SURFACE PROPERTIES OF PHOTO-OXIDIZED BITUMINOUS COALS

**Technical Progress Report for the Period
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by

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ABSTRACT

During this report period, a new whole-seam channel sample (Ohio #4a) was collected and analyzed, together with the Upper Banner and Splash Dam samples obtained last quarter. These additions bring to seven the number of coals obtained for this project and that range in rank from hvCb to mvb.

Polished blocks of each coal containing 3-4 mm wide vitrain bands were prepared for contact angle measurements of fresh and photo-oxidized surfaces. An advancing-drop technique was used to measure contact angle. In this test a droplet of distilled water is grown initially on fresh surfaces and then moved across those irradiated in blue-light for 1, 5, and 10 minutes. The sequence of growth was recorded on video tape, and the change in contact angle measured relative to position at the air/water/surface interface.

Contact angles were measured on five of the coals collected for this study, namely the Illinois #6, Ohio #4a, Lower Kittanning (PSOC-1563), Pittsburgh and Splash Dam seams. Preliminary results show that both coal rank and irradiation time influence surface wettability as measured by contact angle. With one exception, contact angle values decreased and remained low when the droplet advanced into an irradiated area. In most cases, one minute of irradiation resulted in only a slight decrease in contact angle, whereas after 5 and 10 minutes a more significant decrease was observed. The magnitude of change in contact angle values with degree of photo-oxidation decreased as rank increased, such that lower rank bituminous coals show the greatest change and medium volatile coal the least.

In addition to contact angle, the luminescence alteration patterns collected on

various areas during surface irradiation provide some further insight. Alteration patterns for the lower rank coals (hvCb and hvBb) and those of hvAb rank show a dual response in that there is a rapid initial decrease in luminescence intensity followed by an increase. For the lower rank coals, luminescence intensity increases more rapidly and to a much higher level than that achieved by the hvAb coals after 5 and 10 minutes of irradiation. This corresponds well with the relatively greater decrease in contact angle observed for the lower rank coals compared with the more moderate change found for the hvAb coals. Luminescence alteration of the medium volatile coal gave a negative response, in that the initial rapid decrease in intensity is followed by a slow, low rate of decline. This compares well with the fact that contact angles measured for the medium volatile coal reached a minimum after one minute and no further change was observed after 5 and 10 minutes of irradiation.

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PROGRAM OBJECTIVES

The general objectives of this research are 1) to measure the changes in functional group concentration as a function of the duration of photo-oxidation using FTIR and flash pyrolysis GC/MS techniques, 2) to identify the influence of photo-oxidation on coal floatability by measuring contact angles from polished surfaces and powders as well as by microflotation, and 3) to correlate the changes in surface chemistry with measurements of luminescence intensity and alteration. The ultimate objective is to develop new luminescence techniques that will measure quantitatively changes in surface properties as a function of oxidation and relate them to coal cleanability.

PROGRAM OUTLINE

Coal Sampling and Characterization

Vitrinite concentrates and individual particles containing vitrinite will be obtained from eight coals ranging in rank from high volatile B to low volatile bituminous. Every effort will be made to collect fresh coals from the field and to maintain them under conditions that limit their exposure to atmospheric oxygen, to drastic changes in temperature and relative humidity, and exposure to direct sunlight. Moreover, where practical, samples of coal which have been exposed and naturally weathered in outcrop will be collected for comparative studies. Powdered vitrinite concentrates will be generated in a controlled atmosphere glove box under nitrogen, whereas the large particles will have to be sized and polished in air. Vitrinite concentrates will be characterized by proximate, ultimate and petrographic analyses. In some cases, whole seam channel samples may be available from the same vicinity as the blocks removed for concentrates

affording comparisons with whole seam coal samples.

Photo-oxidation and Luminescence Analysis

Luminescence spectral analysis, measurement of emission intensity at a given wavelength (600 nm) and photo-oxidation will be performed initially using a Leitz Orthoplan MPV-II microscope. Basic luminescence properties of the vitrinite from each coal will be measured under a nitrogen atmosphere. Corresponding areas on the same polished surface will be irradiated in air (photo-oxidized) for different periods of time (5, 10 and 15 min) and profiles of the emissions will be measured. Construction of the time-resolved alteration process across the visible frequency range will be necessary to determine the relationship between the change in luminescence and the chemistry associated with photo-oxidation.

Infrared Analysis and Contact Angle Measurements

Once the optimum time intervals of irradiation are determined, separate 1 mm² areas of the polished surface will be photo-oxidized and analyzed by FTIR. We will use a Digilab 60 FTIR with a microspectrophotometric system in the reflectance mode for the FTIR analysis. We expect to have excellent resolution in detecting the changes in functional group chemistry within the top few layers of molecules. Functionalities of interest will be C=O (1710 cm⁻¹), aryl ether (1260 cm⁻¹), alkyl ether (1030-1090 cm⁻¹), C-O (1300-1100 cm⁻¹), C=C (1600-1500 cm⁻¹), δ CH₃ (1400-1320 cm⁻¹), δ CH₂+CH₃ (1480-1400 cm⁻¹), and ar γ CH (920-720 cm⁻¹). The data obtained will be integrated into a set of rate expressions describing the photochemistry for each coal.

These same irradiated areas will be used to measure the sessile-drop contact angle

using a Ramé-Hart Model A-100 contact-angle goniometer. Although the sessile-drop method with water results in slightly more data scattering than captive-bubble measurements, the better control over drop placement afforded by the technique is necessary. Use of pure vitrains ensures that a true rank-related effect will be measured, and that the wide variability introduced into wettability experiments by variation in maceral and mineral composition is avoided.

Pyrolysis GC/MS and Wettability Analysis

Small amounts of powdered coal samples ($\approx 1.0\text{g}$ at -0.25 mm) will be irradiated in air using the same conditions, optical equipment and time intervals as with the polished-surface samples. A small subsample will be prepared for luminescence spectral analysis for comparison with the polished samples; the remainder will be employed for pyrolysis GC/MS and wettability studies.

Pyrolysis GC/MS will be performed using a DuPont 490B GC/MS with a temperature-programmed furnace which fits directly into the injection port. The temperature program we intend to use involves ramping up to 610°C at $5^\circ\text{C}/\text{msec}$ and holding at 610°C for 10 secs; this program compares well with Curie-point pyrolysis methods. The capillary column employed is a low polarity, DB-17 column, which provides excellent resolution of the oxygen-containing molecules. The mass detector employs a magnetic sector mass separator which achieves a resolution of 1 amu at mass 500.

Contact angles will be measured for the powdered vitrinite samples by using the interface partitioning method developed at Penn State. The technique involves placing a

fine coal particle, with its natural irregular surfaces, at the water/air interface. The position of the particle at the interface depends on the particle wettability and its aspect ratio (size and shape). From this analysis contact angles of many naturally shaped particles can be determined. Both fresh and photo-oxidized samples will be tested and, provided that significant differences are found from photo-oxidation, a number of microflotation tests will be conducted.

DESCRIPTION OF TECHNICAL PROGRESS

INTRODUCTION

During this performance period a new sample (Ohio #4a) was collected and analytical results were obtained for this and the whole-seam samples of the Upper Banner (DECS-29) and Splash Dam (DECS-30) coals. This brings to seven the number of coals collected for the project. In addition, coal blocks containing vitrain bands (≈ 3 mm wide) of all samples were prepared into polished sections for photo-oxidation. Using the advancing-drop technique, contact angle measurements were made on five of these samples that were irradiated in blue light for 1, 5 and 10 minutes. The following report summarizes our progress.

COAL SAMPLING, PREPARATION AND ANALYSIS

During this reporting period a new sample, the Ohio #4a of hvBb rank (DECS-33), was collected in July. Along with those coals collected last quarter (1), the Upper Banner (DECS-29, hvAb) and Splash Dam (DECS-30, mvb), analyses have been completed for the whole-seam channel samples. Results for the three coals are presented in Tables 1 - 3. In general, these coals of different rank have similar maceral compositions. Although vitrinite is the dominant maceral, the inertinite is somewhat high compared to most eastern bituminous coals. Also, all of these coals possess some degree of thermoplasticity, even the Upper Banner (Table 1) which was observed to be visibly weathered when inspected under the optical microscope (1). As anticipated by their ranks, the luminescence intensity of the Ohio #4a (Table 3) is considerably higher than that of the Splash Dam seam (Table 2). Also, as shown by duplicate analysis (Alt1, Alt2) in Fig.

Table 1
Analysis of Whole Seam Channel of the hvAb Upper Banner Seam (DECS-29)

	Whole-Seam Channel	Vitrinite Concentrate
<u>Proximate Analysis</u>		
% Moisture, as-received	2.44	Work in Progress
% Ash, dry	6.36	
% Volatile Matter, daf	36.63	
% Fixed Carbon, daf	63.37	
<u>Ultimate Analysis, daf</u>		
% Carbon	86.59	
% Hydrogen	5.53	
% Nitrogen	1.58	
% Total Sulfur	0.63	
% Oxygen (diff.)	5.67	
<u>Forms of Sulfur, dry</u>		
% Pyritic Sulfur	0.09	
% Sulfate Sulfur	0.01	
% Organic Sulfur (diff)	0.50	
<u>Petrography</u>		
Vitrinite, % min.-free	71.1	
Liptinite, % min.-free	6.9	
Inertinite, % min.-free	22.0	
Mean Maximum Vitrinite Reflectance, %	1.00	
Vitrinite Luminescence Intensity in N ₂ (Uranyl Glass=10)	-	
<u>Thermoplastic Properties</u>		
Maximum Fluidity, ddpm	8942	-
Fluid Temperature Range °C	90	-
Free Swelling Index	8	-

Table 2
Analysis of Whole Seam Channel of the mvb Splash Dam Seam (DECS-30)

	Whole-Seam Channel	Vitrinite Concentrate
<u>Proximate Analysis</u>		
% Moisture, as-received	2.06	Work in Progress
% Ash, dry	3.89	
% Volatile Matter, daf	31.35	
% Fixed Carbon, daf	68.65	
<u>Ultimate Analysis, daf</u>		
% Carbon	87.83	
% Hydrogen	5.36	
% Nitrogen	1.57	
% Total Sulfur	0.82	
% Oxygen (diff.)	4.42	
<u>Forms of Sulfur, dry</u>		
% Pyritic Sulfur	0.14	
% Sulfate Sulfur	0.00	
% Organic Sulfur (diff)	0.65	
<u>Petrography</u>		
Vitrinite, % min.-free	76.8	
Liptinite, % min.-free	3.6	
Inertinite, % min.-free	19.6	
Mean Maximum Vitrinite Reflectance, %	1.16	
Vitrinite Luminescence Intensity in N ₂ (Uranyl Glass=10)	1.28	
<u>Thermoplastic Properties</u>		
Maximum Fluidity, ddpm	28188	-
Fluid Temperature Range °C	117	-
Free Swelling Index	8	-

Table 3
Analysis of Whole Seam Channel of the hvBb Ohio #4a, Clarion Seam (DECS-33)

	Whole-Seam Channel	Vitrinite Concentrate
<u>Proximate Analysis</u>		
% Moisture, as-received	5.97	Work in Progress
% Ash, dry	12.02	
% Volatile Matter, daf	48.94	
% Fixed Carbon, daf	51.06	
<u>Ultimate Analysis, daf</u>		
% Carbon	79.01	
% Hydrogen	5.81	
% Nitrogen	1.35	
% Total Sulfur	4.25	
% Oxygen (diff.)	9.58	
<u>Forms of Sulfur, dry</u>		
% Pyritic Sulfur	1.41	
% Sulfate Sulfur	0.01	
% Organic Sulfur (diff)	2.32	
<u>Petrography</u>		
Vitrinite, % min.-free	76.7	
Liptinite, % min.-free	8.3	
Inertinite, % min.-free	15.0	
Mean Maximum Vitrinite Reflectance, %	0.51	
Vitrinite Luminescence Intensity in N ₂ (Uranyl Glass=10)	2.45	
<u>Thermoplastic Properties</u>		
Maximum Fluidity, ddpm	86	-
Fluid Temperature Range °C	75	-
Free Swelling Index	4	-

Fig. 1 - Luminescence Alteration Patterns Whole Seam Channel Sample
Of DECS-33 (Ohio #4a) and DECS-30 (Splash Dam)

1, there is a significant difference in their alteration patterns, i.e. dual alteration compared to negative, respectively. Luminescence intensity and alteration data have not been collected for the Upper Banner seam owing to the observed weathering.

PHOTO-OXIDATION STUDIES

Introduction

With a nearly complete set of coals, individual blocks were selected from each coal sample and prepared into polished blocks. Several polished specimens of each coal were subjected to blue-light irradiation at various time intervals corresponding to short, medium and long term photo-oxidation. Advancing-drop contact angle measurements were performed on five of the coals.

Sample Preparation

The size of each block varied greatly among the coals owing to their particular friability, but for the most part they contained one or more vitrain bands between 3 - 4 mm in height. One surface of each block was polished using a sequence of grinding paper (600 grit) and alumina slurries (0.3 and 0.05 μm polishing compounds). Between each stage the samples were cleaned in an ultrasonic bath containing distilled water. After the final polishing step, the surface was gently cleaned with cotton under a stream of distilled water, dried in a stream of filtered air and placed under vacuum for several hours to remove excess moisture from surface cracks and pores. Samples were then wrapped in a cardboard ring to protect the polished surface and placed in a foil laminate bag, sealed in argon gas and refrigerated until needed.

Irradiation (Photo-Oxidation) of Vitrain Bands

The sequence of irradiation of the vitrain bands was altered from that described in a previous quarterly report (2). In our earlier work, a 500 x 500 μm matrix of 100 μm diameter areas was irradiated in one quadrant defined by orthogonal scribe marks cut into a vitrain band. Although effective, this technique presented some difficulty in precisely locating the photo-oxidized area.

In the modified technique, the light flux was focused into a 200 μm diameter area for surface irradiation. As a guide, a single scribe mark was placed through the vitrain band. Approximately 200 μm from the scribe mark, successive areas of vitrinite were irradiated in a line parallel with the scribe mark until the full height of the vitrain band was traversed. A second line of irradiated areas placed immediately adjacent to the first provided a photo-oxidized area about 400 μm wide for the full height of the vitrain band. This procedure was followed for each period of irradiation, i.e., 1, 5 and 10 minutes. Usually, different vitrain bands in different blocks were irradiated for each time period as it was difficult to locate areas of vitrain free of fractures and inclusions of other macerals or minerals. Also, at regular intervals luminescence alteration patterns were recorded. Following irradiation, the samples were returned to storage in foil laminate bags under argon gas and refrigerated until the contact angles could be measured. Typically, contact angle measurements were made within 48 hours of irradiation.

Contact Angle Measurements

Contact angle studies were carried out using a sessile-drop measurement technique in which a droplet of distilled water was grown or advanced across the polished surface of vitrain. The measurements were performed on fresh and photo-oxidized surfaces. The experimental set-up, given in Fig. 2, contains a high-resolution video camera connected to a microscope to record the profile of a water droplet at the coal/air interface. A Gilmont syringe coupled to an adjustable speed syringe pump was used to grow the water droplet. The droplet was initially placed on an unoxidized area of the coal and allowed to grow across the irradiated area. A schematic representation of the droplet on the surface is given in Fig. 3. The growth of the droplet and the change in the contact angle when it reaches and enters the irradiated area is shown in Fig. 4. The entire sequence of growth is recorded on video tape. Later, the contact angle values are measured at different positions as the droplet advances from the fresh through the photo-oxidized surface.

Results and Discussions

Results from contact angle measurements (Figs. 5 - 9) and from measuring the time-induced alteration of luminescence intensity during irradiation (Figs. 11 - 14) are provided for five of the coals used in this study, i.e., Illinois #6, Ohio #4a, Lower Kittanning (PSOC-1563), Pittsburgh, and Splash Dam seams. Preliminary inspection of both data sets showed that contact angle values were significantly influenced by coal rank as well as irradiation time. Generally, contact angle values were relatively stable when measured through areas that were not irradiated and photo-oxidized. Major differences in

Fig. 2 - Schematic of the contact angle measurement set-up.

1: High-resolution camera; 2: Microscope; 3: Coal sample; 4: Syringe;
5: Syringe pump; 6: Movable stage; 7: Vibration-free table; 8: Light
source

Fig. 3 - Measurement of contact angle at the air/coal/water interface under equilibrium conditions.

γ_{AW} , γ_{CW} and γ_{CA} : air-water, coal-water and coal-air interfacial energies; θ :
contact angle.

Fig. 4 - Measurement of contact angle at the air/coal/water interface (An irradiated patch is shown on the coal surface).

γ_{AW} , γ_{CW} and γ_{CA} : air-water, coal-water and coal-air interfacial energies; θ : contact angle.

Fig. 5 - Advancing Contact Angle Measured on a Polished Surface of Vitrain from the Lower Kittanning Seam (PSOC-1563)

Fig. 6 - Advancing Contact Angle Measured on a Polished Surface of Vitrain from the Pittsburgh Seam (DECS-23)

Fig. 9 - Advancing Contact Angle Measured on a Polished Surface of Vitrain from the Splash Dam Seam (DECS-30)

initial contact angles resulted from measurements made on vitrain bands from different coal blocks, those that are similar were measured on the same band (Figs 5-9). Some minor variability may have resulted from surface preparation (polishing scratches), heterogeneity (presence of other macerals or minerals) or the fact that small non-irradiated areas exist within the photo-oxidized regions. Regardless of the initial contact angle, however, the relative changes resulting from photo-oxidation can be compared and were found to be significant.

In general terms, a decrease in contact angle is measured when the droplet advances into the irradiated area and the value remains lower than the initial value (unoxidized surface) throughout the 400 μm (0.4 mm) area. This decrease was clearly related to irradiation time and to coal rank. For the hvAb Lower Kittanning and Pittsburgh coals (Figs 5 and 6, respectively), one minute of irradiation resulted in a slight decrease (increase for the Lower Kittanning) in contact angle. However, a progressively larger decrease in contact angle was observed between 5 and 10 minutes of irradiation, resulting in a maximum decrease of 5-10 degrees for both coals. A higher level of maximum decrease was observed for the lower rank hvBb Ohio #4a and hvCb Illinois #6 samples (Figs 7 and 8, respectively), but contact angle values fluctuated more within the irradiated area. For the Splash Dam sample (Fig 9), the contact angle reached its minimum value after one minute of irradiation and did not change with longer irradiation times.

Fig 10 uses the maximum change in contact angle (i.e., $\Delta\text{CA} = \text{CA}_{\text{fresh}} - \text{CA}_{\text{min}}$), to illustrate the effect of both rank and irradiation time for all five coals. There is a linear

Fig. 10 - Maximum Change in Contact Angle with Irradiation Time of Five Bituminous Coals

decrease in the change in contact angle with increasing rank resulting from 5 and 10 minute irradiation, with the 10 minute irradiation being significantly greater particularly at lower rank.

Corresponding luminescence alteration patterns for a number of irradiated areas within each time interval and for each coal are given in Figs 11 - 15. Individual patterns identified as Area1, Area2, etc. on the graphs are keyed to maps of the irradiated regions (not shown). These alteration diagrams provide further insight into the contact angle response. For example, alteration of luminescence intensity with time of irradiation for the hvAb coals (Lower Kittanning, Fig 11 and Pittsburgh, Fig 12) are very similar. Both coals show an initial rapid decrease in intensity to some minimum value, followed by a slow, regular increase. After 10 minutes of irradiation, luminescence intensity had not reached the initial value measured at time zero. With the slow recovery there is little difference between the intensity values measured at 5 and 10 minutes of irradiation. This corresponds to the relatively small difference observed in contact angle values for these two times (Figs 5 and 6). For the Ohio #4a (Fig 13) and Illinois #6 (Fig 14) coals, luminescence intensity diminishes rapidly to a minimum and then increases quite rapidly, eventually exceeding the initial value at time zero after 5 and 10 minutes of irradiation. Thus, with a larger difference in luminescence intensity, we expect and in fact do see a comparably greater change in contact angle (Figs 7 and 8) between 1 and 10 minutes compared to the hvAb coals. Also, there is more variability in the apparent rate of increase in luminescence intensity from place to place within individual vitrain bands (Figs 13 & 14). For the Ohio #4a and Illinois #6, contact angles drop sharply then

Fig. 11 - Luminescence Alteration Patterns From Irradiated Areas For PSOC-1563, Lower Kittanning Seam

Fig. 12 - Luminescence Alteration Patterns From Irradiated Areas For DECS-23, Pittsburgh Seam ²²

Fig. 13 - Luminescence Alteration Patterns From Irradiated Areas For DECS-33, Ohio #4a

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Fig. 14 - Luminescence Alteration Patterns From Irradiated Areas For DECS-24, Illinois #6 Seam

Fig. 15 - Luminescence Alteration Patterns From Irradiated Areas For DECS-30, Splash Dam

increase slightly within the irradiated area, suggesting that photo-oxidation of these lower rank vitrains may be more heterogeneous.

In contrast to the coals discussed above, the alteration pattern of the Splash Dam sample (Fig. 15) is negative, i.e., a rapid initial decrease followed by a uniform, slow rate decline. As already noted, the contact angles for this coal decrease by the same amount whether irradiated for 1, 5 or 10 minutes, suggesting that for the medium volatile Splash Dam that photo-oxidation is rapid and, perhaps that changes in surface wettability are less severe compared to lower rank bituminous coals.

In a previous report (3), results from STXM C-NEXAFS analysis of the hvAb Pittsburgh and subbit Wyodak coals showed that increasing irradiation time does lead to photo-oxidation of vitrinite by increasing the concentration of OH and C=O groups attached to aromatic rings. This work also showed that the concentration of oxygen-bearing species decreased with increasing rank. These observations are in accord with results from contact angle and luminescence alteration. It appears that photo-oxidation of vitrain is influenced by coal rank and irradiation time. Low-rank bituminous coals become more hydrophilic with increased photo-oxidation, but perhaps are more variable from place to place within vitrain. Intermediate rank, hvAb coals although effected somewhat less, also tend to become progressively more hydrophilic with exposure time. The medium volatile coal appeared to reach a maximum hydrophilicity with minimal exposure to blue light, suggesting that it has relatively few sites that can be photo-oxidized, but they are highly susceptible.

All irradiated samples have been returned to refrigerated storage under argon gas

until a new reflectance-mode FTIR is installed and operating. Enquiries will be made to other institutions with the same facilities in case our system is further delayed.

POWDERED VITRAIN AND FILM FLOTATION

The effort needed to photo-oxidize powdered vitrain using the optical microscope was demonstrated to be prohibitive in the last quarterly report (1). Consequently, we have begun to investigate the use of an alternative means that will employ a long wavelength (366 nm) ultraviolet lamp. This wavelength is slightly lower than that used in the optical microscope (390 - 490 nm) and possibly considerable lower in luminous energy. Before we consider alternative techniques, the emission of this lamp will be tested on a series of polished surfaces of vitrinite to establish the change in fluorescence intensity with time and condition of irradiation. Tests on polished blocks of the Pittsburgh, Illinois #6 and Splash Dam seams will begin next quarter.

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