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

Quarterly Technical Progress Report

Reporting Period Start Date: July 1, 2002

Reporting Period End Date: September 30, 2002

Principal Author(s):

Robert C. Brown, Robert J. Weber, and Andrew A. Suby

Date Report Issued:

October 2002

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 photo-acoustic technology for measuring unburned carbon in fly ash. The first test instrument is a single microwave frequency system previously constructed to measure photo-acoustic signals in an off-line configuration. This system was assembled and used to test parameters thought important to photo-acoustic signal output. A standard modulation frequency was chosen based upon signal to noise data gained from experimentation. Sample heterogeneity was tested and found not to be influential. Further testing showed that sample compression and photo-acoustic volume do affect photo-acoustic signal. Testing in the fourth quarter focused on signal repeatability, linearity, the effects of ultrasonic shaking, and sample cup variations.

Simultaneously, a second instrument is being constructed based in part on lessons learned with the first instrument, but also expands the capabilities of the first instrument. The power amplifiers for this second instrument were recently completed and tested. Improvements were made to the current leveling loop, which will stabilize the microwave power. Other efforts were spent generating a magnetic field or electric field source for the photo-acoustic effect. The intent of this effort is to be able to discriminate between magnetic contaminants such as iron and non-magnetic contaminants such as carbon. A short coaxial test fixture was fabricated and tested showing the promise of another microwave based test method for determining carbon content in fly ash. The third instrument will be designed based on the experiences of the first two instruments and will operate in an on-line carbon-in-ash monitoring system for coal-fired power plants.

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

TABLE OF CONTENTS

DISCLAIMER	i
ABSTRACT	ii
TABLE OF CONTENTS	iii
INTRODUCTION	1
EXPERIMENTAL	1
The Single Microwave Frequency, Off-Line Instrument	1
The Microwave Spectrometer	2
RESULTS AND DISCUSSION	7
CONCLUSION	10
APPENDIX A	11

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 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

The Single Microwave Frequency, Off-Line Instrument

Testing was continued to determine parameters that affect photo-acoustic measurement accuracy and precision. Variables examined (to date) which may influence the photo-acoustic signal include: sample heterogeneity, sample bulk density, sample compression, sample moisture content, modulation frequency, signal to noise ratio, and photoacoustic volume. Ambient temperature and humidity were also examined.

Testing continued with fly ash samples obtained from Duquesne/Elrama boiler 3B located in Pennsylvania, as well as other fly ash used in previous experiments and some carbon black/dolomite mixtures. Upon review of test procedures and data taken last quarter, tests were repeated for sample moisture content, compression, and ambient temperature and humidity to determine repeatability of the instrument. Several tests were also run to determine the linearity of the % carbon signal. Test parameters for a representative linearity test using the Duquesne/Elrama fly ashes are outlined in Appendix A. Tests to determine possible variations in signal due to differences in the sample cups were conducted as well as tests to see if ultrasonic shaking would improve repeatability.

The Microwave Spectrometer

The power amplifiers for 500 MHz to 10 GHz were completed and tested during this semester. As indicated in the last quarterly report, the configuration of the current leveling loop was improved over the configuration originally planned. Figure 1 shows the current configuration of the power source.

Concurrently, two power source systems have been assembled this quarter. The first works from 0.5 to 2 GHz. The second works from 4 to 10 GHz.

Figure 1: Leveled amplifier system

The second part of the spectrometer effort during the last quarter concerned generating a magnetic field or electric field source for the photo-acoustic effect. The intent of this system is to be able to discriminate between magnetic contaminants such as iron and non-magnetic contaminants such as carbon. Our first intent was to use various impedance level transmission lines to generate a different magnetic to electric field ratio in the region of the sample. However, producing a different ratio of electric to magnetic fields can only be done by introducing standing waves, since the ratio of electric to magnetic fields in a single TEM traveling wave is determined by material quantities of the medium around a transmission line. By the introduction of a controlled standing wave, one is able to introduce a region of predominately magnetic field or a region of predominately electric field in the region of the sample. As shown in Figure 2, by moving a sliding short on the output of the test chamber, the position of the minimum electric field (maximum of magnetic field) can be changed in the chamber. This is somewhat different

from the standing wave that exists with a cavity resonator. Much higher effective field levels are available within a cavity resonator. However, there are multiple reflections within a cavity resonator and the magnitude of the field that results is very dependent on the number of reflections and on the loss within the sample medium. In the technique used here, the circulator (three port circuit element shown in Figure 3) prevents re-reflection resulting in only one reflection and therefore the field magnitude is much less dependent on the loss within the chamber. This technique will be used in the next quarter to attempt to quantify the difference between a magnetic field photo-acoustic effect and an electric field photo-acoustic effect. The earlier proposed setup using different characteristic impedance transmission lines and impedance transformers will not be used.

Figure 2: Electric or magnetic field measurements depend on the position of the sliding short.

In order to measure the carbon content of fly ash using a separate microwave technique, a short coaxial test fixture was fabricated and tested. A cross section of the fixture is shown in Figure RJW-3. The uniform cylindrical coaxial line is fill with the material

Figure 3: Phase shift dielectric measurement cell.

under test. The phase shift through such a test fixture is easily measured using microwave network analyzers. The time delay through the test fixture is easily determined from the phase shift of the fixture. If the phase shift is a linear function of frequency, this indicates that there are no atomic bond resonances over the frequencies of measurement. Various fly ash samples were measured from 300 kHz to 6 GHz. Over that wide frequency range, the phase shift was very linear and therefore the time delay over for the fixture was easily obtained. Assuming that the velocity of propagation for the material in the fixture is due to relative dielectric constant only, the dielectric constant of the material can also be easily obtained. Figure 4 shows a measured dielectric constant of fly ash material versus carbon content. This same technique has direct applicability to measuring the dielectric constant of other powders or pulverized material such as coal. One sample of fly ash with 43% carbon was also measured. That data did not fit this curve. There may be additional material in that sample contributing to the total dielectric constant. However, this method looks promising for quantifying the carbon content of fly ash using phase shift. Assuming a non-magnetic material, the dielectric constant is determined by the following equation.

$$\epsilon_r = \left(\frac{1}{360} \frac{\phi}{f} \frac{c}{len} \right)^2$$

Figure 4: Dielectric versus carbon content.

A typical phase shift and loss curve versus frequency is shown in Figure 5. Notice the linearity of the phase versus frequency curve. The measured microwave parameters are normalized to the characteristic impedance of the line as filled with the material.

Figure 5: Loss and phase curve for forty-three percent carbon fly ash

RESULTS AND DISCUSSION

The results for the linearity test are shown in Figure 6. The data set shows poor linearity due to the signals recorded for the fly ash reported to contain 14.49% carbon as determined by the Unburned Carbon methodology employed in this study. Since photoacoustic theory suggests that under these conditions photoacoustic signal should be proportional and linear to the concentration of the absorbing material (carbon), this result was unexpected.

Figure 6. Signal Measured for Various 3B Boiler Flyashes vs. % Carbon Using the Unburned Carbon (UC) Methodology (Test XVI: Linearity) 2/7/02

The test was repeated and similar results were obtained. One possible explanation for the poor linearity is that the fly ash reported to contain 14.49% carbon may have other absorbing materials (such as potentially iron oxides) causing the non-linear measurements to be obtained. These fly ash samples have been sent in to have Total Organic Carbon (TOC) and ash analysis tests performed on them. The results are pending.

Further testing was done with carbon black and dolomite mixtures, as well as with fly ash obtained from pulverized coal and stoker type combustors. The results are shown in Figures 7

and 8 respectively. These tests were repeated and the results showed larger than expected variations. Possible reasons for the carbon black/dolomite mixture signal variation could be due to the large bulk density differences between the dolomite and the carbon black. Significant settling could be occurring causing large variations in the thermal sampling depth or layer that is thought to generate the photoacoustic signal. This possibility will be further explored in the next quarter.

In light of these larger than expected variations, tests were performed to determine the influences of using different sample holders between tests. Statistical methods applied to the results suggest that there is some variation between signals gained by different sample holders, which may explain variations in lower carbon content signals. The influence of the sample holders should decrease as sample carbon content, and therefore resulting signal strength, increases. Variations in signals gained from high carbon content samples should not be explained by variations due to the sample holder, unless the sample holder is not consistently sealed from test to test. To check for this, one of the cups was selected and tested as is, and then tested with an o-ring to better insure the seal. There was no statistical difference between the two sets of measurements.

Figure 7. Signal Measured for % Carbon Black (Test XVIC: Linearity, Dried, Similarity) 5/30/02

Figure 8. Signal Measured for Various Waller Flyashes vs. % Carbon Using the Total Organic Carbon (TOC) Methodology (Test XVIB: Linearity, Dried) 3/12/02

CONCLUSION

The Duquesne/Elrama boiler 3B fly ash samples need further review to determine if the reported carbon concentrations are correct or whether there are other possible absorbing materials (such as potentially iron oxides) causing non-linear measurements to be obtained. Efforts were made to determine if the experimental methods or equipment used could be the cause of any unexpected variations. Tests showed sample holder variations that may affect low carbon content measurements, but could not explain higher content variations. Sample holder sealing tests showed no difference in signal when efforts were made to better seal the holder with an o-ring. These future tests will be performed in the recently constructed MEPA spectrometer, since it will provide more stable power than the present test rig.

The power amplifiers for the spectrometer were completed and tested. Improvements were made to the current leveling loop, which will stabilize the microwave power. A magnetic field or electric field source for the photo-acoustic effect was constructed. This should allow discrimination between magnetic constituents such as iron and non-magnetic constituents such as carbon. A short coaxial test fixture was fabricated and tested showing the promise of another microwave based test method for determining carbon content in fly ash.

For the next quarter, efforts will focus on 1) inserting the leveled output power source into the setup, 2) performing photo-acoustic measurements near 1 GHz with predominantly electric fields, 3) performing photo-acoustic measurements near 1 GHz with predominantly magnetic fields, and 4) assembling and beginning initial testing on a 2 GHz to 10 GHz test chamber.

APPENDIX A

SYSTEM TEST SETUP

GENERAL INFORMATION

VERSION: 1.0Principle Investigator: Brown (Suby), WeberDate: 1/31/02Test Group: NETL (DOE)Phone: (515) 382-9006Test Name: TestXVIE-mail: asuby@iastate.eduExpected duration of test: 3 hoursProgram: Lock-in2.bas, PAS.xlsData: ☒ Public ☐ PrivateDescription of test: Linearity.-Select 6 fly ashes of different carbon content from the 3B boiler.***Perform the following procedures 3 times for each flyash:-Weigh and record the weight of the empty sample holder.-Fill the sample holder with sample and compress the sample as described in the produres entitled "Filling the Sample Holder" (SampleSOP.doc) and "Compressing a Fly ash Sample" (CompSampSOP.doc).-Measure and record PA volume (depth) and weigh and record the holder and sample.-Measure PA signal, and dump sample back into original sample bottle.-Clean sample holder throughly with compressed air.Objective of test: To assemble a PA signal vs. carbon curve and determine linearityVariables under consideration: Carbon ContentVariables to be held constant: PA Volume, Sample Temp and Humidity (assumed), Ambient Temp and Humidity (assumed), Modulation Frequency

SPECIAL REQUIREMENTS

SYSTEM SETUP

DATA COLLECTED BY: NL

SAMPLE INFORMATION:

Sample Designation: See Notes:

Sample Preparation:

- ☐ "As Received" (no preparation)
☒ Compressed
☐ Ground
☐ Dried

Notes:

Recommend the following fly ash: 21960AT, 22332AT, 22339AT, 22342AT, 22360AT, and 22368AT

Deliverables:

1) Plot PA signal vs carbon content

SYSTEM INFORMATION:

Modulation Frequency: 20

Raw Data File Naming Convention:

1) Refer to the Document Entitled "Raw Data File Naming Convention for the Lock-in Data Acquisition System" (DOSDataCon.doc)

Notes:

1) Refer to the system Standard Operating Procedures (SystemSOP.doc)