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THERMODYNAMIC PROPERTIES OF PULVERIZED COAL DURING
RAPID HEATING DEVOLATILIZATION PROCESSES

QUARTERLY PROGRESS REPORT

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1.0 Executive Summary

Knowledge of the thermodynamic and morphological properties of coal associated with rapid heating decomposition pathways is essential to progress in coal utilization technology. Specifically, knowledge of the heat of devolatilization, surface area and density of coal as a function of rank characteristics, temperature and extent of devolatilization in the context of rapid heating conditions is required both, for the fundamental determination of kinetic parameters of coal devolatilization, and to refine existing devolatilization sub-models used in comprehensive coal combustion codes.

The objective of this research is to obtain data on the thermodynamic properties and morphology of coal under conditions of rapid heating. Specifically, the total heat of devolatilization, external surface area, BET surface area and true density will be measured for representative coal samples. In addition, for one coal, the contribution of each of the following components to the overall heat of devolatilization will be measured: the specific heat of coal/char during devolatilization, the heat of thermal decomposition of the coal, the specific heat capacity of tars, and the heat of vaporization of tars.

Last quarter, matched tar and char samples were generated in the UTRC EFR for 75-106 μm particle size cuts of the two study coals, PSOC 1451D and 1561D. Elemental analysis and microbomb calorimetry results for the tars and chars have been completed in the current quarter. Light gas yields have also been determined from the FT-IR spectra acquired during the EFR runs. The product yields and chemical characteristics are all in agreement with the results of previous investigations in this laboratory using smaller particle sizes. The tar and char samples are being used to determine the thermodynamic properties and morphology of coal as a function of extent of reaction.

Morphological characterization of the parent coal samples has been completed by the University of Pittsburgh. Results are presented for true density, CO_2 surface area, mercury porosimetry, and particle size and shape measurements using image analysis.

Work on Task 3, Low Temperature Specific Heat Capacity of Volatile Coal Samples, is nearly completed and will be reported on next quarter. Task 4, Heat of Devolatilization of Volatile Coal Samples, will be initiated. The heat of thermal decomposition of PSOC 1451D (Task 5) will be calculated from the data reported here. The Task 10 effort, Morphological Characterization of Coal/Char Samples as a Function of Extent of Devolatilization, will continue at the University of Pittsburgh. Work will focus on measurement of the morphological characteristics of the char samples as a function of extent of reaction.

2.0 Introduction and Overview of Technical Objectives

Introduction

Knowledge of the thermodynamic and morphological properties of coal associated with rapid heating decomposition pathways is essential to progress in coal utilization technology. Specifically, knowledge of the heat of devolatilization, surface area and density of coal as a function of rank characteristics, temperature and extent of devolatilization in the context of rapid heating conditions is essential to the fundamental determination of kinetic parameters of coal devolatilization. These same properties are also needed to refine existing devolatilization sub-models utilized in large-scale modeling of coal combustion systems.

In many instances the kinetic analysis of rapid coal devolatilization data acquired in laboratory reactors depends on an assumed set of heat capacity, heat of devolatilization and particle morphology parameters. In other cases, attempts to measure particle temperatures during devolatilization (Refs. 1,2,3) lead to results that are not in agreement either with a priori determinations obtained with energy balance expressions using estimations of thermodynamic properties of coal found in the open literature or inert, carbon-based calibration standards used to calibrate the reactor system. Although it is not always clear if the discrepancies are due to inadequate thermodynamic properties of the particle during devolatilization, particle shape factors, inadequate emittance values or some combination of these factors, it is now apparent that independent determinations of these properties in heating conditions characteristic of disperse phase coal utilization technology is necessary. Moreover, since it is now well-established that the energy transfer rate between an environment and injected coal particles, significantly influences the observable devolatilization response time of the particles (Ref. 4), the independent determination of the specific heat and devolatilization heat of reaction of coal is necessary.

Objective

The objective of this research is to obtain data on the thermodynamic properties and morphology of coal under conditions of rapid heating. Specifically, the total heat of devolatilization, external surface area, BET surface area and true density will be measured for representative coal samples. The coal ranks to be investigated will include a high volatile A bituminous (PSOC 1451D) and a low volatile bituminous (PSOC 1516D). An anthracite (PSOC 1468) will be used as a non-volatile coal reference. In addition, for one coal, the contribution of each of the following components to the overall heat of devolatilization will be measured: the specific heat of coal/char during devolatilization, the heat of thermal decomposition of the coal, the specific heat capacity of tars, and the heat of vaporization of tars.

Project Tasks

The work required to complete the objectives of this investigation has been divided into eleven tasks. Each of these tasks is summarized below. The details of the technical approach have been described previously (Ref. 5).

Task 1 - Project Work Plan

UTRC shall provide the DOE with a detailed Project Plan within 30 days of contract initiation. Work on no other tasks shall proceed until written approval of this plan is obtained by UTRC from the DOE Project Manager. The Project Plan shall include a Management Plan, Milestone Schedule, Status Report, and Cost Plan.

Task 2 - Calibration of the Heated Grid Calorimeter

UTRC shall measure the specific heat of the non-volatile reference coal (anthracite, PSOC 1468) from 25° C to 800° C at heating rates of 1000° C/sec and lower in the heated grid (HG) device. The values will be established relative to NBS calorimeter standards for differential scanning calorimeter (DSC) and differential thermal analysis (DTA) samples. The sensitivity of the heated grid device for determining thermodynamic properties at high heating rates will be established before proceeding with Tasks 3-9.

Task 3 - Low Temperature Specific Heat Capacity of Volatile Coal Samples

UTRC shall measure the specific heat capacity for a minimum of one coal sample over the temperature range of 25° C to 300° C. The measurements will be conducted at heating rates of 1000° C/sec and lower in the UTRC HG.

Task 4 - Heat of Devolatilization of Volatile Coal Samples

UTRC shall measure the heat of devolatilization for a minimum of one coal sample over the temperature range of 25° C to 800° C using heating rates of 1000° C/sec in the calibrated HG. Devolatilization product characteristics will be determined to ensure the devolatilization pathway is that followed in the entrained flow reactor (EFR).

Task 5 - Heat of Thermal Decomposition of Volatile Coal Samples

UTRC shall measure, using the EFR and microbomb calorimeter (MBC), the heat of decomposition for a minimum of one coal sample by: (1) devolatilizing the coal in the EFR, (2) determining the char, tar and gas yields, (3) measuring the tar and char elemental compositions, (4) determining the heat of combustion of the char and tar products using the MBC, (5) estimating the heat of combustion of the gaseous products, and (6) using Hess's and Kirchoff's laws to derive the heat of decomposition.

Task 6 - Specific Heat and Heat of Fusion of Tars from Rapid Heating

UTRC shall measure the specific heat and heat of fusion of tar samples for a minimum of one coal sample over the temperature range from 25° C to 300° C using the HG and tar samples generated in the EFR.

Task 7 - Heat of Vaporization of Tars from Rapid Heating

UTRC shall measure the heat of vaporization of tar samples for a minimum of one coal sample over the temperature range from 300° C to 700° C using the HG and tar samples generated in the EFR.

Task 8 - Specific Heat of Char Samples

UTRC shall measure the specific heat of the coal/char as a function of extent of devolatilization and temperature for a minimum of one coal sample using partially devolatilized samples from the EFR and the HG.

Task 9 - Comparison of Directly Measured Heat of Devolatilization and Thermodynamic Function Determined Value

UTRC shall determine the heat of devolatilization for a minimum of one coal sample based on independent measurements of heat of decomposition (Task 5), specific heat and heat of fusion of tars (Task 6), heat of vaporization of tars (Task 7), and specific heat of coal/chars (Task 3 and Task 8). This value shall be compared and contrasted to the directly measured heat of devolatilization values from 25° C to 800° C obtained in Task 4.

Task 10 - Morphological Characterization of Coal/Char Samples as a Function of Extent of Devolatilization

UTRC shall characterize statistically representative samples of coal/char particles as a function of extent of devolatilization for a minimum of one coal sample. The characterization shall be with respect to combustion related reactivity parameters such as swelling and cenosphere development, CO₂ or O₂ surface areas, and helium (true) density.

Task 11 - Reporting

UTRC shall comply with all required reporting over the 18 month period of the contract.

3.0 Task 5. Heat of Thermal Decomposition of Volatile Coal Samples

The objective of this task is to determine the heat of decomposition of PSOC 1451D by: (1) devolatilizing the coal in the entrained flow reactor (EFR) to generate matched char and tar samples as a function of extent of devolatilization, (2) determining the char, tar and light gas yields, (3) measuring the tar and char elemental composition, (4) determining the heat of combustion of char and tar products using a microbomb calorimeter (MBC), (5) estimating the heat of combustion of the gaseous products, and (6) applying Hess's Law to determine the heat of decomposition.

UTRC-EFR

The essential components of the UTRC entrained flow reactor (EFR) are shown in Figures 1-3. The reactor consists of a coal feeder, a drop tube furnace using argon as the entrainment gas, and a product collection and separation system. The EFR is operated without preheating the entrainment gas so that gas temperatures remain below wall temperatures throughout the reactor. Extra-particle gas-phase reactions of tars are minimized by operating in this mode. This is in contrast to the operation of most entrained flow reactors, where the entrainment gas temperature is matched to the reactor wall temperature. The heat transfer environment of the reactor has been characterized and documented in previous investigations (Refs. 4,6,7).

The coal feed system consists of a jet-stirred, fluidized-bed supply reservoir connected to the reactor primary stream injector via a narrow bore transfer tube. The feed system is capable of sustained delivery of optically thin streams of constant mass delivery rate, provided the feeder is loaded with a narrow particle size range initially. Particle sizes from 10 to 300 microns have been used in the feeder.

The product separation system collects the entire flow stream from the reactor into a water cooled probe. An aerosol phase separation system is employed to obtain char-free tar samples (Figs. 2,3). The separator is designed to "pull off," in quantities proportional to the gas split, all particles or aerosols that are less than 2 microns in diameter. Inertia carries larger particles into the char separation (cyclone train) system. The smaller particles are able to follow the curved flow streamlines and enter the aerosol separation (impactor) system. Both separation trains contain porous metal filters as final stages. Tars are primarily deposited on these filters. Detailed mass fraction analysis of the tars deposited throughout the separation system showed that only about 15% of the total tar mass is deposited in the pre-filter stages (Ref. 4). Furthermore, chemical characterization of the samples collected within the product separation system indicate that the tars collected on the final filters are representative of the collective, captured tar mass. After particulate and aerosol separation, the gas stream is analyzed on line for IR-active gases by use of a Fourier transform infrared spectrometer (FT-IR) coupled to a multipass cell having an equivalent path length of 43.5 m.

EFR Devolatilization Experiments

The two study coals, PSOC 1451D (a HVA bituminous coal from the Pittsburgh #8 seam) and PSOC 1516D (a LV bituminous coal from the Lower Kittaning, PA seam) were devolatilized in the UTRC-EFR (entrained flow reactor) at four different reactor temperatures. These "D" coals were supplied by DOE under an earlier contract and were size classified using the "Vortec" aerodynamic separation process. The 75-106 μm size cut is used in the performance of this contract. All EFR conditions - temperature set points, carrier gas flow rates, and sampling probe position - are identical to those used during two previous contracts (Refs. 7,8). The reactor operating conditions are characterized by peak wall and gas temperatures given in Table I below.

TABLE I
ENTRAINED FLOW REACTOR EXPERIMENTS

Run	Coal Type	Wall Temp. (°C)	Gas Temp. (°C)	% Mass Loss
EFR019	1451D	668	507	16.7
EFR018	1451D	825	660	31.0
EFR020	1451D	939	796	30.0
EFR021	1451D	1058	895	45.7
EFR022	1516D	668	507	NM *
EFR023	1516D	825	660	8.8
EFR024	1516D	939	796	19.3
EFR025	1516D	1058	895	26.0

* NM - Not Measurable - Volatile yields too low to measure reliably with ash tracer technique. (Mass loss in heated grid experiments at similar final temperature is 5-6%.)

Sufficient quantities of matched char and tar samples were produced from the PSOC 1451D coal to complete all the necessary measurements for the performance of this contract. It was not possible to save significant quantities of tars from the PSOC 1516D runs. The high static charge of the samples made it difficult to remove the tars from the porous metal final filters used in the UTRC-EFR sample collection system.

Elemental Analysis

Elemental analyses of the char samples generated in the EFR have been performed. Additional runs were required for several of the PSOC 1516D chars to improve the ash determinations. Elemental analysis for the PSOC 1451D tars has also been completed. However, analysis of the 1516D tars was not possible due to the limited amounts of sample which could be collected and saved for this coal.

The elemental composition of the chars and tars are given in Table II. The reported results are average values for 3-5 replicate analyses of each sample. These results are in excellent agreement with previous results at UTRC using a smaller size cut of the Vortec separated "D" coals (63-75 μm). In particular, the tars are hydrogen-rich and heteroatom-poor relative to the parent coal (Fig. 4). The hydrogen content of the tars approaches that of the parent coal with increasing extent of devolatilization, whereas tar nitrogen content remains nearly equal to that of the parent coal.

EFR Product Yields

Mass loss values calculated using the ash tracer technique are given in Table I. These mass loss results are in agreement with data obtained under two previous UTRC investigations utilizing identical experimental conditions (Figs. 5,6). Total volatile yields for PSOC 1451D obtained in the current investigation are compared with EFR data acquired in 1989 (Ref. 7) for 20-30 μm particles in Fig. 5. The apparent plateau in volatiles yield between 650-800°C is again observed. Results for zero hold time (ZHT) heated grid (HG) runs are also shown. The HG experiments were conducted in one atmosphere helium and utilized a heating rate of 1000°C/s. The heated grid yields show a steeper temperature dependence near 600 °C than is observed in the EFR. Current results for total volatile yields for PSOC 1516D are compared with EFR data acquired in 1991 (Ref. 8) for 63-75 μm particles in Fig. 6.

The FT-IR spectra from the EFR experiments have been quantified to obtain light gas yields. Results for the IR-active gases are given in Table III. Relative yields of the light gases are plotted in Figures 7 and 8 for PSOC 1451D and 1516D, respectively. Relative yield data for 20-30 μm diameter particles of PSOC 1451D obtained in 1989 (Ref. 7) are given in Fig. 9. Data for 63-75 μm particles of PSOC 1516D from 1991 (Ref. 8) are plotted in Fig. 10. The relative yields given in Figs. 9 and 10 were calculated by normalizing the data by the yield observed at the highest EFR temperature run (1241°C wall temp.; 1053°C gas temp.). Since, experiments were not conducted at this temperature for the present investigation, the relative yields in Figs 7 and 8 have been normalized to match those of the previous investigations at the 1058°C wall temperature (895°C gas temp.) condition.

The light gas yields obtained in the present study, using 75-106 μm particles, are in excellent agreement with results obtained for the same coals in previous investigations in this laboratory. The gas yields are dominated by methane and ethylene at gas temperatures below 660°C. Yields of hydrogen cyanide and acetylene increase substantially at peak EFR gas temperatures above 800°C.

TABLE III EFR GAS YIELDS - 75-106 MICRON PARTICLES - 1993 DATA

RUND	PSOC	WALL TEMP (C)	GAS TEMP (C)	%CH4	%C2H6	%C2H4	%C2H2	%C6H6	%HCN	%CO	%CO2	TOT. GAS YLD.
EFR 19	1451D	668	507	0.89	0	0.05	0	0	0	0	0.09	1.04
EFR 18	1451D	825	660	1.21	0	0.15	0.01	0	0.04	0	0.26	1.68
EFR 20	1451D	939	796	1.6	0	0.18	0.04	0	0.07	(0.2)*	0.11	2.19
EFR 21	1451D	1058	895	1.56	0	0.24	0.09	0	0.1	0.1	0.41	2.49
EFR 22	1516D	668	507	0.92	0	0.03	0	0	0	0	0.24	1.18
EFR 23	1516D	825	660	1.07	0	(0.16)*	0.02	0	0	0	0.26	1.51
EFR 24	1516D	939	796	1.38	0	0.08	0.02	0	0.05	0	0.12	1.65
EFR 25	1516D	1058	895	1.62	0	0.14	0.04	0	0.07	0	0.1	1.98
*() Data considered unreliable; not plotted in Figures												

Calorific Values

Microbomb calorimeter measurements of the heats of combustion of the parent coal, char and tar samples have been performed. The raw and ash corrected calorific values of the samples are given in Table IV. Note that, for PSOC 1451D, the 75-106 μm size cut used in this investigation has a much higher ash content (13.9%) than the 63-75 μm size cut (4.09%) used in previous DOE-funded investigations at UTRC (Refs. 7,8). However, the *ash corrected* calorific values for the parent coal and char samples are within 2% of those determined in the previous investigation. The ash content of the larger size cut samples of PSOC 1516D is also somewhat higher (20.0% vs. 16.8%). The ash corrected calorific values agree within 5% to the results for the smaller particle size samples. The consistency of the microbomb calorimeter results between the two investigations provides further support for the repeatability of the UTRC entrained flow reactor experiments.

TABLE IV CALORIFIC VALUES OF PARENT COAL, CHAR AND TAR SAMPLES

		CHARS			
COAL TYPE	WALL TEMP (C)	GAS TEMP (C)	UNCORRECTED HEATING VALUE (BTU/lb)	ASH CORRECTED HEATING VALUE (BTU/lb)	% ASH (PE-ME)
1451D	PARENT COAL	-	12554	14581	13.9
1451D	668	507	12602	14430	12.7
1451D	825	660	12895	14122	8.7
1451D	939	796	12208	14079	13.3
1451D	1058	895	11294	13640	17.2
1516D	PARENT COAL	-	12087	15108	20.0
1516D	668	507	12753	15217	16.2
1516D	825	660	11747	14467	18.8
1516D	939	796	11764	15043	21.8
1516D	1058	895	11818	15324	22.9
		TARS			
COAL TYPE	WALL TEMP (C)	GAS TEMP (C)	UNCORRECTED HEATING VALUE (BTU/lb)	ASH CORRECTED HEATING VALUE (BTU/lb)	% ASH (PE-ME)
1451D	PARENT COAL	-	12554	14581	13.9
1451D	668	507	15100	15100	-
1451D	825	660	15143	15143	-
1451D	939	796	15150	15150	-
1451D	1058	895	14895	14895	-

4.0 Task 10. Morphological Characterization of Coal/Char Samples as a Function of Extent of Devolatilization.

The overall objectives of this task are to characterize statistically representative samples of coal/char particles as a function of extent of devolatilization. The characterization shall be with respect to combustion related reactivity parameters such as swelling and cenosphere development, CO₂ or O₂ surface areas, and helium (true) density.

This task is being performed under subcontract to the University of Pittsburgh under the direction of Prof. George Klinzing. The subcontract was placed and work initiated in this quarter on morphology measurements of the parent coals. As a first step, the relevant equipment and instruments were checked and overhauled and required maintenance done. Trial runs were carried out using other coal samples and the results were found to be satisfactory.

True Density

True densities of the parent coal samples were determined using a Quantachrome Helium pycnometer. Arrangement was made to hook up the pycnometer to the fine vacuum system of the B.E.T. apparatus and samples were degassed in situ at room temperature for 2-3 hours before testing. Four runs were carried out for each sample. The results obtained (Table V) are in good agreement with values obtained in other investigations (Ref. 7).

CO₂ Surface Area

Total surface area was determined using an ORR pore-volume analyzer from Micromeritics Corporation. Samples were degassed for 15 hours at 60°C and CO₂ adsorption was carried out at 298°K. Measurements were obtained at four pressures: 100, 200, 400 and 600 torr. Surface areas were determined using the B.E.T. and Polanyi-Dubinin equations. The latter gives more correct results for these experiments since the maximum partial pressure is around 1/100 of the saturation pressure of CO₂ (800 psi). Results are given in Table V. Maloney (Ref. 9) conducted CO₂ adsorption experiments at 298 K for different samples from the same coal seams as the study coals. (PSOC 1099: Pittsburgh #8 and PSOC 1133: Lower Kittaning) Surface areas calculated from the Polanyi-Dubinin equation were 170 and 220 m²/g (daf) for the HVA and LV coals, respectively. Maloney outgassed the samples for 16 hours at 110 °C at a pressure of 10⁻⁵ torr. The results (daf) for the HVA coal are in excellent agreement. The higher outgassing temperature and smaller particle size (200 x 270 mesh; 62 µm mean dia.) may account for the difference in the two sets of results for the LV bituminous coal.

Mercury Porisimetry

A mercury porisimeter from Aminco Winslow was used to determine internal porosity and pore volume distribution. The porosities are given in Table V.

Image Analysis

A Leitz Tas plus image analyzer is being used for particle size and shape analysis using transmission microscopy. Particle size is determined by averaging three length measurements per particle. Volume is calculated assuming spherical particles. Size distributions based on number (measured) and volume (calculated) have been obtained for both types of coals. Log-probability distribution and differential distribution for size based on volume are given in Figures 11 - 14. As is evident, the distribution is not unimodal. The mean sizes are listed in Table V. Particle size distribution analysis will also be done by using a Laser Microtrac to compare results. Fractal analysis of the image data will be used to describe particle shape.

TABLE V

PARENT COAL MORPHOLOGY MEASUREMENTS

	PSOC 1451D	PSOC 1516D
True density (pycnometer)	1.393 g/cc	1.513 g/cc
Bulk density (volumetric)	0.638 g/cc	0.673 g/cc
Porosity	54.2 %	55.5 %
B.E.T. surface area	77 m ² /g	42 m ² /g
P-D surface area	138 m ² /g	88 m ² /g
P-D surface area (daf)	163 m ² /g	110 m ² /g
Pore size with max. volume	approx. 18 μm	approx. 18 μm
Porosity (Mercury porisimetry)	43.5 %	38.6 %
Number-mean size	14.9 μm	7.6 μm
Volume-mean size	35.8 μm	27 μm

5.0 Planned Activities

The results for Task 3, Low Temperature Specific Heat Capacity of Volatile Coal Samples, which is nearly completed, will be reported on next quarter. Work on Task 4, Heat of Devolatilization of Volatile Coal Samples, will be initiated. The heat of thermal decomposition of PSOC 1451D (Task 5) will be calculated from the data reported here. The Task 10 effort, Morphological Characterization of Coal/Char Samples as a Function of Extent of Devolatilization, will continue at the University of Pittsburgh. Work will focus on measurement of the morphological characteristics of the char samples.

6.0 References

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FIGURE 1

FIGURE 2

FIGURE 3

FIGURE 4

TAR ELEMENTAL COMPOSITION AT VARYING EXTENTS OF REACTION
PSOC 1451D - EFR EXPERIMENTS

FIGURE 5

FIGURE 6

LIGHT GAS YIELDS VS. PEAK GAS TEMPERATURE (C) PSOC 1451D, 75-106 MICRONS, 1993 DATA

FIGURE 7

LIGHT GAS YIELDS VS. PEAK GAS TEMPERATURE (C) PSOC 1516D, 75-106 MICRONS, 1993 DATA

FIGURE 8

FIGURE 9

LIGHT GAS YIELDS VS. PEAK GAS TEMPERATURE (C)
 PSOC 1451D, 20-30 MICRONS, 1989 DATA

LIGHT GAS YIELDS VS. PEAK GAS TEMPERATURE (C)
PSOC 1516D, 63-75 MICRONS, 1991 DATA

Log-probability distribution for coal PSOC 1451d

Log-probability distribution for coal PSOC 1516d

Differential size distribution for coal PSOC 1451d

Differential size distribution for coal PS0C 1516d

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