,353 | 121,638 |

* Cumulative by Quarter

Equipment Inventory
September 1, 1992, through August 31, 1993

Project Title: Investigation of a Sulfur Reduction Technique for Mild Gasification Char

Principal Investigator: Richard A. Knight, Institute of Gas Technology

Department: Process Research

List of Equipment Purchased

| Acct.# | Date Acquired | Short Description | Cost | Tag# | Bldg. & Rm.# |
|---|---|---|---|---|---|
| 40314-02 | 12-01-92 | Custom heating mantle | $865 | none | South B134 |
| 40314-02 | 1-14-93 | Heat gun | $116 | none | Central B33 |
| 40314-02 | 1-14-93 | Digital multimeter | $150 | none | Central B33 |

List of Publications and Presentations
September 1, 1992 through August 31, 1993

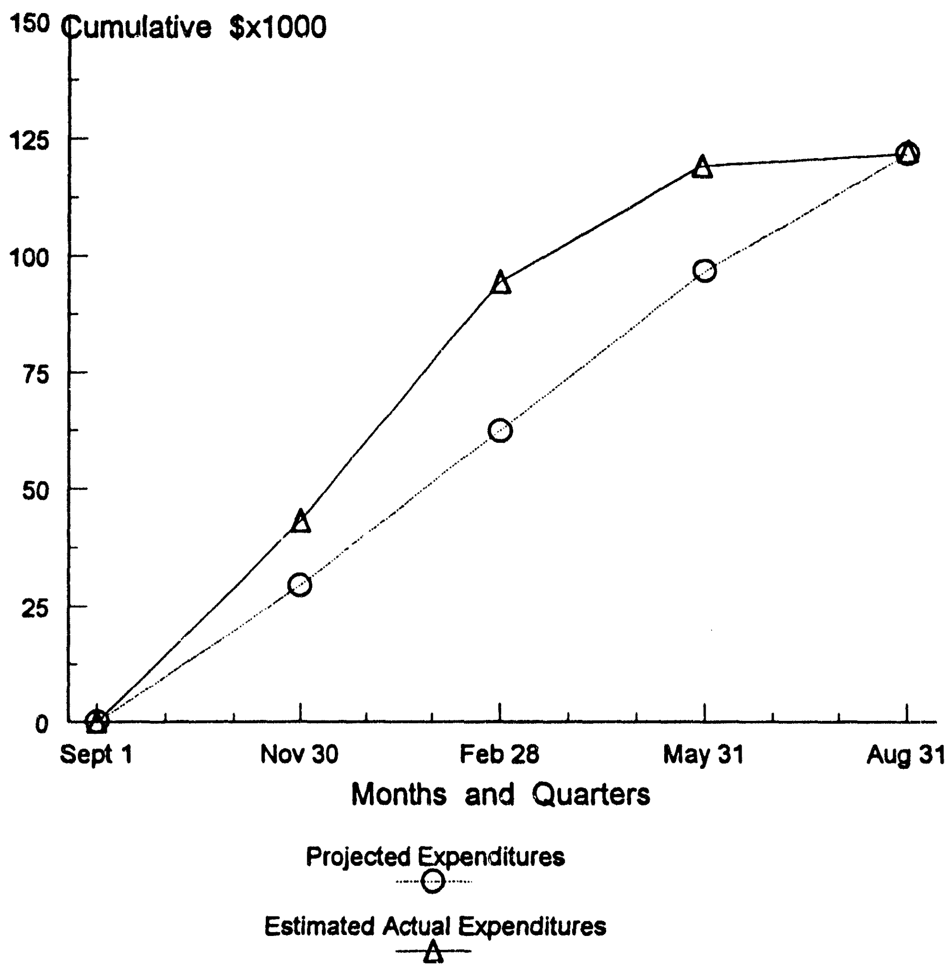
Project Title: Investigation of a Sulfur Reduction Technique for Mild Gasification Char

Principal Investigator: Richard A. Knight, Institute of Gas Technology
Project Monitor: Dan Banerjee, ICCI

Knight, R. A. 1993. "Investigation of a Sulfur Reduction Technique for Mild Gasification Char". Poster presentation at Eleventh Annual Contractors' Technical Meeting, Illinois Coal Development Board, Department of Energy and Natural Resources, August 3-5, 1993, Urbana, Illinois.

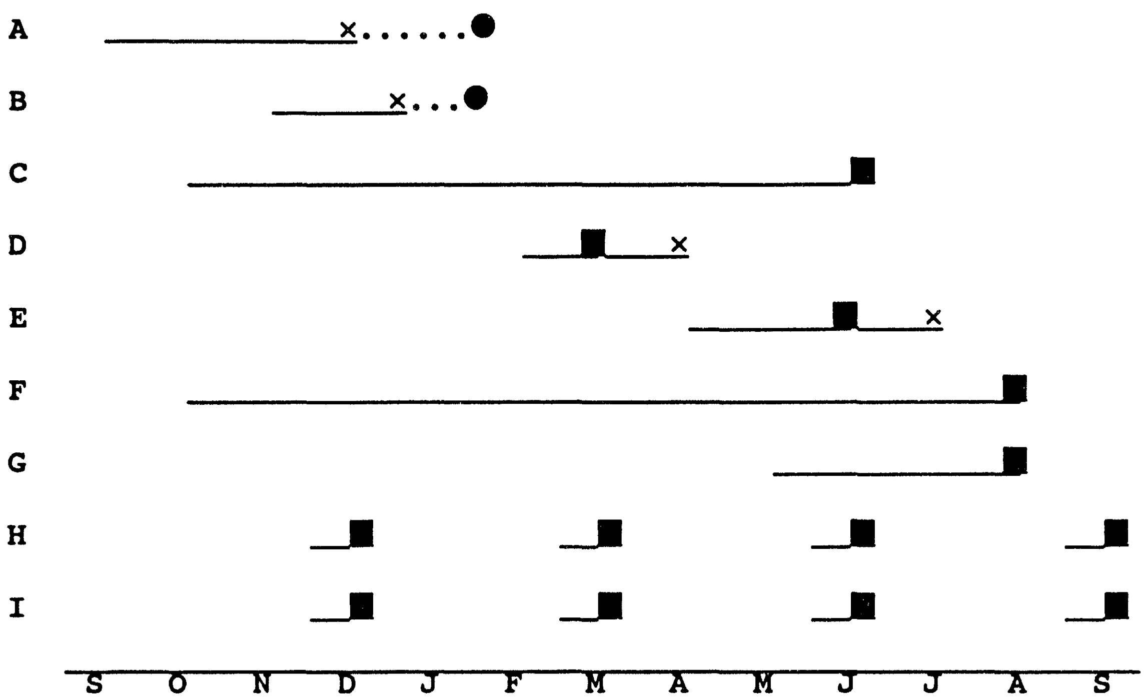
**COSTS BY QUARTER**

**INVESTIGATION OF A SULFUR REDUCTION TECHNIQUE FOR MILD GASIFICATION CHAR**




Illinois Clean Coal Institute Award = $121,638

# SCHEDULE OF PROJECT MILESTONES




Begin
Sept. 1
1992

Hypothetical Milestones:

A. Char samples prepared and characterized (Task 1)
B. Acid-washed chars prepared (Task 1)
C. Char desulfurization experiments completed (Task 3)
D. Briquette preparation equipment shaken down (Task 2)
E. Form coke briquettes prepared and characterized (Task 1)
F. Experimental data analyzed and interpreted (Task 4)
G. Conceptual process design developed (Task 5)
H. Technical reports prepared and submitted
I. Project management reports prepared and submitted

Comments:

None

# HAZARDOUS WASTE REPORT
September 1, 1992, through August 31, 1993

Project Title: Investigation of a Technique for Sulfur Reduction of Mild Gasification Char

Principal Investigator: Richard A. Knight
Organization: Institute of Gas Technology
Department: Process Research

## List of Hazardous Wastes

| Description of Substance | EPA Hazardous Waste Number | EPA Hazard Code | Actual Quantity Disposed | Trans-porter | Disposal Facility | Treat-ment Method |
|---|---|---|---|---|---|---|
| Spent halogenated solvent (methylene chloride) | F002 | (T) | 20 lb | Note *a* | Note *a* | Note *b* |
| Spent solvent (acetone) | F003 | (T) | 75 lb | Note *a* | Note *a* | Note *b* |
| Container residue (acetone) | U002 | (I) | 1 lb | Note *a* | Note *a* | Note *b* |
| Container residue (methylene chloride) | U188 | none | 1 lb | Note *a* | Note *a* | Note *b* |
| Container residue (tetrahydro-furan) | U213 | (I) | 1 lb | Note *a* | Note *a* | Note *b* |

a. There was no transportation or disposal of wastes from this project during the reporting period. The quantities listed are accumulating in collection tanks on-site, and will be disposed of by a qualified licensed disposal company that will be selected by competitive bid. The last disposal of IGT laboratory wastes occurred in June 1992, and was conducted by SET Environmental Inc., 450 Sumac Road, Wheeling, IL 60090. It is anticipated that the next disposal activity will take place in Fall of 1993.

b. Incineration and/or blending with non-hazardous wastes for landfill.

DATE
FILMED
4 / 7 / 94

END