**MONITORING POWER PLANT EFFICIENCY USING THE MICROWAVE-EXCITED
PHOTOACOUSTIC EFFECT TO MEASURE UNBURNED CARBON**

Quarterly Technical Progress Report

Reporting Period Start Date: July 1, 2004

Reporting Period End Date: September 30, 2004

Principal Author(s):

Robert C. Brown, Robert J. Weber, and Jeff Sweterlitsch

Date Report Issued:

October 2004

DOE Award Number:

DE-FC22-01NT41220

Submitted By:

Center for Sustainable Environmental Technologies
Iowa State University
285 Metals Development Bldg.
Ames, IA 50011-3020

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

ABSTRACT

Three test instruments are being evaluated to determine the feasibility of using photoacoustic technology for measuring unburned carbon in fly ash. The first test instrument is a single microwave frequency system previously constructed to measure photoacoustic signals in an off-line configuration. A second off-line instrument was constructed based in part on lessons learned with the first instrument, but which also expands the capabilities of the first instrument. Improvements include a control loop to allow more constant microwave power output and an ability to operate over a range of microwave frequencies. The third instrument, the on-line version of the fly ash monitor, has been designed, constructed, and initial efficiency tests have been conducted on the monitor's electrical components.

Off-line photoacoustic microwave spectra of fly ash and coal were collected and analyzed, and the spectra demonstrated a linear correlation between the photoacoustic response and the carbon content in either fly ash or coal.

Modifications were made to the on-line fly ash monitor to incorporate a dual-accelerometer system that would provide active noise control. Several experiments were conducted with flowing and non-flowing fly ash samples.

Keywords: fly ash, carbon monitor, unburned carbon, boiler instrumentation

EXECUTIVE SUMMARY

The objective of this project is to explore the use of the microwave-excited photoacoustic (MEPA) effect for quantitative analysis of granular and powdered materials, and to develop an on-line carbon-in-ash monitor. The three steps to achieving this objective include: 1) designing a single microwave frequency, off-line instrument as part of proof-of-concept evaluations; 2) construction of a microwave spectrometer based on MEPA used to evaluate a variety of industrial important powders, including fly ash and pulverized coal; and 3) design and construct an on-line monitor that used MEPA technology. The instruments for steps 1 and 2 have already been completed, tested, and evaluated, and the research is currently focused on step 3, the on-line fly ash monitor.

Off-line photoacoustic spectra were generated for six different fly ashes and eight different coal samples. The spectral region was 500 – 1000 MHz, with a resolution of 50 MHz. The dominate features of the spectra are the photoacoustic (PAS) response increases with increased carbon content of the fly ash (and coal), and it also increases with increasing microwave frequency. Additional features present in the spectra could be used to identify or quantify unique constituents in the fly ash other than carbon. Samples of these fly ashes have been sent to the Minnesota Valley Testing Laboratory for mineral analysis, and results are pending.

The off-line photoacoustic data can also be used to predict the carbon content of both the fly ash samples and the coal samples. For fly ash, a linear relationship between the photoacoustic response and the carbon content of fly ash with good correlation ($R^2=0.975$). For coal, the linear correlation was less strong, with an $R^2=0.853$.

The on-line fly ash monitor was modified to accommodate two aluminum diaphragms to provide some active noise control: two accelerometers are subject to random vibrations in the laboratory, as well as the movement of the fly ash as it flows pass the diaphragms, but only the fly ash in the vicinity of the test accelerometer would generate the microwave-induced thermo-elastic waves that would be detected by this accelerometer. Several experiments were conducted with the on-line fly ash monitor to determine the effectiveness of the on-line monitor to quantify the carbon content of six different fly ashes.

TABLE OF CONTENTS

DISCLAIMER.....	i
ABSTRACT.....	ii
EXECUTIVE SUMMARY	iii
INTRODUCTION.....	1
EXPERIMENTAL	1
Off-line photoacoustic experiments	1
On-line thermo-elastic experiments	2
RESULTS AND DISCUSSION	5
Off-line photoacoustic results	5
On-line thermo-elastic results.....	10
CONCLUSIONS AND FUTURE WORK	13

INTRODUCTION

The objective of this project is to explore the use of the microwave-excited photoacoustic (MEPA) effect for quantitative analysis of granular and powdered materials. The focal point of the research centers on the measurement of unburned carbon in fly ash, an important parameter in the electric utility industry used to determine plant efficiencies. The culmination of this project will be an on-line carbon-in-ash monitor for coal-fired power plants. However, evaluations also will be made on other powdered solids, particularly coal.

The approach to this project includes work with three MEPA instruments. The first instrument is a single microwave frequency, off-line instrument built at Iowa State University as part of proof-of-concept evaluations. It is being used to evaluate precision and accuracy of the MEPA technique. The second instrument is being constructed as a microwave spectrometer based on MEPA. It will be used to evaluate a variety of industrial important powders, including fly ash and pulverized coal. The final instrument will be built based on the results of work with the previous two instruments and will be used as an on-line monitor of unburned carbon in fly ash.

EXPERIMENTAL

Off-line photoacoustic experiments

Off-line photoacoustic spectra were generated for six different fly ashes and eight different coal samples. A Hewlett-Packard 8648C Signal Generator was used to generate the microwaves at frequencies of 500 – 1000 MHz. This signal generator temporarily replaced the Mini-Circuits ZOS-1025 Microwave Oscillator because the signal generator could generate the variable microwave frequencies with more control and ease.

The photoacoustic microwave spectra of the fly ash samples and coal samples were created by collecting individual photoacoustic signals for microwave frequencies between 500 – 1000 MHz, with a resolution of 50 MHz, or 11 total microwave frequencies. The data collection parameters of the SRS Lock-in Amplifier were set to a sensitivity of 200 μ V, a 1 second pre-time constant, normal dynamic reserves, and the band-pass, line filter, and line-X2 filters were used. The amplitude modulation of the microwaves was 20 Hz.

An individual sample was prepared and loaded into the off-line fly ash monitor, and the signal was allowed to stabilize for two minutes before microwaves were introduced into the off-line monitor. The microwaves were then introduced into the fly ash monitor, the signal stabilized, and data was collected for two minutes, which for the described data collection parameters refers to 88-90 data points. After collecting data for one microwave frequency, the microwave frequency was adjusted with the signal generator. The same sample remained in the cell for all 11 microwave frequencies.

On-line thermo-elastic experiments

The on-line fly ash monitor was modified to accommodate two aluminum diaphragms, each with an Applied MEMS SF1500-L accelerometer mounted on the surface of the diaphragm with double-sided Kapton® Tape. The second accelerometer is located upstream of the original accelerometer, sufficiently far enough away so that the microwaves do not induce a thermo-elastic effect in the fly ash in the vicinity of the second accelerometer. The purpose of installing a second diaphragm and accelerometer is to provide some active noise control: both accelerometers are subject to random vibrations in the laboratory, as well as the movement of the fly ash as it flows pass the diaphragms, but only the fly ash in the vicinity of the original accelerometer would generate the microwave-induced thermo-elastic waves that would be

detected by this accelerometer. The signal outputs from both accelerometers are fed into a instrumentation-quality differential op-amp, which is designed to cancel out all signals except those at the amplitude modulation frequency of the microwaves. Figure 1 shows the two accelerometers mounted on individual diaphragms, and between the accelerometers is mounted an electrical box where the power supply for accelerometers and the op-amps are contained and controlled, and also the differential op-amp circuitry is located. Also shown are the power supply toggle switches for the accelerometers and op-amps, the battery power supply, and toggle switches that control whether the desired signal comes from an individual accelerometer or from the differential op-amp. Unless otherwise noted, the top accelerometer is referred to as the “control” accelerometer, and the bottom accelerometer is referred to as the “test” accelerometer.

Figure 1. On-line fly ash monitor showing with dual accelerometers and electrical control box.

The individual responses of the accelerometers are not identical. The control accelerometer has a response of 1.136 V/g, and the test accelerometer has a response of 1.171 V/g. The differential op-amp was designed to incorporate two potentiometers that can be used to adjust the gain for each accelerometer to account for the difference in response. The goal was to obtain an overall gain close to 2, and the actual gain for the control accelerometer was tuned to 1.944, and the gain for the test accelerometer was tuned to 1.886. Figure 2 is a detail of the electrical box that shows the op-amp circuitry with the two, blue potentiometers in the middle of the circuit board.

Figure 2. Differential operational amplifier circuit board detail.

For the on-line measurements, the data collection parameters of the SRS Lock-in Amplifier were set to a sensitivity of 20 μV , a 10 second pre-time constant, normal dynamic reserves, and the band-pass, line filter, and line-X2 filters were used. The amplitude modulation of the microwaves was 1000 Hz.

RESULTS AND DISCUSSION

Off-line photoacoustic results

Photoacoustic microwave spectra were collected for six different fly ashes and eight different coals. Table 1 presents the types of fly ashes and coals. Concentrations are listed “as-received”.

Table 1. Summary of fly ashes and coals used for off-line photoacoustic measurements.

Sample ID	Description
Fly ash #1	0.4% Carbon content from LOI measurements
Fly ash #2	0.75% Carbon content from LOI measurements
Fly ash #3	1.3% Carbon content from LOI measurements
Fly ash #4	2.5% Carbon content from LOI measurements
Fly ash #5	2.74% Carbon content from LOI measurements
Fly ash #6	4.5% Carbon content from LOI measurements
DECS-7	High-volatile C bituminous coal, 41.11% fixed carbon
DECS-11	Lignite A coal, 22.89% fixed carbon
DECS-18	High-volatile B bituminous coal, 43.40% fixed carbon
DECS-20	High-volatile A bituminous coal, 55.41% fixed carbon
DECS-23	High-volatile A bituminous coal, 50.12% fixed carbon
DECS-26	Subbituminous B coal, 35.06% fixed carbon
DECS-27	Subbituminous A coal, 41.46% fixed carbon
DECS-30	Medium-volatile bituminous coal, 64.62% fixed carbon

Figure 3 presents the off-line photoacoustic microwave spectra for the fly ashes. The two dominate features of the spectra shown in Figure 3 are the photoacoustic (PAS) response increases with increased carbon content of the fly ash, and the PAS response also increases with increasing microwave frequency, which would be expected because the fly ash is exposed to higher intensity energy, so the fly ash is able to absorb more energy. Theoretically, the energy absorption should increase at the same ratio as the microwave frequency. At 500 MHz, 4.5% fly ash absorbs 80 μ V, but at 1000 MHz, 4.5% fly ash absorbs 140 μ V, which is a 1.75:1 ratio instead of a 2:1 ratio. The discrepancy is most likely due to the design limitations of the off-line fly ash monitor. The ratio of 1.75:1 is consistent, however, for all six fly ashes.

Figure 3. Microwave photoacoustic spectra for fly ashes of six different carbon contents.

Figure 4. Normalized photoacoustic microwave spectra of different fly ashes.

Figure 4 also presents the photoacoustic microwave spectra for the same fly ashes, but the spectra have been normalized, forcing the local minimum of each spectrum to a value of

zero, and forcing the local maximum of each spectrum to a value of unity. For these six spectra, the local minima occurred at 550 MHz, and the local maxima occurred at 1000 MHz. The objective for normalizing the spectra in this manner cancels out the variation in baseline levels, and greater emphasizes the curvature of each spectrum, which could be used to identify or quantify unique constituents in the fly ash other than carbon. Samples of these fly ashes have been sent to the Minnesota Valley Testing Laboratory for mineral analysis, and the results are pending.

Figure 5 presents the off-line photoacoustic microwave spectra of the eight coal samples. Again, the dominate features are that the PAS response generally increases with increasing carbon content, and with increasing microwave frequency. Figure 6 presents the same spectra, normalized in the same manner as the fly ash spectra presented in Figure 4.

Figure 5. Photoacoustic microwave spectra of coal samples.

Figure 6. Normalized photoacoustic microwave spectra of coal samples.

In addition to collecting microwave spectra of fly ash and coal, which could be used to identify individual constituents within the sample, the photoacoustic data can also be used to predict the carbon content of both the fly ash samples and the coal samples. For the fly ash samples, a signal frequency was selected that would generate a photoacoustic response in the fly ash that would be proportional in intensity with the carbon content in the fly ash. Data at 1000 MHz was used for this prediction capability. Figure 7 presents the linear relationship between the photoacoustic response and the carbon content of fly ash with good correlation ($R^2=0.975$).

Figure 7. Photoacoustic response as a function of carbon content in fly ash.

Initial attempts to generate a prediction curve for the fixed carbon content of coal based on a signal microwave frequency for did not generate good correlations between the photoacoustic response and the carbon content; a linear fit produced only a correlation of $R^2=0.501$. Instead, the data that comprised each spectrum was analyzed collectively using GRAMS/AI (Thermo Electron Corp.) spectroscopic software with the PLS (partial least-squares) chemometrics tool. Multiple linear regressions were performed on the spectra, and Figure 8 is a predicted-vs.-actual concentration plot for the fixed carbon content of the coal samples based on the photoacoustic microwave spectra. Ideally, all the data points would lie on the diagonal line, which has a slope of unity. Although the correlation is not as good for coal as it is for fly ash, additional coal samples should be analyzed to confirm or deny that there is a correlation between the microwave spectra of coal and the fixed carbon content of coal.

Figure 8. Predicted vs. Actual Fixed Carbon Content of Coal.

On-line thermo-elastic results

Twelve different experiments were conducted with the on-line fly ash monitor to determine the effectiveness of the on-line monitor to quantify the carbon content of six different fly ashes (See Table 1 for a list of the fly ashes used). The experiments conducted on each fly ash are listed in Table 2.

Table 2. Summary of on-line fly ash monitor experiments for each fly ash.

Experiment	Signal Output	1000 MHz Microwaves Modulated at 1000 Hz	Flowing or Non-Flowing
1	Test Accelerometer	Power off (baseline)	Non-Flowing Fly Ash
2	Test Accelerometer	Power on	Non-Flowing Fly Ash
3	Control Accelerometer	Power off (baseline)	Non-Flowing Fly Ash
4	Control Accelerometer	Power on	Non-Flowing Fly Ash
5	Differential Op-Amp	Power off (baseline)	Non-Flowing Fly Ash
6	Differential Op-Amp	Power on	Non-Flowing Fly Ash
7	Test Accelerometer	Power off (baseline)	Flowing Fly Ash
8	Test Accelerometer	Power on	Flowing Fly Ash
9	Control Accelerometer	Power off (baseline)	Flowing Fly Ash
10	Control Accelerometer	Power on	Flowing Fly Ash
11	Differential Op-Amp	Power off (baseline)	Flowing Fly Ash
12	Differential Op-Amp	Power on	Flowing Fly Ash

On-line fly ash experiments were conducted over a seven-week period from July 13, 2004 – August 31, 2004, to check for repeatability of the experiments. All data shown in the following figures are averages of up to five trials conducted during this time period.

Figure 9 present the results from Experiments #1 and #2, and data compares the effect of the microwaves on the fly ash when the fly ash is not flowing, and only data from the test accelerometer is being recorded (the differential op-amp circuitry was by-passed). The data in Figure 9 demonstrate that the fly ash does exhibit a thermo-elastic effect due to the microwaves, but unfortunately there is no apparent correlation between the intensity of the thermo-elastic effect and the carbon content of the fly ash.

Figure 10 present the results from Experiments #7 and #8, and the data compares the effect of the microwaves on the fly ash when the fly ash is flowing. Again, only data from the test accelerometer is shown here. Both signals are higher than those shown in Figure 9, most likely due to the movement caused by the flowing fly ash particles, and what appears to be a correlation for low-carbon content fly ash disappears for higher carbon content fly ashes.

Figure 11 present the results from Experiments #11 and #12, and the data compares the baseline data of flowing fly ash when using the differential op-amp circuitry to actively cancel out background noise and vibrations due to the flowing fly ash particles. It should be noted that the op-amp provides a signal gain of approximately 1.9, which would explain the greater signal strength as compared to the data shown in Figure 10. But there is no apparent correlation between carbon content and the thermo-elastic response induced by the microwaves and detected by the accelerometers.

Figure 9. Thermo-elastic response of non-flowing fly ash comparing the baseline (no-microwave) signal and the active signal of the test accelerometer.

Figure 10. Thermo-elastic response of flowing fly ash comparing the baseline (no microwaves) signal with the active signal of the test accelerometer.

Figure 11. Thermo-elastic response of flowing fly ash employing active noise control, comparing baselin (no microwaves) and the active signal from the differential op-amp.

CONCLUSIONS AND FUTURE WORK

The off-line photoacoustic fly ash monitor successfully demonstrates that the carbon content of unburned fly-ash can be quantified by measuring the microwave-induced photoacoustic effect in the fly ash. Additional tests will be conducted to determine the repeatability of these results. This technique may also apply to coal samples, although further investigation is also required.

The off-line photoacoustic fly ash monitor also can be used to generate microwave spectra of fly ash and coal. Additional experiments will be conducted on pure samples of specific compounds that are present in fly ash to determine if there are characteristic spectral features of these pure samples that may be present in the fly ash spectrum. The primary species of interest include iron (III) oxide, magnesium oxide, calcium oxide, silicon dioxide, and aluminum trioxide.

Attempt to quantify the unburned carbon content in fly ash using the on-line thermo-elastic technique has been unsuccessful. Possibly reasons for the lack of a correlation may include bulk properties of the fly ash, the quality of the mechanical coupling of the accelerometers with the diaphragms, dampening effects due to the weight of lead wires, or the signal may simply be too small to be reliable. The on-line fly ash monitor can be retro-fitted with a microphone, and experiments are pending to determine if this is a viable alternative.