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THERMODYNAMIC PROPERTIES OF PUL

RAPID HEATING DEVOLATILIZATION

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CONTENTS

1.0 Executive Summary	1
2.0 Introduction and Overview of Technical Objectives	2
3.0 Task 3. Low Temperature Specific Heat Capacity of Volatile Coal Samples	5
PSOC 1451D	5
PSOC 1516D	5
4.0 Task 4. Heat of Devolatilization of Volatile Coal Samples	6
PSOC 1451D	6
PSOC 1516D	7
5.0 Task 10. Morphological Characterization of Coal/Char Samples as a Function of Extent of Devolatilization	8
Pore Volume Distributions	8
Particle Size and Size Distributions	9
Summary	10
6.0 Planned Activities	11
7.0 References	12

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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.

The low temperature heat capacity of a HVA bituminous coal (PSOC 1451D, Pittsburgh #8) and a LV bituminous coal (PSOC 1516D, Lower Kittanning) have been measured over the temperature range from 25°C to 300°C. Results for two runs for each coal sample are presented to provide an indication of the magnitude of the errors associated with the measurements. The data are the first ever heat capacity measurements for coal at high heating rates. Improvements in the sample mass and area measurements are required in order to reduce the size of the error bars.

The "effective" heat capacity, or overall heat of devolatilization, of a HVA bituminous coal (PSOC 1451D, Pittsburgh #8) and a LV bituminous coal (PSOC 1516D, Lower Kittanning) have been measured over the temperature range from 25°C to 800°C. The "effective" heat capacity for the HVA coal exhibits a sharp rise between 550 - 650°C and the area under the curve, representing the heat of devolatilization, is estimated to be 50-55 cal/g. Effective heat capacity measurements for PSOC 1516D appear to show a higher heat capacity for the higher rank LVB coal than the HVA coal. However, these are effective heat capacities, and as such include not only the specific heat of the coal/char, but also the heat of devolatilization during the volatiles evolution process. Mass loss for the LVB coal begins at a lower temperature and has a much weaker temperature dependence than that for the HVA coal. Thus, the heat of devolatilization is spread over a much broader temperature range for this coal and cannot be determined from the effective heat capacity data by simply inferring a baseline for the "true" specific heat of the coal/char.

Morphological characterization of the parent coal samples has been completed by the University of Pittsburgh. The previous quarterly report presented results for true density, CO₂ surface area, porosity from mercury intrusion porosimetry, and particle size distributions using image analysis. This quarter results are given for pore volume distribution and particle size distributions using a Laser Microtrac. The similarity of size distributions from both the Microtrac and the image analyzer confirms the reliability of the use of the latter for size distribution measurements.

Work on Task 8, Specific Heat of Char Samples, will be initiated in the next quarter. 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 continue on the 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 3. Low Temperature Specific Heat Capacity of Volatile Coal Samples

PSOC 1451D

The heat capacity of PSOC 1451D, a HVA bituminous coal (Pittsburgh #8) has been measured over the temperature range from 25°C to 300°C. Four separate heated grid runs have been conducted; one using the blank stainless steel screen as the reference material, the others using 100 μm thick carbon foil (99.9% pure, Goodfellow) as the reference material. The results obtained using the blank screen material as the reference are not considered to be as reliable (see discussion under Task 4 below) and have been discarded. Also, anomalous results were observed for one of the runs using the carbon foil reference material. Heat capacity was overestimated as a result of the existence of a greater than nominal sample mass loading in the vicinity of the sample thermocouple. Low temperature heat capacity measurements for the remaining two runs are plotted in Fig. 1. Results from Run A agree quite well with the Merrick model, while those for Run B are considerably higher. These runs provide an indication of the magnitude of the errors associated with the measurements. The data are the first ever heat capacity measurements for coal at high heating rates. Improvements in the sample mass and area measurements are required in order to reduce the size of the error bars. The local mass loading around the thermocouple must be measured with greater accuracy; use of the nominal mass loading (total sample mass/total sample area) is valid only in the limit of a perfectly uniform sample distribution. The current set of experiments represent our best attempts at obtaining a uniform loading of the coal samples on the grid. No further refinement of the grid sample loading technique is possible for particulate samples.

PSOC 1516D

The heat capacity of PSOC 1516D, a LV bituminous coal (Lower Kittanning) has also been measured over the temperature range from 25°C to 300°C. Results for two independent determinations are presented in Fig. 2. The run-to-run variability in the measured heat capacities is of about the same magnitude as for PSOC 1451D runs. The observed low temperature heat capacity of the LV coal is somewhat higher than that of the HVB coal. This is contrary to the expectation that the higher hydrogen content of the HVB coal will result in lower average coal molecular weight and hence higher specific heat on a per gram basis. An explanation for this apparent discrepancy is discussed in Section 4.0 on overall heat of devolatilization measurements.

4.0 Task 4. Heat of Devolatilization of Volatile Coal Samples

The objective of this task is to measure the overall heat of devolatilization of PSOC 1451D over the temperature range from 25 to 800 °C, essentially extending the methods used in Task 3 to higher temperatures. Initial results using the blank screen as the reference material were obtained. The accuracy of these measurements above 500 °C is poor due to the dominance of the heat loss correction term. The blank grid temperature is 900 - 1000 °C when the sample temperature is 500 °C. The resulting heat loss correction term is about 1- 1.5 J/g-K, which represents 60% of the total heat capacity. A 10% error in the heat loss term will result in a 6% error in the heat capacity.

Measurement errors can be minimized at high temperature by performing runs utilizing discrete sample and reference material loaded regions on the *same* grid. Matching the ρC_p of the reference material loaded area of the grid to that of the sample results in a closer balance in temperature between the two areas of the grid and therefore the heat loss and power dissipation correction terms are significantly smaller. However, the use of particulate materials for both the sample (coal) and the reference material (NIST sapphire) presents significant operational problems. The required handling of the grid during experimental set-up makes it extremely difficult to obtain uniform loading of *both* the sample and reference materials on the grid simultaneously. For these reasons, a 100 μm thick carbon foil (99.9% pure, Goodfellow) is used to replace the sapphire reference material.

PSOC 1451D

Two experiments for PSOC 1451D parent coal samples have been performed utilizing 100 μm thick carbon foil as the reference material. The resulting "effective" heat capacities determined from the runs are plotted in Fig. 3. The sharp rise in effective heat capacity between 550 - 650°C corresponds to the steep temperature sensitivity of mass loss observed for this coal in heated grid experiments (Fig. 4). Since, changes in the mass of the sample are accounted for in the analysis of the data, the sharp rise in effective heat capacity represents the heat of devolatilization for the coal. The area under the effective heat capacity curves from 550°C to the final temperature is found to be 50-55 cal/g. More accurate estimates for the heat of devolatilization will be made when the heat capacity measurements for the char as a function of extent of reaction have been completed (Task 8). The char heat capacity will be used as the baseline for the integration.

PSOC 1516D

Effective heat capacity measurements to 700°C for PSOC 1516D have also been completed. Results for two runs, shown in Fig. 5, appear to show a higher heat capacity for the higher rank LVB coal than the HVA coal. However, these are effective heat capacities, and as such include not only the specific heat of the coal/char, but also the heat of devolatilization during the volatiles evolution process. Mass loss for the LVB coal (Fig. 6) begins at a lower temperature and has a much weaker temperature dependence than that for the HVA coal. Thus, the heat of devolatilization is spread over a much broader temperature range for this coal and cannot be determined from the effective heat capacity data by inferring a baseline for the "true" specific heat of the coal/char. Again, separate heat capacity measurements for the char as a function of extent of reaction would be required to deconvolute the heat of devolatilization from the effective heat capacity data. Such measurements, however, are beyond the scope of this contract.

5.0 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. Morphological characterization of the PSOC 1451D and 1516D parent coal samples has been completed. The previous quarterly report presented results for true density, CO₂ surface area, porosity from mercury intrusion porosimetry, and particle size distributions using image analysis. This quarter results are given for pore volume distribution and particle size distributions using a Laser Microtrac.

Pore Volume Distributions

Mercury intrusion porosimetry measurements of the pore volume distributions for the parent coal samples have been completed. Results for PSOC 1451D and PSOC 1516D are given in Figure 7 and 8, respectively. The highest pore volume at around 20 microns corresponds to the highest volume macropores in bulk coal. Cumulative pore volume distributions are given in Figure 9 and 10, respectively. As is evident from the figures, the pore distributions are similar.

Particle density, microporosity and macroporosity for both coals have been calculated from the pore volume distribution measurements and are given in Table I. Particle density is the weight of the sample divided by the total volume, including pores and cracks. These particle densities, in mercury, are significantly lower than the "true" densities obtained in helium as pores and cracks are included in the particle volume. Other media, like water, benzene, methanol, are not used as they have been observed to cause swelling of coal particles. The pore size corresponding to the sharp change in slope of the pore volume distribution indicates the transition from macropores to micropores owing to totally different characteristics of the two. Micropore and macropore volumes are calculated as the volume of pores below and above, respectively, the transition pore size. Some relevant definitions are as follows:

Total Sample Volume	=	Sample Weight / Bulk Density
Macroporosity	=	Macropore Volume (Ma) / Total Volume
Microporosity	=	Micropore Volume (Mi) / Total Volume
Particle Volume	=	Total Volume - (Ma + Mi)
Particle Density	=	Sample Weight / Particle Volume

Micropore size distribution for the coals based on Madek's method (Refs. 6,7) is given in Figure 11. Distributions for both coals are similar except that PSOC 1516D has higher magnitude of the maximum volume which corresponds to 1 nm pore size.

Table I

Particle Density, Microporosity And Macroporosity For Parent Coals

<u>Coal</u>	<u>Bulk density</u>	<u>Sample wt.</u>	<u>Total volume</u>	<u>Macro-pore vol.</u>	<u>Micro-pore vol.</u>	<u>Particle density</u>	<u>Micro-porosity</u>	<u>Macro-porosity</u>
	(g/cc)	(g)	(cc)	(cc)	(cc)	(g/cc)	(%)	(%)
1451D	0.638	0.2317	0.363	0.15	0.008	1.088	3.76	41.3
1516D	0.673	0.2775	0.252	0.16	0.008	1.101	3.17	38.8

Particle Size and Size Distributions

Cumulative and differential volume distributions corresponding to results from a Laser Microtrac are given in Figures 12-15. The Microtrac measurements are based on projected area of the particles and are converted to volume distributions by the instrument. The shape of the distributions on a log-probability plot resembles that of those obtained by the image analyzer (Ref. 8).

Mean sizes for the image analyzer results are determined as follows:

$$\begin{aligned} \text{Number Mean Size} &= nd / n \\ \text{Volume Mean Size} &= (nd^3 / n)^{1/3} \end{aligned}$$

where n = number of particles and d = particle diameter. The Microtrac instrument determines mean size as that corresponding to the 50% point on a cumulative volume distribution plot. Mean sizes for the parent coal sample are given in Table II.

Table II
Mean Sizes for Parent Coals

Coal	Image Analysis Number mean size	Image Analysis Volume mean size	Laser Microtrac Volume mean size
	(μm)	(μm)	(μm)
1451D	14.9	35.8	39.2
1516D	7.6	27	22.8

The current results (UPitt) can be compared with those obtained at DOE/PETC in 1988 at the time the Vortec separated coal samples were originally distributed (Ref. 9). The following observations are made:

1. The DOE tests show a very high value for dv in the laser Microtrac print out. These are about 100 times the dv values of the UPitt results. The DOE dv values, an indicator of particle concentration, seem excessively high.
2. The DOE results show substantial numbers for particle sizes of 106, 150 and 212 microns. While the UPitt results show zero signal above 88 microns.

From these observations it appears that coal particles might be agglomerating in the DOE tests owing to either very high concentrations or inadequate amounts of dispersant to cope with the high concentrations. To test this hypothesis, the concentration of coal particles in the circulating stream of the Microtrac was increased. Some signal was observed in the 125 and 176 micron size categories. This confirms the possibility of agglomeration in the DOE tests. Image analysis of the samples conducted at the University of Pittsburgh (Ref. 8) also do not show any particles above 100 microns.

Summary

The following observations can be made from the morphological characterization carried out on the parent coal samples:

- True densities of 1.393 g/cc for Pittsburgh #8 coal and 1.511 g/cc for lower Kittanning, Pa coal are seen. This is a reflection of the higher mineral matter content of the 75-106 μm size cut of the lower Kittanning coal relative to Pittsburgh #8 (20% vs. 13.9%).

- Pore volume distributions for the coals give a maximum at about 20 microns which is the macropore size, or the void size with largest volume. Macroporosity of 41.3% for Pittsburgh #8 coal and 38.8% for lower Kittanning, Pa coal result from the similar size distribution and close mean sizes of 35.8 and 27 microns (volume basis) respectively.
- Microporosity of 3.76% for Pittsburgh #8 coal and 3.17% for lower Kittanning, Pa coal are close. So also is the micropore size distribution. This is significant in determining the diffusion rate of gases through the pore structure.
- The similarity of size distributions from Microtrac and the image analyzer confirms the reliability of the use of the latter for size distribution.

6.0 Planned Activities

Work on Task 8, Specific Heat of Char Samples, will be initiated. 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 continue on the measurement of the morphological characteristics of the char samples as a function of extent of reaction.

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LOW TEMPERATURE HEAT CAPACITY OF PSOC 1451D
UTRC FHG MEASUREMENTS AT 1000 C/S

Fig. 1

LOW TEMPERATURE HEAT CAPACITY OF PSOC 1516D
UTRC FHG MEASUREMENTS AT 1000 C/S

Fig. 2

EFFECTIVE HEAT CAPACITY OF PSOC 1451D
UTRC FHG MEASUREMENTS AT 1000 C/S

Fig. 3

1451 CHAR EVOL

CHAR EVOLUTION - PSOC 1451D HVA BITUMINOUS COAL

Fig. 4

EFFECTIVE HEAT CAPACITY OF PSOC 1516D
UTRC FHG MEASUREMENTS AT 1000 C/S

Fig. 5

1516 CHAR EVOL

CHAR EVOLUTION - PSOC 1516D LV BITUMINOUS COAL

Fig. 6

Figure 7 Differential pore volume distribution for coal PSOC 1451d

Figure 8 Differential pore volume distribution for coal PSOC 1516d

Figure 9 Cumulative pore volume distribution for coal PSOC 1451d

Figure 10 Cumulative pore volume distribution for coal PSOC 1516d

Figure 11 Micropore volume distribution for the coals by Medek's method

Figure 12 Log probability volume distribution for coal Psoc 1451d
from Laser Microtrac

Figure13 Log probability volume distribution for coal Psoc 1516d from Laser Microtrac

Figure 14 Differential volume distribution for coal Psoc 1451d
from Laser Microtrac

Figure 15 Differential volume distribution for coal Psoc 1516d from Laser Microtrac

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