**SURFACE AREA, VOLUME, MASS, AND DENSITY DISTRIBUTIONS FOR SIZED
BIOMASS PARTICLES**

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Surface Area, Volume, Mass, and Density Distributions for Sized Biomass Particles

ABSTRACT

This semi-annual technical progress report describes work performed at Morehouse College under DOE Grant No. DE-FC26-04NT42130 during the period January 01, 2005 to June 30, 2005 which covers the second six months of the project. Presently work is in progress to characterize surface area, volume, mass, and density distributions for sized biomass particles. During this reporting period, Morehouse has completed testing of the gravimetric technique measurement system that was set up in the last reporting period, and a preliminary result for the mean particle mass of the biomass sample tested was obtained. Simultaneously, REM, our subcontractor, has completed calibration of the video based imaging systems and diode-array imaging system that are employed to characterize shape and mass for individual biomass particles. Subsequently, a few actual biomass particles were caught in the electrodynamic balance (EDB), raw data for shape and mass collected and analyzed, and the shape measurement system was found to be working satisfactorily. Preliminary results of the mean mass and the shape data obtained in this reporting period are very encouraging, and more data collection is in progress.

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INTRODUCTION

The term biomass refers to material of terrestrial plant origin. Hardwoods, softwoods, wheat straw, corn kernel, cassava roots, sugar cane bagasse, and coconut shells are some of the largely available agricultural biomass materials. Wood is one of the most important biomass materials that could potentially be diverted to other uses. The amount of biomass produced has been estimated to be 170 billion tons per year, of which about 70% is from forests [1]. An estimate of the amount of biomass available in the U.S. for conversion to fuels or chemicals is 2 billion tons per year [2]. The conversion of 20% of this material provides an energy equivalent of 6.5×10^{15} BTU, roughly 10% of the U.S. annual energy needs [1].

The political (indigenous supply) and environmental (low sulfur, no net CO₂, biodegradable) benefits of using biomass will continue to provide impetus to the development of co-firing coal with biomass feed stocks. Co-firing of biomass and coal has been identified as a promising way of reducing net CO₂ emissions with minimum modifications in the existing technologies. In fact, some developed countries, e.g., Denmark, have already mandated the use of coal biomass blends in all coal fired boilers. Coal and biomass are certain to remain a primary source of energy for at least several decades, and this is why a great need exists to develop modern combustion systems characterized by high carbon utilization (combustion efficiency) and low emission of pollutants (SO₂, NO_x, N₂O, air toxics, etc.). One of the most important factors affecting the performance of utility boilers is fuel type. In some cases, blending biomass with coal may solve a pollution compliance problem or an operational problem or provide an economic benefit.

Coal and biomass particles are irregular in shape. Early attempts to characterize shape relied primarily on sieve analyses to classify particle "size." This approach was subsequently augmented by microscopic measurements and sizing methods based on sedimentation rates in fluids. These methods however, give no indication of the inclination of the axes of the body with respect to three coordinates, the type of geometrical shape, the volume or the surface area [3]. Heywood [4] dealt with this issue by defining shape-dependent coefficients in his analysis of volume and surface area. These coefficients are functions of the proportions of the particle, i.e., the relative values of breadth (B), thickness (T), and length (L). These are obtained by resting the particle in its most stable position. Breadth, B, is defined as the minimal distance between two parallel tangents to the profile or outline of the particle; length, L, is defined similarly but taken at right angles to the breadth; and thickness T, is the height normal to the resting plane.

Detailed information on shape, drag, volume, density, and surface area is needed to improve our understanding of transport phenomena of irregular particles. From a fluid mechanics standpoint, a data base on various shapes of particulates would enhance the current ability to design and analyze feeder systems, cyclones, fluidized beds, and particulate separation systems. From a heat and mass transfer perspective, particle mass and shape are important considerations. In general, to simplify the analysis, heat transfer calculations are performed assuming particles to be spheres. Several studies have been published in recent years where this approach has been used and temperature measurements have been compared with model predictions [5-7]. In each of these studies, however,

large empirical corrections were required in order to match model predictions with measurements. Maloney and coworkers [5] concluded that these corrections were necessary primarily because of the shape and density, and thermal property assumptions applied in the heat transfer analysis.

Energy absorption and emission mechanisms depend on a particle's surface area whereas its temperature response depends strongly on mass (ρv). For irregular particles such as coal, the equivalent diameters for external surface area and volume can differ significantly [8]. Moreover, the density for these particles is found to be non-uniform [8]. Hence, in-situ measurement of particle shape and density in addition to temperature measurement would allow one to better characterize and predict the thermal transport characteristics of coal particles during devolatilization and combustion. Hurt and Mitchell [9] reported large particle-to-particle temperature variations in their combustion studies of single char particles. They concluded that particle-to-particle variations in physical properties are a leading cause of these large temperature differences. Thus, individual particles have unique surface area, volume, and density and a unified approach comprising measurements of these properties is necessary if reliable predictions of transport phenomena are to be achieved.

In pursuit of this unified approach, various experimental techniques were developed recently in the single particle laboratory at the National Energy Technology Laboratory (NETL), Morgantown. These techniques involve the use of an electrodynamic balance (EDB) to characterize particle properties. The principal advantage of this instrument is the capability of suspending a charged particle motionless at the balance's geometric center, thus facilitating particle characterization.

Using a video-based imaging system, Maloney et al. [10] developed capabilities for measuring 3-D surface areas and volumes of irregular particles. Individual particles were rotated about the EDB center axis using a set of tangentially directed gas jets. As the particle rotates, a video-based imaging system records the particle images and stores perimeter data from successive video fields. Rotation rates were measured with the aid of a second video system positioned above the balance. Surface areas and volumes were calculated by summing the surface and volume elements swept out during rotation from one video field to the next.

Maloney et al. [11] also developed capabilities for measuring drag coefficient/mass ratios (C_d/m) for particles in the EDB. Using a high-speed diode array imaging system, they measured particle trajectories resulting from an applied stimulus. Particle C_d/m ratios were found from these trajectory measurements by means of a force balance model which matched theoretical predictions with measurements. Surface area and volume data were then used to estimate the particle drag coefficient by applying an analysis for deformed spheres derived by Brenner [12]. The particle mass was then calculated based on the measured C_d/m and the calculated drag coefficient [8].

SamPATH [13] developed a second method of characterizing the volume, external surface area, and drag equivalent diameter of an irregular particle based on conventions established by Heywood [4]. This method incorporated the same EDB measurement system and associated instruments that

were developed by Maloney et al. [10] to characterize irregular particles. In this method, irregular particles were characterized by obtaining directly the magnitudes of length, breadth, thickness, and projected areas from two view images in the planes parallel and perpendicular to the orientation of the particle during the measurement. The Cd/m ratio was also found for individual particles. This ratio was then used to obtain particle mass and density. A mean particle mass for the sample studied was obtained using a direct gravimetric method [8,13]. This method involved weighing and counting several thousand coal particles. Finally, the mean mass obtained using the EDB measurement system was validated by comparison with the results of the direct gravimetric method.

In view of the anticipated rapid development of technologies for co-feeding of coal and biomass, a great need exists for the development of a data base on the shape and density distributions of biomass particles for use in combustion models. Detailed property data including surface area, volume, mass, and density distributions for several coal samples are now available [8,13] for use in coal combustion models.

To this end, applying our experimental and analytical capabilities in the particle characterization research [5,8,10-11,13], this project seeks to characterize the shape and mass for biomass particles. Individual biomass particles will be characterized for their external surface area, volume, and drag coefficient/mass ratios. Analysis methods will be employed using shape and drag information to calculate mass and density distributions for these particles. Results of these measurements and analyses will be validated by independent mass measurements using a particle weighing and counting technique.

The specific objectives are:

- 1) Apply unique measurement systems to characterize external surface area, volume, mass, and density for a statistically significant number of individual biomass particles (20 particles) in the size range of 100 - 200 μm .
- 2) Obtain mean mass per particle of the biomass sample tested in Objective (1) by independent mass measurements of several thousand particles using a particle weighing and counting technique.

Experiments and data analysis are being carried out to meet the project objectives. Mean mass of several thousand biomass particles obtained in Objective (2) will be used to validate the mean mass per particle obtained in Objective (1). Co-firing of biomass and coal has been identified as a promising way of reducing net CO₂ emissions with minimum modifications in the existing technologies. The successful accomplishment of the above objectives will provide detailed particle property data required for developing improved combustion kinetic models for technologies involving co-firing of coal and biomass feedstocks.

EXECUTIVE SUMMARY

In this semi-annual report, the work performed under DOE Grant No. DE-FC26-04NT42130 during the period January 01, 2005 to June 30, 2005 which covers the second six months of the project is described and the major accomplishments are highlighted summarizing the most important research results.

Over the next decade there will be a renewed emphasis on co-firing biomass with coal. Co-firing of biomass and coal has been identified as a promising way of reducing net CO₂ emissions with minimum modifications in the existing technologies. Coal and biomass particles are irregular in shape. From a combustion perspective, particle sphere assumptions employed in most coal combustion models were found to yield significant errors (20 to 25 percent) in calculated particle volume and associated thermal mass. Even if surface area and volume differences were adequately handled in a heat transfer analysis, large uncertainties still resulted in coal particle temperature response due to particle to particle density variations. Recently, shape and density for coal particles have been characterized and detailed property data including surface area, volume, mass, and density distributions for several coal samples are now available for use in coal combustion models.

This project seeks to characterize the shape and mass for biomass particles. Individual biomass particles will be levitated in an electrodynamic balance (EDB) and their external surface area, volume, and drag coefficient/mass (C_d/m) ratios will be characterized applying highly specialized video based and high-speed diode array imaging systems. Analysis methods will be employed using shape and drag information to calculate mass and density distributions for these particles. Results of these measurements and analyses will be validated by independent mass measurements using a particle weighing and counting technique. Experiments involving counting and weighing of several thousand biomass particles employing a microscope and a sub-milligram balance experimental system will be performed by Morehouse College in Atlanta. Experiments involving imaging systems will be performed by REM Engineering Services, our subcontractor in this project, using the EDB measurement system available at the single particle laboratory, National Energy Technology Laboratory (NETL), Morgantown. Morehouse will analyze the raw data collected in this project including that by REM. The successful accomplishment of the above goals will provide detailed particle property data required for developing improved combustion kinetic models for technologies involving co-firing of coal and biomass feedstocks.

During this reporting period, Morehouse has completed testing of the gravimetric technique measurement system that was set up in the last reporting period, and a mean particle mass for the biomass sample tested was obtained. Simultaneously, REM, our subcontractor, has completed calibration of the video based imaging systems and diode-array imaging system that are employed to characterize shape and mass for individual biomass particles. Subsequently, a few actual biomass particles were caught in the electrodynamic balance (EDB), raw data for shape and mass collected and analyzed, and the shape measurement system was found to be working satisfactorily. Preliminary results of the mean mass and the shape data obtained in this reporting period are very encouraging, and more data collection is in progress.

EXPERIMENTAL

Presently work is in progress to characterize surface area, volume, mass, and density distributions for sized biomass particles. During this reporting period, REM Engineering Services, our subcontractor, has completed calibration of the video based imaging systems, and the diode array imaging system. Morehouse has completed testing of the gravimetric technique, and also collected mean mass data for several thousand biomass particles.

Calibration of Video-Based Diode-Array Imaging Systems:

The video-based imaging systems (side view and top view) are both used to assess the shape characteristics of biomass particles.

The calibration procedure involved suspending DVB (divinyl benzene) spheres in the electrodynamic balance (EDB). The spheres were backlit with a He:Ne laser at the side and a LED from the bottom. The side view is projected onto the detector of a video camera and a photodiode array. The bottom view is projected onto the detector of a second video camera positioned above the balance. The horizontal counts and the vertical lines blocked by the projected images are recorded in both cameras. The total number of horizontal counts blocked is proportional to the cross-sectional area of the sphere. Similarly, the total number of pixels blocked is proportional to the cross-sectional area of the sphere. The sphere was then retrieved from the EDB and sized to within ± 2 μm diameter using an optical microscope. This procedure was repeated for a number of calibration spheres in the particle size range of interest (64 - 230 μm).

Calibration of Diode-Array Imaging System:

The diode-array imaging system is used to measure the trajectory of the particle in the electrodynamic balance (EDB) resulting from an applied stimulus. The array is made of 16 x 62 elements of silicon photodiodes spaced 100 μm apart. A magnified image of the particle projected onto the array blocks a certain number of photodiode elements yielding an output in volts that is proportional to the location of the particle in the EDB.

The position calibration involved the following procedure. A polystyrene sphere was placed on a glass plate. The glass plate was suspended in the center of the balance by a clamp. The clamp was attached to an extended pole system mounted on a XYZ translation stage. The flat portion of the plate was aligned perpendicular to a He:Ne laser beam. Next the XYZ stage was translated until a good focus of the polystyrene sphere was observed at the bottom of the monitor. The micrometer reading of the XYZ stage (in mm) and the diode-array output (in volts) are recorded. The particle was moved 50 micron increments until it was seen at the top of the monitor. Each time the particle was moved, the micrometer reading and the diode-array output were recorded.

Mean Mass Measurements by Gravimetric Technique:

A mean particle mass for the biomass sample tested was obtained using the direct gravimetric measurement system that was set up in the last reporting period. This involved weighing and counting several thousand biomass particles. A paper boat was made with a grid paper and its empty

weight measured using a sub- milligram balance (uncertainty $\pm 10 \mu\text{g}$). Several thousand biomass particles were dispersed on the grid surface and the weight of the particles plus the boat was measured. The particles were then counted under a microscope. The experiment is being repeated several times to obtain a statistically significant mean mass value of the sample studied.

RESULTS AND DISCUSSION

The calibration plots for the Side and Top View Imaging Systems are provided in Figures 1 and 2. The calibration equations are presented in each of the plots. The R squared value in these equations is greater than 0.99. This yielded a single size measurement with an uncertainty of not more than $\pm 5 \mu\text{m}$ (worst case) over the entire particle size range (64 to 230 μm) tested.

The calibration plot for particle position versus diode-array output for particle travel from bottom-to-top is provided in Figure 3. The calibration equation is presented in the plot. The R squared value in this equation is greater than 0.99. The slope of this line yielded the distance moved by the particle per volt and was found to be $362 \pm 01 \mu\text{m/volt}$.

Preliminary result of the direct gravimetric method for weighing and counting of about 13,388 biomass particles yielded a mean mass per particle of $1.64 \times 10^{-7} \text{ g}$. More counting and weighing measurements are in progress.

Plans for the next Reporting Period

The EDB measurement system was tested by successfully catching a few actual biomass particles in the balance. The imaging systems were also tested by obtaining raw shape data (rotational surface area and volume counts, and raw mass data) for these test particles. The EDB shape measurement system has been found to be working satisfactorily. Actual shape and mass data will be collected using the imaging systems in the next reporting period. Mean mass measurements using the gravimetric measurement system will be continued in the next reporting period.

Students Supported under this Research

Michael D. Young, an undergraduate student, Morehouse College has been supported in the fall semester 2004. George Weirko-Brobby, a second undergraduate student from Morehouse has been supported in the spring semester 2005. George has graduated this May, 2005. Currently, Gautam Saha, a graduate student is being supported.

LIST OF PAPERS PUBLISHED

R. Sampath, R. M. Dixon, M. D. Young, and G. Weirko-Brobby, Surface Area, Volume, Mass, and Density Distributions for Sized Biomass Particles, 2005 University Coal Research / Historically Black Colleges and Universities and other Minority Institutions Contractors Review Meeting, sponsored by NETL/U.S. DOE, June 7-8, 2005, Pittsburgh, PA.

Figure 1. Side Imaging System Calibration

Figure 2. Top Imaging System Calibration

CONCLUSION

The project is progressing well. To date, supply requests were processed and supplies including biomass test particles (hardwood sawdust AII4546) in the size range of 100-200 microns were obtained from a cofiring pilot plant research facility owned by Southern Company, Birmingham, AL. Setting up of the of the gravimetric measurement system in the Heat Transfer Laboratory, Department of Physics and Dual Degree Engineering, Morehouse College has been completed by Morehouse, and mean mass measurements are in progress as planned. Also completed was the setting up of the EDB measurement system to characterize shape and mass for individual biomass particles by our subcontractor, REM. Calibration of the video based imaging systems and diode-array imaging system using known sizes of polystyrene particles were completed in this reporting period. Shape measurements for biomass particles using the imaging systems are in progress as planned.

FUTURE WORK

Setting up, calibration, testing of the measurement systems with actual biomass particles have been completed to date. This amounts to 50% of the proposed project work to date. Data collection and analysis of raw data are planned for the next performance year. Counting and weighing of another about 20,000 more biomass particles to obtain mean mass per particle is planned and is anticipated to be completed in the next reporting year. Also, statistically significant number of individual biomass particles (about 20 particles) are being caught in the EDB and collection of raw data for their shape and mass is expected to be completed in the second year. In other words, close to 100% of the data collection proposed in this project will be obtained by the end of the second performance year. This would amount to completion of 75% of the proposed project by the end of the second reporting year. Remaining 25% of the project work including completion of data analysis and reporting will be performed in the third performance year of the project.

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