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COLLEGE OF ENGINEERING
BUREAU OF ENGINEERING RESEARCH

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FEASIBILITY STUDIES OF IN-SITU COAL GASIFICATION
IN THE WARRIOR COAL FIELD

by

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Quarterly Report to
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TABLE OF CONTENTS

	<u>Page</u>
I. Forward Combustion in Char	1
II. Thermal Conductivity and Thermal Diffusivity of Coke	6
III. Reactivity of Coke Made from Alabama Coals	14
A. Reaction Kinetics	14
B. Reaction Rate Constants for the Coke from Alabama Coals	21
C. Conclusions	24

I. FORWARD COMBUSTION IN CHAR

Beginning on the tenth of July, a series of six shakedown burns were made in the scaled down tube furnace combustor described in the previous quarterly report. In each burn the combustor was packed with 50 grams of blast furnace coke obtained from a local concern. The coke charge consisted of sieved particles of a fairly uniform 0.25 inch average diameter; this led to a fuel bed thickness of approximately 8 inches. Temperature readings were taken using type K thermocouples located externally along the combustion tube and internally within the coke charge itself. The combustor assembly was mounted within a tube furnace. Input lines for air and water were installed so that the air (or air/steam) mixture flowed in from the bottom of the fuel charge and upward out of the combustor, where product gas was analyzed continuously by gas chromatography.

With this setup the procedure was as follows. The combustor was sealed (no flow). The furnace was activated and allowed to reach a pseudo-equilibrium temperature where the burn would start. This usually took around two hours. Once the fuel charge was brought to the desired temperature, air flow at various constant rates was applied.

As hoped, the coke charge was observed to burn in a forward combustion mode. This could be deduced from readings of both internal and external thermocouples. Unpacking and the upward migration of the external thermocouple readings revealed that the coke bed did not "migrate" downward as had been a problem with earlier combustors. Rather, the ash particles formed a rigid porous mass which supported the unburned coke above.

During the course of a burn, oxygen concentration in the off gas would rapidly fall to a range of 0.1 - 0.3%, depending on the flows and temperatures.

Once it reached these levels, it remained virtually constant for the duration of the burn. As the burn ended, O_2 percentage showed a steep step function back to atmospheric levels. This leads one to the deduction that the oxidation zone is very thin, possibly as small as two or three particle diameters. The geometry of the reduction zone was not easily deduced from these runs, although in many burns it appeared to exist for the most part in a length less than the fuel bed.

It was desired to use a coke from a known Alabama coal. This was necessary since blast furnace coke is much more reactive than chars produced from local coals. With this in mind a pyrolysis chamber was built in which coal from the Blue Creek seam was pyrolyzed. The pyrolysis was done in a nitrogen atmosphere in a furnace heated to $800^{\circ}C$ for a period of ten hours or more. After several loads of char were produced, it was crushed and sieved to yield 0.25 inch particle size. This coke was used in all later burns.

For the remaining experimental burns, a water cooled sampling system was constructed to allow a gas sample to be pulled off the top of the fuel bed without further reaction. The coke bed was placed above the center line of the furnace also. This was done to allow sufficient preheating of air, and to locate the coke in the most isothermal region of the furnace.

Runs were continued systematically throughout August and September at three temperatures ($1200^{\circ}F$, $1400^{\circ}F$, $1800^{\circ}F$) and various flows. As in previous burns, the oxidation zone was seen to be very thin, with reduction zone length varying with applied flows and temperatures. We intend to use temperature data along with reactivity data produced by others in this project to compare experimental results with those produced by reaction calculations. Samples of gas quality versus time for various burn conditions are included.

II. THERMAL CONDUCTIVITY AND THERMAL DIFFUSIVITY OF COKE

Two new thermocouple assemblies with 446 stainless steel sheaths were ordered. Preliminary tests showed that they were capable of withstanding high temperatures and corrosive sulfur atmosphere.

A proximate analysis was performed earlier on twenty samples of coal from various coal mining areas in the north Alabama region. The tests were done according to the ASTM standard method of laboratory sampling and analysis of coal and coke (D271-48). The coals were tabulated according to their volatile matter, ash content, moisture and fixed carbon respectively in ascending order. Coals with similar properties were grouped together and a representative was chosen. After much iteration, selection and elimination, eleven samples of coal were chosen to be the representative. They are listed as follows: Utley (1-B), Pratt (2), Utley (3), Thomas (6), Gillespy (8), Blue Creek (9-A), Blue Creek (9-B), Mary Lee (10), Mary Lee (11), Blue Creek (13), and Jefferson (15).

In order to perform tests to determine thermal conductivity and thermal diffusivity of coke, coke was prepared from coal through the following arrangement. There was a slight change in procedure due to the use of the new thermocouple assemblies. A $2\frac{1}{2}$ mm diameter hole was drilled centrally through a lump piece of coal sample and then two other $1\frac{1}{2}$ mm holes were drilled 6.35 mm deep parallel to the central hole; one as close as possible, and the second one 15 mm away. The heating wire was put through the central hole and the thermocouples into the other two. The two ends of the heating wire were coiled around at the edge of the container. The top and bottom lids were bolted securely to the lip of the container, which was sealed with an asbestos gasket. An exhaust tubing was screwed onto the top lid for the escape of volatile

matter. Thermocouples were connected to the data logger (Figure 1).

Fig. 1. Schematic of Coke Making Process

A vacuum pump was used to evacuate the air and water from the coal sample. After the maximum temperature of 800°C had been reached, the coal sample was left in the furnace for two more hours to ensure complete pyrolysis. After the furnace had cooled down, the exhaust tubing was removed and one end of the heating wire was retrieved through the hole. On removing the bottom lid, the other end of the heating wire was exposed. The two ends of the heating wire were connected to the DC power supply.

The hot-wire method was carried out on sample 11 Mary Lee coal. Thermal conductivity and thermal diffusivity of coke are important properties because they determine the rate and extent of pyrolysis away from a burning zone and

also the rate of propagation of the flame front in reverse combustion. A review of the literature on methods of measuring coke thermal conductivities and diffusivities led to the conclusion that the method developed by E. F. M. VanderHeld, et al., was best suited for our measurement.

With the hot-wire method, the thermal conductivity and thermal diffusivity can be determined from the same experiment, and then by measuring the density of coke, the specific heat of the coke can be calculated.

The thermal conductivity can be evaluated from the slope of a plot of $(\theta_r - \theta_o)$ versus nt as shown in equation 1. This plot is made from the thermocouple nearest the heater.

$$(\theta_r - \theta_o) = \frac{q}{4\pi k} \left(\ln t + \ln \frac{4}{r} - 0.578 \right) \quad 1$$

θ_r = Temperature at a point at radial distance r from the heater wire after time t

θ_o = Initial temperature

q = Heat supplied to the coke sample through the heater wire

k = Thermal conductivity

α = Thermal diffusivity

The thermal diffusivity can be evaluated from the following relationship:

$$(\theta_r - \theta_o) = \frac{q}{2\pi k} I(\beta) \quad 2$$

$$\beta = r / 2 (\alpha t)^{1/2} \quad 3$$

From equation 2, $I(\beta) = \frac{2\pi k}{q} (\theta_r - \theta_o)$, we get $\frac{2\pi k}{q}$ from the slope of equation 1. This calculation is made using the thermocouple furthest from the heater. Then, from a table of $I(\beta)$ vs. β and equation 3, the value of α can be determined.

Since the coal becomes electrically conducting at high temperatures, all wires are insulated from the coal with a silical sleeving. The coke was placed in an electric furnace and heated in a slow stream of nitrogen to the temperature at which a test was to be made. Before each test, the heating rate was reduced to less than 0.5 C/min and the rate of rise of temperature of the lump was measured over a period of ten minutes. The current for the heater wire was supplied by the DC power supply. Temperature/time readings of the thermocouples were recorded by the data logger (Figure 2).

The thermal conductivity was calculated from the slope of the plot of temperature against $\ln t$ which is equal to $q/4\pi k$. A plot of thermal conductivity and thermal diffusivity of Mary Lee coke vs. temperature is attached. An overall conclusion will be drawn upon completion of tests on all samples.

Fig. 2. Schematic for Hot Wire Method

MARY LEE COKE		
Temperature (°C)	Thermal Conductivity (Watt/m°C)	Thermal Diffusivity (cm ² /sec)
80.0	0.666	
105.1	0.28	0.00234
169.8	0.185	0.00277
263.7	0.635	0.0032
340.0	0.608	0.0036
365.0	0.652	0.0030
401.4	0.56	0.00315
474.8	0.9098	0.00398
497.2	0.578	0.0037
549.2	0.666	0.00373
574.4	0.997	0.00472
597.4	0.901	0.00400
663.4	0.709	0.0039
699.2	0.643	0.00435
739.6	0.829	0.00356
793.1	1.5313	0.00447

III. REACTIVITY OF COKE MADE FROM ALABAMA COALS

The last quarterly report (June 1979) contains a description of the apparatus and procedures used to determine reactivities of coke made from Alabama coals. Also included was the reactivity of the cokes toward air at 500°C. The current report concludes the work done on coke reactivities.

The reactivities of eight samples with CO₂ at 900°C are reported in Table 1, along with the reactivities with air reported last time. The data on CO₂ reactivities are plotted in Figure 3 against fixed-carbon content of the parent coal. This figure shows that reactivity toward CO₂ does not correlate well with fixed carbon content, whereas the reactivity toward air showed a definite decrease with increased fixed carbon content.

A. Reaction Kinetics

The amount of sample used in this study was small (i.e., about 4 mg to 5 mg for each Alabama coke investigated). When the sample (-40 + 100 U.S. mesh) was placed in the sample pan, the depth of the coke bed was small (around 1 mm). It was proved in the study performed by Jenkins, et al¹ that for samples under this condition the bulk diffusional effect of the reactant gas was minimized. It could thus be assumed that the concentration of reactant gas surrounding each coke particle in the sample pan was constant. This assumption is supported by the study of Jefferson 15 to react with air at different temperatures. The reaction between Jefferson 15 and air at 500°C was chemical reaction controlled which means that extraparticle diffusion resistance was negligible. The unreacted-core-shrinking model⁵ could thus be used under this assumption to study the reactions between Alabama cokes and reactant gases.

Table 1

REACTIVITY OF ALABAMA COKES WITH AIR AT 500°C
AND WITH CARBON DIOXIDE AT 900°C

Sample Name	Fixed Carbon	Reactivity With Air	Reactivity With CO ₂
PSOC 189	42.8	1.51 ⁺ , 0.67 ⁺⁺	
Blue Creek 9-B	44.4	0.37	0.27
Upper Cobb 4	51.5	0.37	0.33
Utley 3	52.8	0.18, 0.16, 0.17, 0.16	0.60
Blue Creek Type B	53.5	2.76 ⁺⁺⁺ , 0.17 ⁺⁺⁺⁺	0.41
Thomas 6	53.6	0.20	0.17
Blue Creek 9-A	55.0	0.16	0.11
Jagger 12	56.3	0.13	0.11, 0.11
Jefferson 15	59.3	0.12	0.24

+ Reactivity of coke prepared in the TGA.

++ Reactivity of coke prepared in the tube furnace.

+++ Reactivity of Mr. R. Mohan's sample.

++++ Reactivity of coke prepared in the tube furnace.

Fig. 3. Variation of Reactivity in CO_2 of 1000°C Alabama Cokes as a Function of the Fixed Carbon Content in the Parent Coal.

With the weight versus time curves generated by the TGA, it is possible to determine reaction order for carbon, and to determine whether the reaction was ash diffusion controlled or reaction rate controlled.

R. G. Jenkins, et al.,¹ reported in their paper that all of their experiments showed that the carbon-air and carbon-carbon dioxide reactions were zero order reactions with respect to the solid phase (carbon) over the portion of the reaction curve which was used to determine reactivity. Most of the reactions of Alabama cokes showed a rectilinear portion on the reaction curve which indicated a zero-order reaction with respect to the carbon over this rectilinear range.

For those reactions which did not show a clear rectilinear portion on the reaction curve, a different reaction model was tested. C. Y. Wen⁵ developed the unreacted-core-shrinking model for the gas-solid reaction which was zero-order with respect to the solid phase and first-order with respect to the fluid phase. In the case that the reaction products were gas and ash, Wen offered a way to determine if the reaction was chemical reaction controlled or ash diffusion controlled. Let x represent the fractional conversion of carbon during the process. If a log-log plot of $(1 - (1 - x)^{1/3})$ versus the reaction time had been straight with a slope of unity, chemical reaction would have controlled the rate, whereas a slope of 2 would indicate that the ash diffusion was the controlling factor. Figure 4, as adopted from the reaction between Jagger 12 and air at 500°C, is a typical plot of $\log 1 - (1 - x)^{1/3}$ versus $\log (\theta)$ for the reactions involved in this work. It was determined that for the carbon-air reactions at 500°C for all except Mr. Mohan's sample the slope was slightly higher than unity, hence the reactions were consistent with zero-order with respect to carbon, and with chemical reaction controlled. The $\log 1 - (1 - x)^{1/3}$

Fig. 4. A Typical Plot for the Fit of the Unreacted-Core-Shrinking Model.

versus $\log (\theta)$ for Mr. Mohan's sample (Blue Creek Type B) was not quite straight, but the segments on the graph had slopes close to two which indicated that this reaction was ash-diffusion controlled. The importance of ash diffusion for Blue Creek Type B was verified by the work of Mohan³.

Not all reactions fit the zero-order behavior with respect to carbon. For the reactions of Jefferson 15 with air at 650°C, 700°C, and the reactions of Blue Creek 9-A, Blue Creek 9-B, Jagger 12, Jefferson 15 with CO₂ at 900°C, the slopes of the above-mentioned curves were less than unity, which meant that these reactions took place in a different manner from those of the other Alabama cokes. N. Kayembe and A. H. Pulsifer² observed that the reaction between coke and steam was of first order with respect to carbon. The kinetics for the first order reaction is:

$$\frac{-dC_c}{d\theta} = kC_c$$

where C_c is the fraction of carbon remaining in the reactor at time θ and k is the first order specific rate constant. Upon integration,

$$\ln C_c = -k\theta + \text{constant}$$

at

$$\theta = 0, C_c = 1, \text{ constant} = 0,$$

and

$$C_c = \exp (-k\theta)$$

Thus, a plot of natural logarithm of C_c as a function of reaction time is a straight line with $-k$ as slope and zero as intercept. Figure 5 is a typical plot of $\ln C_c$ versus θ curve for those reactions which did not show a rectilinear portion on the weight versus time curve connected with this work. Except for Jagger 12, all the other reactions of this kind fit the first order model satisfactorily in the time intervals selected. It was

Fig. 5. A Typical Plot for the Fit of the First Order Model.

difficult to estimate the reaction order of the Jagger 12-CO₂ reaction but for simplicity it was approximated by the first order model with respect to carbon. In all the experiments the reaction rate slowed down after a certain period of time. This was considered to be the result of these two factors. As the reaction proceeded, there was an ash layer developed which caused the reactive gas molecules reaching the reacting surface to be limited by diffusion through the ash layer. The active sites per unit reacting surface area were reduced during the process.

B. Reaction Rate Constants for the Coke from Alabama Coals

With the present information, it is possible to determine the reaction rate constants according to the following equations:

1. For the zero order (with respect to carbon), reaction controlled reactions:

$$\theta = \frac{R}{k_s C_{AO}} (1 - (1 - X)^{1/3})$$

R: particle radius before the reaction, cm

k_s : reaction rate constant based on the reacting surface area $\frac{\text{cm}^4}{\text{hr-gmole}}$

C_{AO} : concentration of the gaseous reactant in the bulk of the gas, moles/l

X: fractional conversion of carbon during the process

2. For the first order (with respect to carbon) reactions:

$$-k = \text{slope of the plot of } \ln C_C \text{ vs. } \theta$$

Since the coke was crushed to -40 + 100 U. S. mesh, the shape and the size of the particles were irregular. The equation to evaluate the rate constants of the reactions which fitted model (A) was used as an

approximation. The particles were approximated to spherical particles. The R value was taken to be the logarithmic average of the opening of the two mesh sizes used⁴.

$$d_2: \text{ opening of U. S. No. 40 mesh} = 0.0164 \text{ in.}$$

$$d_1: \text{ opening of U. S. No. 100 mesh} = 0.0058 \text{ in.}$$

$$d_m = (d_2 - d_1) / \ln \left(\frac{d_2}{d_1} \right) = 0.0102 \text{ (in.)}$$

$$R = dm/2 = 0.0051 \text{ in.} = 0.013 \text{ cm.}$$

$$C_{AO} = 3.31 \times 10^{-6} \text{ gmole/cm}^3 \text{ in the case that coke reacted with air}$$

$$C_{AO} = 1.039 \times 10^{-5} \text{ gmole/cm}^3 \text{ in the case that coke reacted with CO}_2$$

The reaction rate constants were evaluated and tabulated in Table 2 by using the least square fit and by using the dm value discussed above. Each of the rate constants in Table 2 were the average of four values obtained from different conversions in a single run. It is worthwhile to point out that the calculated rate constants for the reactions which fit model (A) agree with the theory. This can best be illustrated by an example.

Since k_s should be constant at a certain temperature, then the following should hold:

$$\frac{\theta_1}{\theta_2} = \frac{\frac{R}{k_s C_{AO}} (1 - (1 - X_1)^{1/3})}{\frac{R}{k_s C_{AO}} (1 - (1 - X_2)^{1/3})}$$

for two different fractional conversions of carbon during the same run.

Table 2

REACTION RATE CONSTANT OF ALABAMA COKES TO
 REACT WITH AIR AT 500°C AND WITH
 CO₂ AT 900°C

With Air at 500°C		With CO ₂ at 900°C	
Sample Name	Rate Constant, $\frac{\text{cm}^4}{\text{h-gmole}}$	Sample Name	Rate Constant
Utley	209	Utley 3	1155 $\frac{\text{cm}^4}{\text{h mole}}$
Upper Cobb 4	457	Upper Cobb 4	635 $\frac{\text{cm}^4}{\text{h mole}}$
Thomas 6	228	Thomas 6	289 $\frac{\text{cm}^4}{\text{h mole}}$
Blue Creek 9-A	171	*Blue Creek 9-A	0.090 (h ⁻¹)
Blue Creek 9-B	476	*Blue Creek 9-B	0.14 (h ⁻¹)
Jagger 12	171	*Jagger 12	0.081 (h ⁻¹)
Jefferson 15	127	*Jefferson 15	0.23 (h ⁻¹)
		Blue Creek Type B	0.0070 $\frac{\text{cm}^4}{\text{h mole}}$

* : First order (with respect to carbon) reaction rate constants.

Others: Zero order (with respect to carbon) reaction rate constants.

Take the reaction between Utley 3 and CO_2 at 900°C as the example:

at $\theta_1 = 30$ min. $X_1 = 0.2684931507$

at $\theta_2 = 45$ min. $X_2 = 0.397260274$

at $\theta_3 = 60$ min. $X_3 = 0.4931506849$

$$\frac{\theta_2}{\theta_1} = 1.5 \frac{1 - (1 - X_2)^{1/3}}{1 - (1 - X_1)^{1/3}} = \frac{0.1552855202}{0.098969555} = 1.569$$

$$\frac{\theta_3}{\theta_1} = 2 \frac{1 - (1 - X_3)^{1/3}}{1 - (1 - X_1)^{1/3}} = \frac{0.2026016948}{0.098969555} = 2.048$$

Similar relationships hold for other reactions of the same kind.

C. Conclusions

The following conclusions have been drawn from this work:

1. Most of the Alabama coals swell when heated. Care must be given to address this problem in both laboratory and field work.
2. Coke is much less reactive toward CO_2 than toward air at a given temperature. The gasification reaction is considered to be primarily accomplished by oxygen when air is used as reactant gas in a real underground gasification process.
3. The reactivity toward air of the coke from Alabama coals made at 1000°C decreases in general as the fixed carbon in the parent coal increases. Coals of higher carbon content are more difficult to gasify.
4. The reactivity toward CO_2 of the coke from Alabama coals made at 1000°C cannot be correlated to the fixed carbon content in the parent coal.

5. Pyrolyzing conditions have a strong influence on coke reactivity.
Heat treatment temperature has the most significant influence.
Higher heat treatment temperature results in lower reactivity.
Thermal aging also has appreciable influence on coke reactivity.
The longer a sample stays at a high temperature, the less reactive it becomes.
6. The reactivity of coke increases with reaction temperature in an exponential order.
7. The gasification reactions between the completely pyrolyzed Alabama cokes and air at 500°C were consistent with zero-order reaction kinetics with respect to carbon and chemical reaction rate controlling when analyzed by the unreacted-core-shrinking model.
8. The reaction order with respect to carbon for the carbon-carbon dioxide reactions is difficult to predict. The unreacted-core-shrinking model was used to analyze the reaction data and showed some of the reactions to be consistent with zero-order reaction kinetics with respect to carbon, while others appeared to be first order. These reaction rates appear to be controlled by reaction kinetics in the experiments of this work.

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