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TECHNICAL REPORT
December 1, 1991 through February 29, 1992

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

Principal Investigator: Richard A. Knight, Institute of Gas Technology
Project Monitor: Frank Honea, CRSC

ABSTRACT

The object of this program is to investigate the desulfurization of mild gasification char using H_2:CH_4 mixtures in a laboratory-scale experimental study. Mild gasification is a coal conversion technique which produces solid, liquid, and gaseous co-products. Char is the major co-product, about 60% of the dry coal yield. Form coke for steelmaking and foundries presents the best potential high-value markets for chars from eastern bituminous coals. Conventional metallurgical cokes generally contain about 1 wt% or less sulfur. Mild gasification char from high-sulfur Illinois coals must be upgraded to meet these criteria. One method to accomplish this is desulfurization with reducing gases derived from the mild gasification co-product gases. Because form coke has a market value up to $200/ton, it can accommodate desulfurization costs and still be economically attractive. The desulfurization can be performed either on the granular char or on formed briquettes.

In the first year of the two-year program, granular char is being treated with H_2:CH_4 blends at temperatures of 1100°-1550°F and pressures of 50-100 psig. The effects of temperature, pressure, residence time, gas velocity, and gas composition on sulfur removal and carbon gasification are being determined. The batch experiments are being performed in a nominal 2-inch-ID stainless-steel, batch fluidized-bed reactor. The test char was produced by the IGT 100-lb/h process research unit (PRU) in a recently completed DOE/METC-sponsored program. The parent coal was Illinois No. 6 from a preparation plant, and the char from the selected test contains 4.58 wt% sulfur.

In the second year, the study will be extended to include form coke briquettes made from mild gasification char. The data obtained from both years of the program will be used to design a desulfurization process to be integrated with the IGT mild gasification process.

During the second quarter, eight tests were performed with the selected char. Fluidized-bed tests were conducted at 1290°-1575°F, 50-100 psig, and 30-60 min residence time. The bed gas velocity was varied from 0.10 to 0.15 ft/s, and the gases used were either 10% or 25% CH_4 in H_2. The data from these tests show desulfurization between 2.3% and 13.3%, with carbon conversions from 1.2% to 4.0%. The maximum desulfurization was 13.3% at 1300°F and 100 psig in 90% H_2 for 30 minutes residence time. Exit gas H_2S concentrations were 155-404 ppm, with maximum H_2S concentrations occurring in the first 15 minutes.

Future tests will focus on increasing desulfurization by increasing residence time and reducing H_2S concentration by increasing gas velocity.

MASTER

This project is funded by the U. S. Department of Energy (METC) and by the Illinois Department of Energy and Natural Resources as part of their cost-shared program.

EXECUTIVE SUMMARY

The object 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° to 1300°F, <50 psig). A mild gasification process which uses a coaxial fluidized-bed/entrained-bed reactor system has been developed by IGT under U.S. DOE sponsorship (DOE Contract DE-AC21-87MC24266). Char is the major co-product, comprising about 60% to 70% of the yield from dry coal.

One major target use for mild gasification char is metallurgical form coke. Form coke for blast furnaces and foundries has a higher market value at low sulfur content. Conventional cokes generally contain about 1 wt% sulfur. Mild gasification chars from high-sulfur Illinois coals contain up to 3 wt% sulfur and must therefore be upgraded. One method that can accomplish this is desulfurization with reducing gases, which can be derived from the mild gasification co-product gases. The desulfurization can be performed either on the char particles exiting the mild gasifier or on formed briquettes made by a binderless technique, wherein hot (1100°F) char is blended with additional caking coal to provided an *in-situ* binder.

The sulfur content of char from mild gasification is largely dependent on the sulfur content of the parent coal. In the DOE-sponsored study, the parent coal, a conventionally washed Illinois coal, contained 3 to 5 wt% sulfur. The char sulfur content from a series of temperature-dependency tests ranged from 2.1 wt% to 2.8 wt%, which was 27% to 46% lower than that of the coal. It is estimated that desulfurization of the char by an additional 50% or more would be required to yield an attractive product for form coke production.

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. Although pure hydrogen is much more effective than pure methane, H_2/CH_4 mixtures are also more effective than pure methane, and the presence of methane inhibits carbon hydrogasification, thus allowing desulfurization to proceed with reduced carbon losses, compared to treatment with pure hydrogen. Also, the use of a process-derived gas mixture avoids the separation costs associated with the use of pure H_2. The product gas from mild gasification, on an inert-free basis, contains 20 to 28 vol% CH_4 and 28 to 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.

The ultimate goal of the 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 concept, develop a preliminary process flow scheme, and make recommendations for integration of the desulfurization step into an ongoing mild gasification development program.

In the first year, granular char from the IGT mild gasification PRU is being treated with mixtures of H_2 and CH_4 at the following ranges of conditions:

- Temperatures of 1100° to 1550°F
- Pressures of 50 to 100 psig
- Residence times of 30 to 60 minutes
- Reducing gas H_2 content of 50 to 90 vol%

The batch experiments are being performed in a nominal 2-inch-ID stainless-steel, batch, fluidized-bed reactor. The char being desulfurized was produced by the IGT mild gasification process research unit (PRU) in a previous DOE/METC-sponsored technology development program. The parent coal was Illinois No. 6 coal from Peabody Coal Company's Randolph preparation plant, and the char contains 4.58 wt% sulfur. The effects of temperature, pressure, residence time, gas velocity, and gas composition on sulfur removal and carbon gasification are being determined.

In the second year, the study will be extended to include form coke briquettes made from the same mild gasification char. The briquettes will be made by a binderless technique, wherein hot (1100°F) char is blended with additional parent coal to provided an *in-situ* binder. A parametric study similar to that conducted in the first year will be performed on the "green" (uncalcined) briquettes, followed by calcination of the briquettes at 1800°F, which is required to cure the binder and develop optimal properties. In addition to the effects of process parameters on sulfur removal and carbon gasification, their effects on the strength, density, tumbler-test stability, and reactivity of the resultant form coke, which are important to their use in blast furnaces, will be investigated. The data obtained from both years of the program will be used to design a desulfurization process to be integrated with the IGT mild gasification process.

During the second quarter, we have conducted eight fluidized-bed tests on the mild gasification char prepared during the previous quarter. These tests were performed at 1275° to 1575°F, 50 to 100 psig, and with residence times of 30 to 60 minutes. The superficial gas velocity in the bed was 0.10 to 0.15 ft/s, and the gas mixtures used were 10 vol% or 25 vol% CH_4 in H_2.

The analyzed data show that sulfur conversion was greater than carbon conversion in six of the eight tests, with the maximum sulfur conversion being 13.3% at 1300°F and 100 psig in 10%CH_4 for 30 minutes. The bed gas velocity was 0.10 ft/s in that test. Overall, sulfur conversion ranged from 2.3% to 13.3% and carbon conversions ranged from 1.2% to 4.0%. Char gross heating value was likewise reduced by 0.2% to 4.1%. However, the sulfur content of the desulfurized char did not drop below 4.26 wt% sulfur, which is well above the value required for satisfactory upgrading.

Analyses of gas samples showed H_2S levels in the exit gas ranged from 155 to 404 ppmv, with levels declining as each test progressed. It was also found that N_2 in the system lines and vessels was not adequately replaced by H_2 and CH_4 until well after 15 minutes of operation. This definitely reduced the

effective residence time of the char in contact with reducing gases. This problem is being eliminated by installation of a reactor bypass system to allow complete purging of the gas delivery lines prior to introducing reactant gas to he reactor.

Future tests will attempt to improve desulfurization by increasing residence time and decreasing H_2S gas phase concentration in the reactor by increasing superficial gas velocity.

OBJECTIVES

The ultimate goal of this project is to develop a method for desulfurization of mild gasification char, using process-derived gases. The overall objectives of the 24-month program are to conduct laboratory studies that assess the technical viability of the process concept, 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 that emphasizes simple reactor and process design, low-severity processing conditions, and the use of advanced technical knowledge to bring a slate of value-added co-products (char, fuel gas, and oils/tars) to the marketplace in the next five years. The U.S. DOE has been supporting the development of this technology since 1987, and a project team consisting of Peabody Holding Company, Bechtel National, and IGT has completed a literature and market survey and technology development program including the design, construction and operation of a 100-lb/h PRU and the design of a 24-ton/day process development unit (PDU).[1,2,3,4] The mild gasification reactor consists of a coaxial fluidized-bed/entrained-bed vessel which can process all types of coals. IGT has completed 47 mild gasification tests on four coals in the PRU, at temperatures ranging from 1034° to 1390°F.

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[1] Knight, R.A., J. Gissy, M. Onischak, S.P. Babu, J.M. Wootten, and R.G. Duthie, "Development of An Advanced, Continuous Mild Gasification Process for the Production of Co-Products", Task 2 Topical Report to DOE/METC, Contract No. DE-AC21-87MC24266, (July 1990).

[2] Knight, R.A., J. Gissy, M. Onischak, R.H. Carty, S.P. Babu, J.M. Wootten, and R.G. Duthie, "Development of An Advanced, Continuous Mild Gasification Process for the Production of Co-Products", Task 4 Topical Report to DOE/METC, Contract No. DE-AC21-87MC24266, (December 1990).

[3] Wootten, J.M., M. Nawaz, R.G. Duthie, R.A. Knight, M. Onischak, S.P. Babu, and W.G. Bair, "Development of An Advanced, Continuous Mild Gasification Process for the Production of Co-Products", Task 1 Topical Report to DOE/METC, Contract No. DE-AC21-87MC24266, (August 1988).

[4] Carty, R.H., M. Onischak, S.P. Babu, R.A. Knight, J.M. Wootten, and R.G. Duthie, "Development of An Advanced, Continuous Mild Gasification Process for the Production of Co-Products", Task 3 Topical Report to DOE/METC, Contract No. DE-AC21-87MC24266, (December 1990).

Figure 1 shows the variation of char sulfur content with mild gasification temperature from a series of PRU tests using Illinois No. 6 coal from Peabody's Randolph preparation plant. The parent coal, containing 3 to 5 wt% sulfur, is typical of a conventionally washed Illinois coal. As shown, the char sulfur content, ranging from 2.1 wt% to 2.8 wt%, is 27% to 46% lower than that of the coal. Expressed on a Btu basis, this represents a reduction of 20% to 38%.

For form coke made from mild gasification char, the guidelines on sulfur content are not strictly defined. In the blast furnace, coke acts as both a fuel and as a reductant for iron ore. Limestone is added to the furnace charge to bind the sulfur, and the amount of limestone added directly affects the economics of the process by limiting the steel output, consuming sensible heat, and adding material cost for the limestone. For these reasons, in this project, the target sulfur limit of 1.0 wt% for the form coke has been selected.

In the 1970's, research on coal and char desulfurization increased along with synfuels research in general. U.S. Steel Corporation, with ERDA support, developed the Clean Coke process, in which desulfurization of the char with recycled product gas played an important role.[5] The recycle gas used for fluidization in the PDU was 71% CH_4, 13% C_2H_6, 10% CO, 2% H_2, and 4% higher hydrocarbons. At 1400°F, char was reduced in sulfur content from 1.7 wt% to 0.3 wt% in 180 min residence time, which is an 82% reduction. The H_2S concentration in the recycle gas was identified as a critical factor in achieving effective desulfurization, as shown by the finding that increasing the volumetric H_2S concentration in the recycle gas from 50 ppm to 500 ppm increased the char sulfur content from 0.21 wt% to 0.71 wt%. An associated study of char-sulfur chemistry[6] found that, with an Illinois No. 6 hydrogasification char, desulfurization with reducing gases occurred in two steps: an initial rapid-rate step associated with reduction of FeS_2 to FeS, and a slower secondary step where FeS is reduced to elemental iron. The study 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 methane inhibited carbon gasification at longer residence times while allowing desulfurization to continue.

The same study also found that, for a given char, the relationship between sulfur removal and carbon loss through gasification in the first 15-30 minutes follows a monotonic curve, regardless of the temperature, pressure, or gas atmosphere used. Figure 2 shows this relationship for an Illinois No. 6 char prepared at 1112°F. 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

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[5] Boodman, N.S., T. F. Johnson, and K.C. Krupinski, "Fluid-Bed Carbonization/Desulfurization of Illinois Coal by the Clean Coke Process: PDU Studies", ACS Div. of Fuel Chem. Prepr. 22:2, 28-44 (1977).

[6] Kor, G.J.W., "Desulfurization and Sulfidation of Coal and Char", ACS Div. of Fuel Chem. Prepr. 22:2, 1-27 (1977)

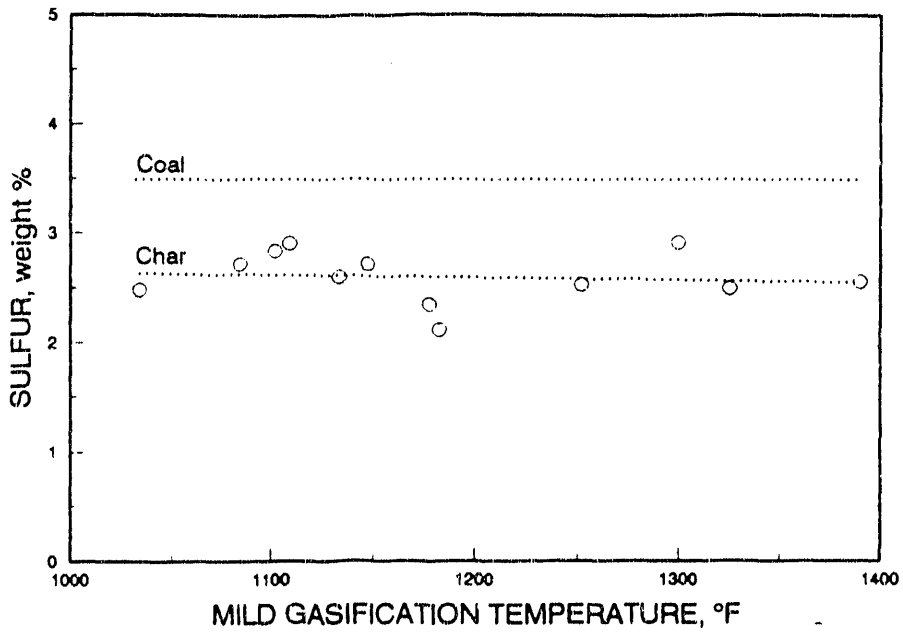

Figure 1. SULFUR CONTENT OF MILD GASIFICATION CHAR AS A FUNCTION OF TEMPERATURE

accompanying 10% to 13% carbon conversion.

In similar work, IGT studied the hydrodesulfurization of four bituminous coals at 1300° to 1500°F for 30 min in hydrogen.[7] A reduction of 74% in the lb SO_2/MMBtu was achieved with Illinois No. 6 coal. This study emphasized the importance of maintaining a low H_2S concentration in the treatment gas, suggesting the use of a solid sulfur sorbent such as calcium oxide. The investigators also concluded that a mild pre-oxidation of the coal substantially increases subsequent sulfur removal by hydrodesulfurization. However, a more recent CRSC-funded study[8] found that this interaction was dependent on the coal tested and the type of physical cleaning to which the coal had been subjected.

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

[8] Stephenson, M.D., A.D. Williams, M. Rostam-Abadi, and C.W. Kruse, "The Effect of Mild Oxidation on the Thermal Desulfurization and Hydrodesulfurization of two Illinois Bituminous Coals in a Fluidized-Bed Reactor", ACS Div. of Fuel Chem. Prepr. 33:4, 960-967 (1988)

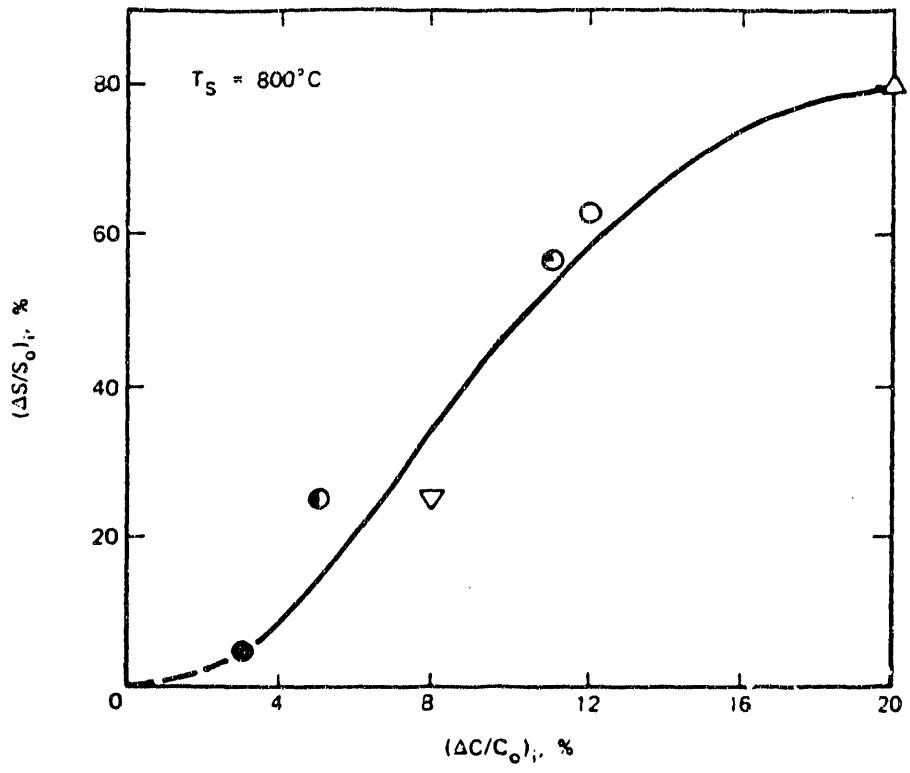

Figure 2. GENERAL RELATIONSHIP BETWEEN INITIAL LOSS OF SULFUR AND CARBON FOR ILLINOIS NO. 6 CHAR PREPARED AT 1112°F

Research on char hydrodesulfurization was also done by Garrett Research and Development (later Occidental Petroleum). A 1976 paper[9] discusses the effectiveness of acid-washing of char to remove Ca and Fe compounds which appear to contribute to the severity of H_2S inhibition. The hydrogen requirement with an acid-washed char was 12% of that required for an untreated char.

This study is evaluating the technical feasibility of desulfurizing mild gasification char and/or form coke briquettes with process-derived reducing gases. The unique aspects of this research are: the application of this technique to mild gasification char and/or briquettes made therefrom, and the integration of the technique with form coking. In the IGT fluidized-bed mild gasification 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 a systematic control over the relationship of the incoming coal feed rate to the volumetric char inventory in the fluidized bed.

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[9] Robinson, L., "Hydrodesulphurization of Char", *Fuel* 55:3, 193-201 (1976)

The char thus differs in physical and chemical properties from both coal and from gasification or hydrogasification chars that have previously been studied. In form coke production, the hot (1100°F) char would be contacted with fresh coal which would supply an *in-situ* binder for briquetting. The resulting "green" briquettes would then be calcined in a shaft furnace at about 1800°F. The relative feasibility and impact on overall process efficiency of desulfurization before and 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 methane inhibiting the carbon gasification during desulfurization, the use of a process-derived gas mixture would be more economical than using pure hydrogen. If necessary, the hydrogen content of the desulfurizing gas can be adjusted via the shift reaction or partial steam reforming of the methane and other hydrocarbon gases. The effectiveness of varying mixtures of H_2 and CH_4 for desulfurization are being determined in the program.

The information thus obtained will be unique in its direct applicability to mild gasification, and thus will be of immediate value to further process development and the expanded utilization of high-sulfur Illinois coals.

EXPERIMENTAL PROCEDURES

Existing samples of mild gasification char from PRU tests conducted under a DOE/METC contract are being used instead of char made from IBCSP coals, because the former has been produced by the 100-lb/h PRU, and the cost of operating the PRU to make a char sample for this program would be prohibitive. We feel that a char produced in a laboratory unit would not necessarily be as truly representative of mild gasification char, and therefore the use of this material is justified. The coal used was an Illinois No. 6 fines (-1/4-inch) circuit from the Peabody Coal Company's Randolph preparation plant. Prior to feeding into the mild gasification reactor, the coal was blended with diluent char in a 1:1 weight ratio. Chars will be chosen for testing that contain a minimum of diluent coke breeze in the sample.

Task 1. Sample Preparation and Characterization

For the first-year experiments on granular char, samples will be crushed and screened to -80+100 mesh, divided by riffling into representative portions, and characterized in IGT laboratories according to the following protocol:

- proximate and ultimate analyses
- heating value
- pore volume and surface area
- sieve analysis

Task 2. Equipment Preparation and Shakedown

The desulfurization experiments will be conducted in a nominal 2-inch batch reactor, which will be employed in a fluidized-bed mode for the first-year granular char experiments and in a fixed-bed mode for the second-year briquette desulfurization experiments. The schematic diagram of the reactor system is shown in Figure 3. Existing furnaces, controls, instruments, and gas supply equipment will be used. A reactor vessel will be fabricated from Sch 80 316 stainless steel to safely operate up to 1800°F and 100 psig. The reactor system will also be modified to allow operation up to 100 psig by the addition of high-pressure mass flow controllers and a high-capacity sintered metal filter to retain fines, replacing the existing low-pressure cyclone.

In the first year, this task will cover the reactor fabrication, system cleanup, mechanical and electronic testing of components, and shakedown testing of the system.

Task 3. Desulfurization

This Task is divided into two Subtasks as shown below. In the first year, the experimental work will be performed with granular char, and in the second year, with briquettes made from granular char and coal according to the procedure described in Task 1.

Subtask 3.1. Parametric Experiments

Char desulfurization experiments will be performed to determine the effects of temperature, pressure, residence time, and gas composition on the char sulfur content. Table 1 shows the specific values of these parameters to be tested. Not all of the combinations in the test matrix will be used. The combinations of parameters will be selected, based on the ongoing data interpretation, to maximize char desulfurization. A total of 24 experiments will be performed in each year of the program.

Table 1. EXPERIMENTAL PARAMETERS TO BE TESTED

Temperature, °F	1100, 1300, 1550
Pressure, psig	50, 100
Residence time, min	30, 60
Gas composition, vol% CH_4[a]	10, 25
Superficial gas velocity, ft/s[b]	0.10, 0.15

[a] Balance H_2; CH_4 concentrations are based on gas stability with respect to solid carbon deposition

[b] For first-year fluidized-bed experiments only

In a typical experiment, 50 to 100 grams of char or green briquettes will be charged to the reactor, which will then be sealed. The reactor and associated

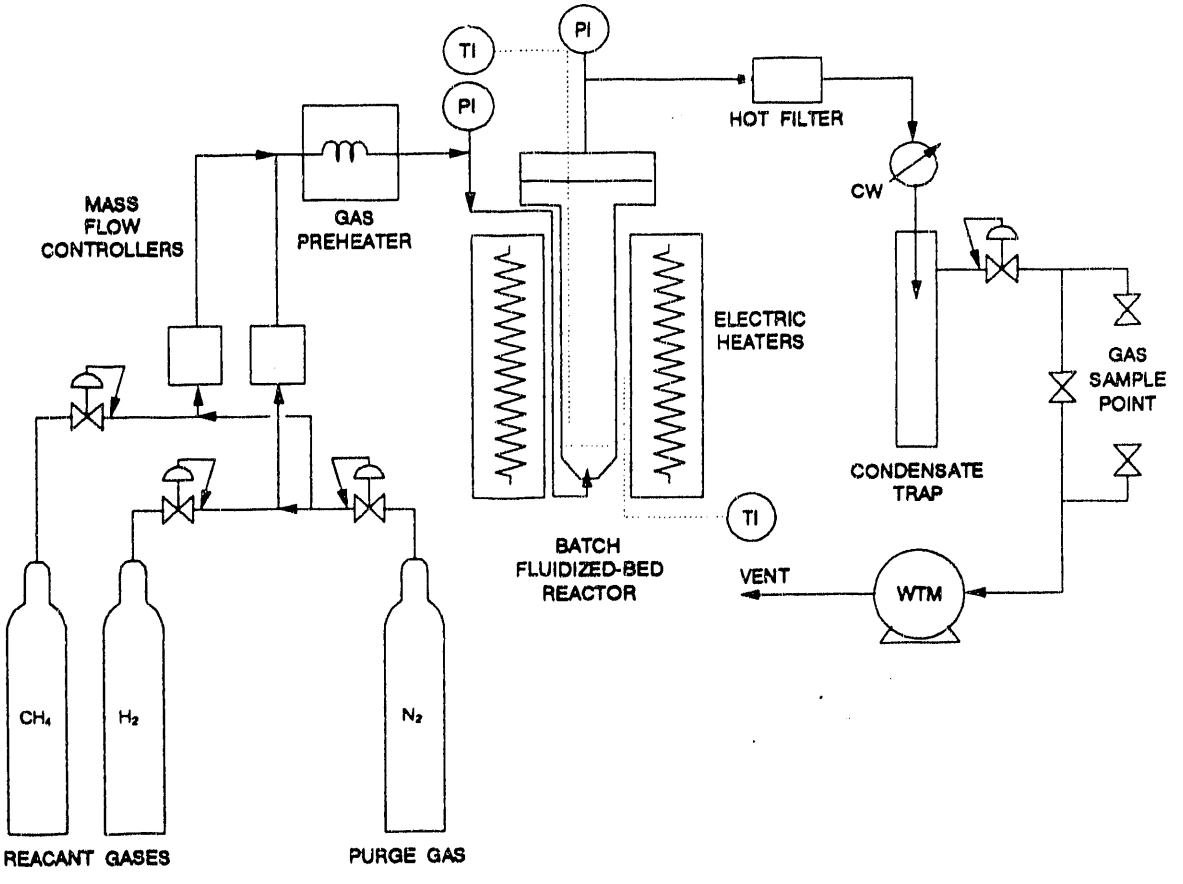

Figure 3. SCHEMATIC OF EXPERIMENTAL DESULFURIZATION REACTOR

tubing will be leak-tested and purged with inert gas. The reactor will then be heated to the desired desulfurization temperature while continuing the flow of inert gas. When the desired temperature is attained, the gas flow will be switched to the desired premixed gas (10 vol% to 25 vol% CH_4 in H_2). Gas samples will be retrieved at 15-minute intervals until the end of the desired residence time. Inert gas flow will then replace the reactive gas mixture, and the reactor will be rapidly cooled. When cooled to room temperature, the reactor will be opened to retrieve the desulfurized sample for analysis.

For first-year tests, the gas flow to the fluidized-bed reactor will be based on the required minimum fluidization velocity (u_{mf}). The parameters selected for gas velocity are slightly below and slightly above three times the u_{mf} for the largest (80-mesh) char particles. The required gas flow to attain the desired gas velocity will be calculated for each experiments based on the

density and viscosity of the gas mixture used at the conditions of the experiment.

Subtask 3.2. Product Characterization and Evaluation

The desulfurized char or briquettes from each experiment will be evaluated by proximate and ultimate analyses. The amount of sulfur removal will be calculated based on the weight% of sulfur in char (from first-year experiments) or form coke (from second-year experiments). The heat content of the product chars will be calculated according to the following formula from the IGT Databook:[10]

$$Q_v \text{ (Btu/lb, dry basis)} = 146.58C + 568.78H + 29.4S - 6.58A - 51.53(O+N)$$

where C, H, S, A, and (O+N) are the respective weight % of carbon, hydrogen, total sulfur, ash and oxygen + nitrogen by difference.

Selected char samples (approx 1 in 8) will also be analyzed for sulfur by type and measured calorific value, in order to study effects on the distribution of inorganic or organic sulfur removed.

Gas samples from the desulfurization unit will be analyzed for H_2S content by the GC/FPD method.

Task 4. Data Analysis and Interpretation

The experimental data will be analyzed and interpreted to determine the effects of the selected parameters (temperature, pressure, residence time, gas velocity, and gas composition) on the following properties of the granular mild gasification char:

- Sulfur content, wt% of dry char
- Percent S or C conversion, $100\ (1 - \frac{m_r(\%C_r/100)}{m_f(\%C_f/100)})$

  where m_f = feed char mass, m_r = residue mass, $\%C_f$ = feed char C or S content, and $\%C_r$ = residue C or S content.
- Higher heating value of char, Btu/lb (dry)

If the gas-phase H_2S concentration appears to be significantly affecting the desulfurization rates under some conditions, some modification of the test plan may be made in order to further study this effect. In this case, gas flow rates different from those dictated by the test plan parameters may be used.

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[10] *Coal Conversion Systems Technical Data Book*, U.S. DOE Document HCP/T2286-01, Section IA.30.5 (Feb 1978), pp. 1-2

RESULTS AND DISCUSSION

Task 1. Sample Preparation and Characterization

A char sample from the IGT mild gasification PRU was obtained, prepared, and analyzed in the first quarter. The analysis of this material is shown in Table 2 below.

Table 2. ANALYSIS OF MILD-GASIFICATION CHAR FROM PRU TEST MG-8

Proximate Analysis	wt %
Moisture	1.46
Volatile matter	11.64
Ash	34.81
Fixed Carbon	52.09
	100.00
Ultimate Analysis	
Carbon	54.63
Hydrogen	0.94
Sulfur	4.58
Nitrogen	1.11
Oxygen (by diff.)	3.41
Ash	35.33
	100.00
Bulk density, g/cc	0.739
Surface area, m^2/g	17.68

Task 2. Equipment Preparation and Shakedown

The schematic diagram of the desulfurization reactor system constructed last quarter is shown in Figure 3. Shakedown of this unit was begun and completed during this quarter.

Task 3. Desulfurization

Eight char desulfurization tests on MG-8 char were conducted during the quarter. The conditions of these tests are shown in Table 3.

Task 4. Data Analysis and Interpretation

The product char analyses and results of the char desulfurization are summarized in Table 4. Carbon, sulfur, and HHV conversions were calculated on the basis of a forced ash balance. The most obvious change in all cases was the extensive conversion of volatile matter and oxygen, with much lesser

Table 3. DESULFURIZATION CONDITIONS FOR TESTS 1220 TO 0107

Test	1220	1226	1227	1230	0102	0103	0106	0107
Temperature, °F	1290	1320	1575	1275	1300	1310	1545	1545
Pressure, psig	50	50	50	100	100	52	51	100
Residence time, min	30	30	30	30	30	60	60	60
Bed velocity, ft/s	0.15	0.15	0.10	0.10	0.10	0.10	0.15	0.10
CH_4 conc, vol%	25	10	10	25	10	25	10	10
Total gas flow, scfh	11.2	11.2	6.6	13.3	13.3	7.5	9.8	11.6

conversions of C, H, N, and S. In several cases, the char oxygen content was determined by difference to be negative, which indicates that the char contains essentially zero oxygen.

Sulfur conversion ranged from 2.25% to 13.25%, and calculated carbon conversion ranged from 1.24% to 4.02%. Compared to the parent char sulfur content of 4.58 wt%, the product chars contained 4.26% to 4.90% sulfur. This represents a maximum reduction in sulfur content of only 7.0%, far below the 78% reduction required to yield a char with 1 wt% sulfur. In these tests, there is no clear trend in sulfur or carbon conversion, or the ratio of one to the other, with respect to temperature, pressure, residence time, gas velocity, or CH_4 concentration.

The heating values of the treated chars showed only slight reductions, reflecting the fact that the primary hydrogasification reaction was deoxygenation, which has minimal effect on heating value.

Average exit gas H_2S concentrations are also shown in Table 4. The values shown are mean values over the entire test. Samples were taken at 15-minutes intervals. Again, no clear trends are indicated as a function of process parameters. In general, the H_2S concentration tended to peak sometime in the first 30 minutes and gradually decline thereafter.

Gas compositions from the tests indicated that nitrogen was not being displaced from the gas delivery system quickly enough at the selected flow rates, and consequently the exposure time of the char to reducing gases was about 15 minutes less than required. A reactor bypass section will be installed to rectify this problem.

Because char desulfurization in this first test series was far below the required level for upgrading char, the original test matrix will be modified. Future tests will be conducted at extended residence times of 90 to 180 minutes at 1550°-1600°F to attain more extensive desulfurization of the char.

Table 4. ANALYZED DATA FROM TESTS 1220 TO 0107

Test	1220	1226	1227	1230	0102	0103	0106	0107
CHAR ANALYSIS								
PROXIMATE, wt% as rec'd								
Moisture	0.25	0.35	0.44	0.32	0.15	0.23	0.32	0.30
Volatile Matter	6.32	4.59	2.42	6.23	4.67	5.21	2.42	2.26
Ash	36.81	37.34	38.23	36.91	37.82	36.48	38.55	38.16
ULTIMATE, WT% dry								
Ash	36.90	37.47	38.40	37.03	37.88	36.56	38.67	38.27
Carbon	56.78	56.81	57.08	56.55	56.80	57.09	57.39	57.51
Hydrogen	0.81	0.80	0.46	0.85	0.80	0.78	0.54	0.51
Nitrogen	1.07	1.05	0.88	1.00	0.86	0.88	0.82	0.79
Sulfur	4.53	4.42	4.81	4.35	4.26	4.56	4.90	4.62
Oxygen (by diff)	-0.09	-0.54	-1.63	0.22	-0.60	0.13	-2.32	-1.70
HHV, Btu/lb	8623	8639	8556	8594	8643	8653	8686	8651
S conversion, %	5.30	9.21	3.37	9.38	13.25	3.79	2.25	6.88
C conversion, %	0.49	1.85	3.87	1.24	3.03	-0.99	4.02	2.82
HHV conversion, %	-0.55	0.80	4.14	0.15	1.83	-1.83	3.36	2.74
H_2S in gas, ppmv	241	381	248	310	390	240	221	366

CONCLUSIONS AND RECOMMENDATIONS

Shakedown of the batch fluidized-bed desulfurization reactor was completed, and eight desulfurization tests were performed on mild gasification char. Under the conditions originally chosen for the project test matrix, sulfur conversions were, on average, higher than carbon conversions. However, the char sulfur reduction was very low, the maximum being 7% reduction in S content at 13% S conversion. The target char sulfur content of 1% requires a sulfur content reduction of 78%.

Testing will continue on mild gasification char, but residence times will be extended to attain higher levels of desulfurization.

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 Center for Research on Sulfur in Coal, and by the U.S. Department of Energy (Grant Number DE-FG22-91PC91334). However, any opinions, findings, conclusions, or recommendations expressed herein are those of the author(s) and do not necessarily reflect the views of IDENR, CRSC, and the DOE.

PROJECT MANAGEMENT REPORT
December 1, 1991 - February 29, 1992

Project Title:	**Investigation of a Technique for Sulfur Reduction of Mild Gasification Char**
Principal Investigator:	Mr. Richard Knight, Institute of Gas Technology
Project Monitor:	Mr. Frank Honea, CRSC

COMMENTS

No change to the budget or management were implemented this quarter.

This project is funded by the U. S. Department of Energy (METC) and by the Illinois Department of Energy and Natural Resources as part of their cost-shared program.

EXPENDITURES

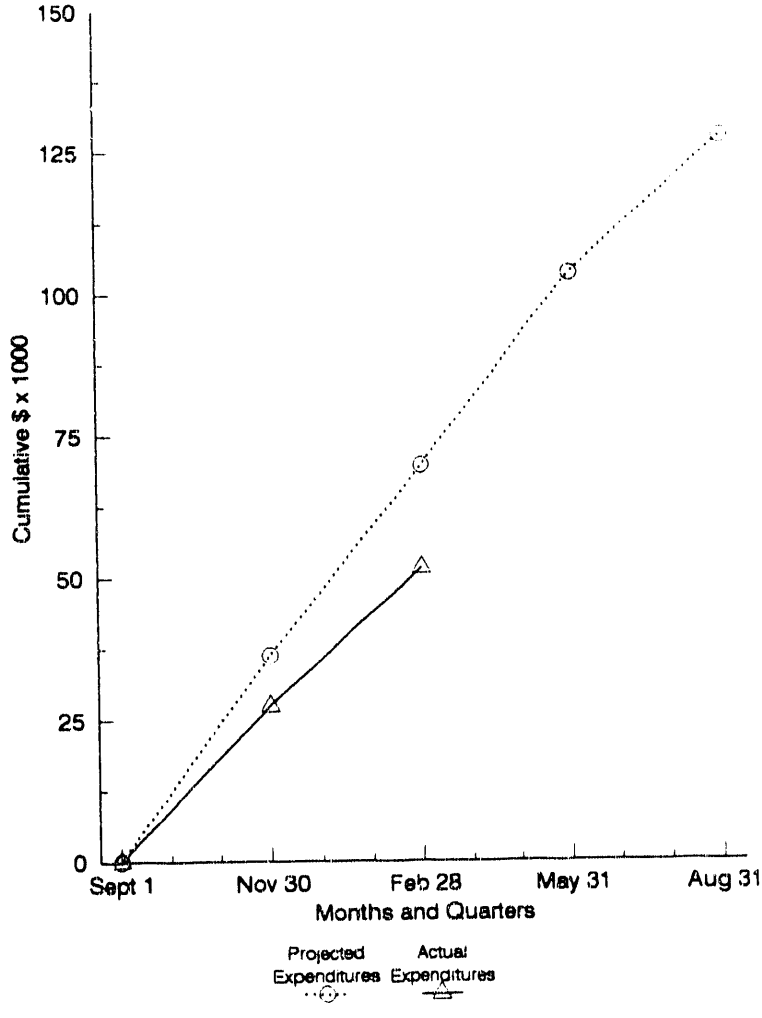
Investigation of a Sulfur Reduction Technique for Mild Gasification Char
Projected and Estimated Actual Expenditures by Quarter

Quarter*	Types of Cost	Direct Labor	Materials and Supplies	Travel	Major Equipment	Other Direct Costs	Indirect Costs	Total
Sept 1, 1991 to Nov 30, 1991	Projected	11,500	995		2,294		21,473	36,262
	Estimated Actual	8,902	1,550		450		16,508	27,410
Sept 1, 1991 to Feb 28, 1992	Projected	23,000	1,995		2,294		42,466	69,755
	Estimated Actual	16,953	2,892		458		31,341	51,644
Sept 1, 1991 to May 31, 1992	Projected	34,500	2,995	258	2,294		63,513	103,560
	Estimated Actual							
Sept 1, 1991 to Aug 31, 1992	Projected	43,065	2,995	258	2,294		78,992	127,604
	Estimated Actual							

* Cumulative by Quarter

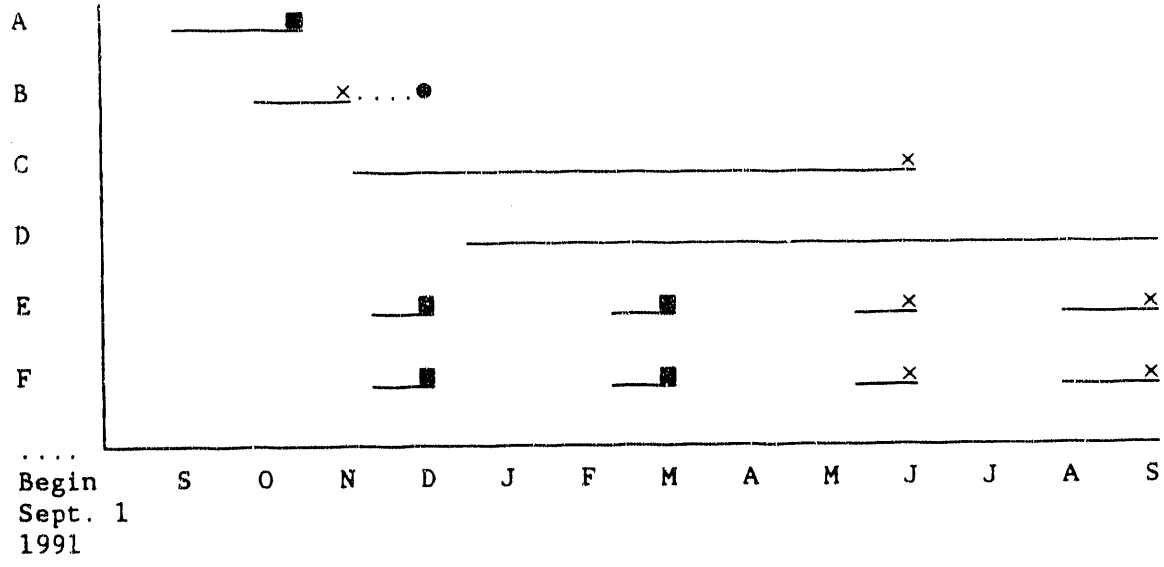
COSTS BY QUARTER

Investigation of a Technique for Sulfur Reduction of Mild Gasification Char



Total CRSC Award = $127,604

SCHEDULE OF PROJECT MILESTONES (First Year: 9/91-8/92)



Hypothetical Milestones:

A. Granular char sample prepared and characterized (Task 1)

B. Equipment fabrication and shakedown for first-year tests completed (Task 2)

C. Granular char desulfurization experiments completed (Task 3)

D. Experimental data analyzed and interpreted (Task 4)

E. Quarterly and annual technical reports prepared and submitted

F. Quarterly project management reports prepared and submitted

END

DATE
FILMED
9/01/92