# **Final Report**

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# **Project Title:**

# Biomass Testing Laboratory for Physical and Thermal Characteristics of Feedstock of North Dakota

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Renewable Energy Program

North Dakota Industrial Commission

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# **ABSTRACT**

A substantial void exists among producers and processors of North Dakota biomass regarding its quality, suitability for densification, and energy applications. North Dakota State University (NDSU) Research Extension Centers (REC) have initiated 10-year biomass research trials at 5 locations and 50 alternative mixtures. However, facilities to test these materials are scarce. Moreover, new processors in the state (GRE and other biomass based industries) also have need for independent material testing in advance of market development. Evaluation of the physical and thermal characteristics of raw and processed biomass forms the important phase of evaluation of baseline data. This information guides various efficient operations of biomass processing and handling as well as aiding in development of new processes.

This two year, \$450,000 equipment grant project established a "Biomass Testing Laboratory" (BTL). Four major pieces of equipment (thermo gravimetric analyzer, universal testing machine, environment control chamber, and calorimeter) were purchased, installed and tested successfully. The lab has the capability to evaluate physical and thermal characteristics of diverse ND feedstock and densified biomass. In addition to being a new resource available to researchers, the equipment would be available to producers and processors seeking biomass quality information. The project was lead by the Department of Agricultural and Biosystems Engineering, NDSU in collaboration with NGPRL, USDA-ARS at Mandan. The test results of various biomass feedstocks studied are described in this final report. The lab is fully operational from June 2012.

Total Project Cost: \$450,000 (NDIC cash award: \$225,000)

Participants: NDSU and NGPRL, USDA-ARS

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# 1. INTRODUCTION

# 1.1. Objectives

Establish a Biomass Testing Laboratory (BTL) to evaluate physical and thermal characteristics of diverse North Dakota feedstocks and densified biomass products that have commercial market potential.

# 1.2. Background

Measurement of relevant physical, mechanical and thermal properties of North Dakota's biomass resources is the first essential stage towards market development, commercialization, and formation of purchase contracts. These biomass characteristics influence a) design and development of biomass processing machinery, b) optimized operation of processing machinery, c) mechanical strength of biomass and products, d) economics of product and energy production, and e) feasibility of alternative biomass species for densification and energy applications.

Raw biomass is low value, bulky, and expensive to transport. Yet, processors seek to diversify biomass purchases regionally to minimize production disruptions due to adverse weather and spread economic impacts of their activity farther. Consequently, a need exists to densify biomass and reduce transportation costs. However, optimal densification often requires pregrinding.

Mechanical properties of biomass, such as compression, tension, and shear strength directly influence the size reduction process (e.g. grinding), energy expended and economics. Proper mechanical strength analysis of biomass helps in identifying the best method for efficient grinding and possible development of an efficient grinder. Biomass testing and quality analyses will also be performed on densified products, such as stored compressed bales, pellets, and briquettes. The testing of material before and after a densification provides important information on energy and mechanical efficiency. The mechanical strength of products, such as pellets, can be well correlated to their durability (integrity) during handling and transportation.

Moisture content is the single most important characteristic that influences most physical, mechanical, and thermal properties of biomass. Hence, these properties need to be reported against moisture content. When developing an industry, biomass testing is an integral joint process that establishes quality benchmarks, which facilitate trade, develops opportunities for additional value creation through blending and densification, and leads to systemic improvement in production efficiency and cost reduction. The various tests required performing size reduction, densification (pelleting, compaction, etc), and energy characteristics of the biomass and the products are shown in Fig. 1.

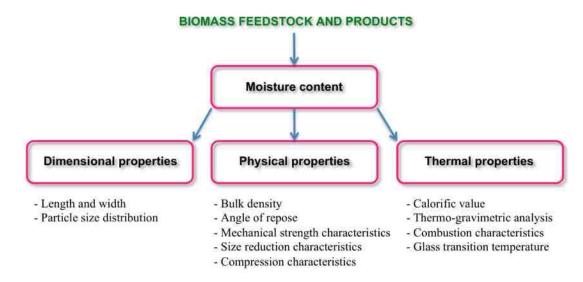


Fig. 1. Biomass testing laboratory activities on dimensional, physical, and thermal characteristics determination of selected biomass

### 1.3. Methodology

Well-established procedures to determine the various properties (Fig. 1) were available in standard materials that include technical journal articles, standards, and supplied manuals of the equipment. Since measurements are based on standard protocols, the success of utilizing the equipment of the project follows automatically, once equipment are installed and tested. In a sense, measurement of these properties is straightforward. However, with application of mathematical modeling (e.g. moisture hydration) and statistical analysis the results can be well interpreted and represented.

#### 1.4. Facilities

The Northern Great Plains Research Laboratory (NGPRL), USDA-ARS, Mandan, ND has provided a laboratory space of 20 feet by 30 feet (600 ft²; "in-kind" match) with necessary scientific laboratory facilities for the BTL. Other facilities such as NDSU Biomass Preprocessing Laboratory (1800 ft²) will also be used to support the activities of BTL. Capabilities of the laboratory were further developed using other funds (NDSU) that include several equipment that include muffle furnace, drying oven, commercial microwave oven, durometer, desiccators, weighing scale, hot-wire hygrometer, anemometer, infrared temperature gun, and digital temperature probe, analytical balance, digital calipers, machine vision application system using digital high resolution scanners, fluorescent illuminators, magnifiers, and computer, Wiley and hammer mills, pellet mill (10 hp). Fresh samples were also stored in BPL using cabinet and upright freezers. Necessary power (band saw and cordless drill/driver) and hand tools (wrenches and spanners) were also available for maintenance, repair, and small fabrication, and utility carts for sample handling. The capabilities of these labs were currently expanded as several

equipment/devices were being added through other project funds.

Feedstock from NGPRL, USDA-ARS, Mandan, ND research plots, Research and Extension Centers (REC) of NDSU, and agricultural suppliers were used for biomass performance testing. The central location of this testing facility will provide ready access to all state residents and researchers. The site also offers ample acreage for biomass production studies and the new faculty member is expected to collaborate with other NDSU and NGPRL scientists already working there on various biomass production trials. This growing network of scientists and engineers includes: (1) agronomists working on biomass production and ecological modeling, (2) engineers working on biomass harvest, densification, and storage, and (3) end users, such as GRE testing biomass co-firing and other NDSU researchers in Fargo using materials for fermentation to ethanol. This collaboration will help integrate results across the biomass supply chain to show producers and processors which crops will be most desirable in North Dakota.

# 1.5. Major Equipment Purchased through this Project

The four major pieces of equipment, such as (1) thermo gravimetric analyzer, (2) universal testing machine, (3) environmental control chamber, and (4) calorimeter were purchased through this project for the BTL. An illustration and short description of the application of these equipment is given below (Fig. 2).



Fig. 2. Major equipment of Biomass Testing Laboratory and their applications

This NDIC fund is essential an equipment grant. These equipment were purchased through Department of Agricultural and Biosystems Engineering (ABEN), North Dakota State University (NDSU) purchase procedure. The NDIC contract number for this project is R-008-018 (Appendix-I). The NDSU project information for this project is Fund Number: 46000, Control Dept: 7620, Program #: 01463, and Project#: FAR0016577. These equipment were installed at BTL of NGPRL, USDA-ARS, Mandan, ND. The description of these equipment, measurement technique, and results are given in appropriate sections later (Sec. 2). Although there were some

issues during installation (Appendix-IV) that affected the productivity, these equipment were successfully installed and tested and they are fully operational from June 2012.

### 1.6. Application of the Project

The results will find application in aiding efficient industrial operations, handling, and quality control. The results will give producers knowledge about the quality of their biomass product and its value. This provides a better understanding of how simple value-added steps, such as controlling moisture (drying) and pelleting would command a premium price. Similarly, the results will help the industrial processors know the quality of the material they receive from the producers and appropriately price the supplied biomass and their products. This is similar to the quality control laboratory of established industries such as grain, feed, and dairy processing. Thus, the laboratory will help ascertain the quality for both parties (farmers and industry) involved in biomass utilization in North Dakota. Hence, the laboratory forms a vital link in the ultimate goal of developing a bio-based industry and market in North Dakota.

Knowledge of the biomass material from the standpoint of producers and processors is highly important in supporting and sustaining the supply and production of bio-based industrial development. The outputs of the BTL influence all the processing, handling, and utilization activities of biomass based industries. Quality attribute determination also guides efficient operations at every stage thereby improving the economic impact of biomass utilization. Cataloging the physical and thermal characteristics will also lead to the establishment of biomass quality standards that will be useful to both producers and processors.

Quality assessment of feedstocks and densified products is of prime importance to the biomass industry. Hence for the development of biobased industries, the quality attributes of the feedstock, various intermediate products of processing stages, and the final product need to be assessed to ensure overall success of the industrial operation. The producers need to know about the quality attributes of their produce to meet the requirements of processing industries. Producers will also recognize the value of their feedstock from the quality attributes for proper pricing of their produce, and possible premium if their produce exceeds set quality standards. Biomass characterization procedures and results of the laboratory will be useful to the entrepreneurial and established industries equally. Therefore the BTL will have a supporting role in the development of biobased industries of the state.

Development of bio-based industries in the state, including allied industries of supply, logistics, and product streams of bioenergy and bioproducts, is a positive step towards clean, renewable, and homegrown energy and products. The activity of the BTL that supports North Dakota biomass and their products is an integral part of establishing a biomass industry in the state. Thus, the ultimate goal of this laboratory project is to serve as an important component of biomass input and product quality evaluation in biomass processing in the state.

Characterization of the input and output material determines whether a particular process is yielding the desired outcome. Because the cellulosic biomass based industry is in its infancy in the state, special emphasis needs to be given to characterization of locally available, homegrown biomass crops, and assess their suitability for value-added processing. Although knowledge generated from other parts of the country serves as a basic guideline, the BTL addresses specific local biomass production opportunities and the individual needs of local proprietary processing methods. Various component operations of the whole processing chain need to be properly understood, developed, and efficiently operated. Since such facilities do not exist in North Dakota, this project assumes greater importance serving to the benefit of the local communities and the state. Needless to say, establishment and maintenance of this laboratory is the initial, yet indispensable, step in creating North Dakota's future cellulosic biomass based industry and economy.

Technical data in a form that guides efficient and economical market development at every process step is not readily available. This proposed laboratory will address bridging this gap, and positively impact the establishment of bio-based industries in the state. Creation of this laboratory will help producers determine the quality of their products and learn how they will be compensated for their resources. Similarly, the processors can assess the quality and value of their products. The laboratory will continue to serve as a testing facility to serve the needs of farmers, researchers, industrial personnel, entrepreneurs, and general public of the state. Consolidated results will be published as factsheets, extension bulletins, journal articles, demonstrations, and presentations for everyone to use. The presence of NDSU Extension across the state, a wealth of faculty research expertise, and contacts with agricultural leaders, energy firms, and environmental groups assure development and delivery of high quality educational materials targeted to the specific interests of potential attendees.

# 2. MATERIALS AND METHODS

#### 2.1. Test Material

Biomass feedstock materials from the fields of NGPRL grown in 2011 were used as the test material. Biomass crops tested include corn stalks, switchgrass, big bluestem, bromegrass, and wheat straw. As these biomass were collected after full maturity and allowed to dry in the field or storage they are mostly dry.

#### 2.2. Moisture Content

The procedure of ASABE Standards 2008 (S358.2 Moisture Measurement – Forages, ASABE, St. Joseph, MI) was used to determine the moisture content of the samples. Three replications were used for each biomass. Samples were oven dried at 103 °C for 24 h and mass measurements were recorded using a 0.0001 g accuracy analytical balance. From the initial and final (bone dry) masses, the moisture content was evaluated and expressed in percent wet basis (% w.b.).

# 2.3. Description and Principle of Operation of Equipment

Following is a brief description, principle of operation, and experimental procedure followed for the four major equipment purchased in this project.

# 2.3.1. Thermal Analyzer - TGA/DSC

#### Description:

Thermal analyzer (Model – Mettler Toledo; TGA/DSC1/SF) that produces the thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) characteristics curves of biomass samples when they are subjected to different type of thermal environments. The system consists mainly of the TGA/DSC unit (Appendix-II), computerized control, specialty gas (e.g. nitrogen, air), cooling water unit, and analytical balance (Fig. 3).

# Principle of Operation:

TGA/DSC unit consist of a precision electrical furnace (ambient to 1100 °C) where a precision cantilever arm microbalance (microgram resolution) holds the sample (about 50 mg) while furnace environment is filled with specialty gases (e.g. nitrogen, helium, argon, air) of metered flow rate. The furnace temperature can be controlled so that a combination of constant



Fig. 3. Thermal analyzer - installed with analytical balance and computer controls

(isothermal) or dynamic (adiabatic; increase or decrease; e.g. 20 K/min) can be programmed and the sample will be subjected to these temperature and gaseous (up to 3 gases can be chosen) environment. While the sample was under specified thermal and gas composition, the mass of the sample (TGA curve) and the temperature of the sample as well as the temperature of the reference (microbalance pan) were recorded (DSC) continuously. TGA and DSC are characteristics of the sample and gives good insight of their thermal behavior. Loss of moisture and volatiles will give sudden drops in TGA curves, and melting and burning events will give peaks/valleys in DSC.

A calibration test results with material of known melting point used to verify/calibrate the system is shown in figure 4. It can be seen from the calibration results, the calibration sample materials such as indium, zinc, aluminum, and gold exhibited sharp melting peaks at their respective melting temperatures that can be read from the temperature (x) axis or from the textual outputs of the operating software on the curve. Such calibration ensures the system is operating correctly and can be used to study actual samples (unknown) materials.

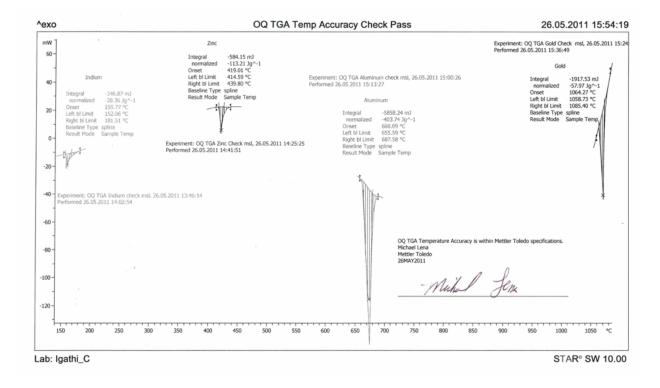


Fig. 4. DSC curves of standards (metals) – calibration curves

# Experimental Procedure:

An overall step-by-step experimental procedure for operation is presented as follows:

- 1. Open the supply of purge (e.g. nitrogen) and reaction gases (e.g. air) and ensure appropriate pressure (e.g. 30 bar).
- 2. Switch on the water cooler (chiller unit and control panel).
- 3. Switch on the gas control unit of TGA/DSC system.
- 4. Switch on the TGA/DSC system.
- 5. Enough time is allowed for the system to stabilize the temperature (e.g. 2 h) before actual experiments. If experiments are to be conducted daily the cooler and the system should be left on (recommended). The power saving mode will take care of the system.
- 6. Open the computer control program (e.g. STARe) that runs the system.
- 7. Open the "methods" file that stores all the temperature and gas profiles to be subjected to the samples. New method files can be created or existing can be copied and edited to suit the particular experiment. Some of the common inputs are selection of gases at different temperatures, isothermal and dynamic temperature selection, heating or cooling rates, and start and end temperature of heating and cooling events.
- 8. A small quantity of biomass (about 50 or less microgram) sample to be tested should be weighed in the alumina micro-crucible (70 microgram) so that the sample occupies about

- a third of the micro-crucible volume. High precision analytical balance 0.0001 g should be used to measure the sample mass. The net sample mass can be input while launching the "method" from computer to start the system. Moisture content of the sample should be evaluated separately.
- 9. Using the system touch screen display (SmartSens Terminal) the furnace was opened for loading the crucible with sample on to the pan of the system microbalance.
- 10. Close the furnace using touch screen display.
- 11. Input the test conditions (e.g. sample name, sample size, ID number, replication, etc.) in appropriate fields or comment text boxes through the software.
- 12. Press start button from the control software. This will make the method to go to the "Experiments on module" state and will wait for the users action.
- 13. Press "Proceed" button on the system touch screen to start the experiment. The terminal will show any combination of the temperature of furnace, temperature of the sample, or mass of the sample that were chosen (that can be changed).
- 14. The computer monitor will display both the TGA and DSC curves as the experiment proceeds.
- 15. The experiment will proceed up to the final temperature specified and the furnace after completion will return automatically ambient temperature (cooling return phase). During which time the next sample (as well as method if required) can be prepared.
- 16. After reaching the ambient condition noted from the system touch screen terminal, the furnace can be opened and the crucible was unloaded and final mass measured.
- 17. Usually a run with empty crucible (blank) was performed before running the actual (crucible + material) sample. This combination of blank and actual sample will nullify the effect of crucible and determine the thermal characteristics of only the sample.
- 18. Proper cleaning procedure suggested should be applied for reusing the crucibles (muffle furnace heating to clean them).
- 19. Generated curves can be opened using "evaluation" commands that could evaluate several outputs (e.g. integration area under curve, onset and end points of events, pdf outputs of curves).
- 20. A change in "method" always needs a run of blank before actual experiments so that proper blank separation occurs with the altered method.
- 21. Calibration using standard materials (supplied or purchased) should be carried out once a year using the special calibration "method" already available in the control software methods archive. Similarly the system backup (methods and results) should be performed regularly using appropriate commands.
- 22. Shutdown procedure should follow the opposite direction of various operations outlined thus far.

# 2.3.2. Universal Testing Machine (UTM)

# Description:

UTM produces the force-displacement characteristics of samples when subjected to mechanical modes of forces such as shear, tension, and compression. These mechanical characteristics will evaluate the mechanical strength properties of the biomass material that is unique to sample species. The system consists mainly of the basic unit with frame, crosshead, and load cells, (Appendix-II), computerized control, specialty attachments (e.g. shearing device, tensile grips, cutting device, compression platens), and printer (Fig. 5).



Fig. 5. Universal testing machine with tensile grips for tensile strength testing for biomass

# Principle of Operation:

The high capacity universal testing machine (Model - MTI) is a standard device for quantifying mechanical strength characteristic of biomass materials. The UTM essentially applies force to the sample (load) with precision movement (deformation or displacement) and records the force and displacement. Increase in load indicates high strength while sudden decrease the failure of the material. After a complete failure the load goes to the zero or minimum level. Computer software controls the device. The UTM, with suitable attachments, can produce load-deformation curves of biomass samples during compression, tension, and shear modes. From these load-deformation curves, the mechanical strength (e.g. cutting, crushing, shearing, friction, tensile, breaking, bending) of the biomass materials and associated energy can be obtained for analysis. These results can be used to evaluate the energy requirement for grinding and densification of biomass material.

# Experimental Procedure:

An overall step-by-step experimental procedure for operation is presented as follows:

- 1. Prepare the sample. Tensile testing requires notching, while shear and cutting do not.
- 2. Evaluate the moisture content of the sample.
- 3. Load the sample in the attachment.
- 4. Set the crosshead limits according to the type of the experiment using the limiting nuts behind the crosshead.
- 5. Through the computer control (MTI System Software), the type of tests (tensile or compression) can be performed by selecting appropriate procedure files (Setup Condition File).
- 6. Through the software it is possible to select the type of plot (stress vs strain or force vs displacement), specify X and Y axis labels and limits, specification of load cell, cross section geometry of samples, output units, data plotting as points or line connecting points, speed and direction of crosshead, load limits to specify stoppage conditions, file storage location, etc. Pressing "...Proceed to Test" initiates the testing.
- 7. The width and thickness or diameter based on the geometry can be input after measuring these dimensions using calipers. The "Proceed" command button will start the test.
- 8. The software controls the entire test and the results will appear in the form of X-Y curve.
- 9. Results will appear in the form of curve and numerical data, and the analysis results and other options can be made available by pressing "Switch Graph" command button.
- 10. The ascii results of X-Y data stored in specified location can be analyzed using a spreadsheet application to determine the energy involved (area under force-displacement).
- 11. Proper selection of crosshead speed should be selected to obtain the good curve with

sufficient data for analysis.

- 12. The parasitic forces (friction) associated with each attachment were compensated by conducting a no-load test (blank) and subtracting this characteristics from that with the sample separately using the spreadsheet program.
- 13. Based on the data obtained proper number of replications should be used in the experiment.

# Developed UTM Attachments:

UTM system is a basic unit on which specific attachment should be used to perform different types of strength testing. Most of these are rarely available that are suitable to use with biomass feedstocks. Thus these attachments for biomass testing need to designed, developed and fabricated locally. We have designed and fabricated three attachments and some of the drawings and fabricated attachments are shown here under.

# A. Double-shear device attachment:

The double-shear device (Fig. 6) consists of a cage (Fig. 6. bottom left) and a solid insert (Fig. 6. bottom right) that slides freely inside the cage. Matching holes of various sizes ranging from ½" to 1½" that run through both the case on insert. Cage is attached to the bed of the UTM that contained the load cell and the insert is attached to the crosshead. Load cell can be either attached on the bed or crosshead of UTM – and both arrangements are technically equal.

Stem of selected biomass, after dimension measurements, was inserted through appropriate hole and the UTM was operated through software and the mechanical strength characteristic curves obtained. Testing will proceed until the sample was sheared by the action of upward moving crosshead. It can be noted that two planes of failure (3 pieces) results, hence the name double-shear. The failure force thus was actually responsible for this two shear areas generated. From the force and distance data the shear strength is calculated as follows:

$$\tau = \frac{F_s}{2A_s} \times 10^{-6}$$

where,  $\tau$  is the shear stress at failure, Mpa;  $F_s$  the shear force at failure, N; and  $A_s$  the single failure area of sample, m<sup>2</sup> (two shear areas result).

The area under the force-displacement curve between suitable start and end limits will represent the shear energy (N m). Specific shear energy (per unit area) was then calculated by dividing the shear energy by the area of failure (N m<sup>-1</sup>).

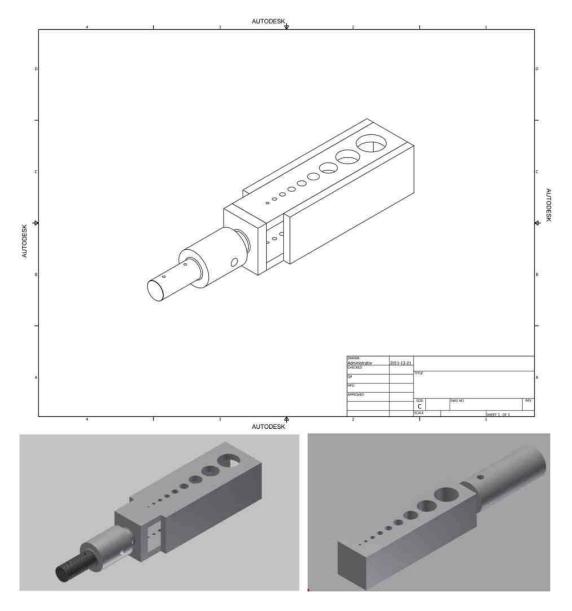


Fig. 6. Technical drawing of the designed double-shear device for shear strength of biomass with capability to test samples from grasses to corn stalks

Failure area of sample based on whether hollow or solid was calculated using the following equations:

Solid stem: 
$$A_s = \frac{\pi}{4} D^2$$

Hollow stem: 
$$A_s = \frac{\pi}{4}(D^2 - d^2)$$

Where, D is the outer diameter of the stem, m; and d is the inner diameter of the hollow stem, m.



Fig. 7. Indigenously designed and fabricated double-shear device attachment for shear strength determination of biomass

# B. Tensile grip attachment:

Commercial tensile grip (Curtis "Sure Grip") clamps attachment was used for tensile testing of biomass stems (Fig. 8). The grip jaws held selected biomass stems. Slipping was avoided by wrapping the ends using emery paper before gripping. Notch was given on the approximate middle portion of the stem to ensure breakage occurs on the region of the notch. Dimension of the notch area and wall thickness were measured using digital calipers, from which the area of failure will be evaluated.

UTM was operated through the software selecting appropriate tensile strength testing procedure and the mechanical strength characteristic curves obtained. Testing will proceed until the sample was failed under tension by the upward action of the moving crosshead. It can be noted only one plane of failure (2 pieces) result in tensile tests. The failure force thus was actually responsible for the single tensile failure area generated. From the force and distance data the tensile strength

and energy are calculated as follows:

$$\sigma = \frac{F_t}{A_t} \times 10^{-6}$$

where,  $\sigma$  is the tensile stress at failure, Mpa;  $F_t$  the tensile force at failure, N; and  $A_t$  the failure area of sample,  $m^2$ .



Fig. 8. Commercial tensile grips (Curtis "Sure Grip") attachment used in the tensile strength determination of biomass

Failure area of sample is usually rectangular and was calculated using the following equation:

$$A_t = W \times T$$

where, W is width of the failure area under tension, m; and T is the failure area under tension diameter of the stem, m. and d is the inner diameter of the hollow stem, m. Several measurements should be made and the average values of W and T should be used in the calculations.

# C. Biomass cutting attachment:

Biomass cutting attachment is essentially a modified Warner-Bratzler shear device, which was originally meant for testing of typical softer food products. The cutting edge of the blade of the device is blunt and the blade comes in two configurations, the straight blade used for rectangular specimens, and the notched blade used for cylindrical specimens. Thus the existing cutting edge of the blade being blunt was not suitable for cutting corn stalks or similar tougher biomass materials such as corn stalks. Therefore, we modified the blunt edge of the notched blade into a sharp cutting edge; hence called "Modified Warner-Bratzler" shear device. The attachment essentially has a block with two vertical guides, horizontal guide or platform, two solid support columns (Fig. 9 - left) and a moving blade (Fig. 9 - right).

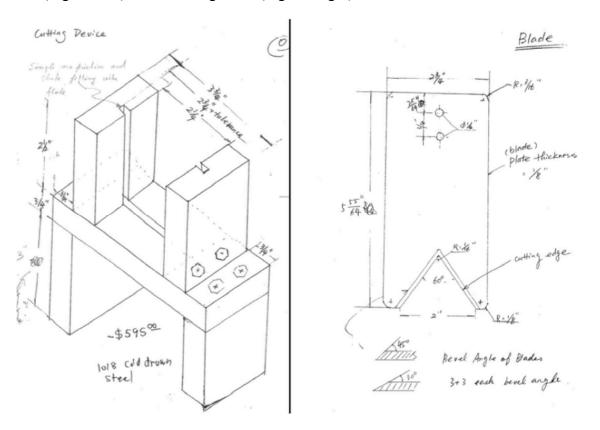


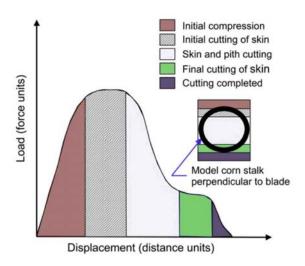
Fig. 9. Freehand sketch of designed modified Warner-Bratzler cutting device for cutting strength determination for biomass; Right – design of self-aligning knife

Guides, horizontal platform, and support columns were bolted together by screws (Fig. 10). Blade is attached to the crosshead and the block was simply kept on the bed of the UTM on the load cell. Cutting load is applied in compressive mode. Guides allow the blade to self-align, and the blade fully penetrates and emerges on the other side of the horizontal platform. A bevel angle of  $30^{\circ}$  was used. The triangular notch of the blade helps self-centering of samples during cutting. All the components (Fig. 10 - left) can be easily assembled (Fig. 10 - right) and cutting energy of biomass stalks can be tested.



Fig. 10. Components of the indigenously designed modified Warner-Bratzler cutting device for cutting strength determination for biomass; Right – assembly of components illustrating cutting of corn stalks

A typical cutting force-displacement characteristics could contain several regions explaining the nature of the cutting process as depicted in figure 11. Similar regions can also be indentified in



**Fig. 11. Typical distinct regions of the corn stalk cutting force-displacement characteristics** (Source: Igathinathane et al. 2010. Corn stalk orientation effect on mechanical cutting. Biosystems Engineering 107: 97-106)

cutting characteristics of other biomass crops. Cutting force and energy calculations are similar to tensile testing procedure (refer equations presented already), while the differences are in the mode of failure and direction of travel of cross heads. The area of cross-section should be based on the geometry of the stalks. Most of the stems will follow the elliptical cross-section and the new area of cut is given by:

$$A_{\rm e} = \frac{\pi}{4} a \times b$$

where,  $A_e$  is the elliptical cross-sectional area of stalk in cutting failure,  $m^2$ ; a is major axis (or width) of the stalk, m; b is the minor axis (or thickness) of the stalk, m. This cross-sectional area will be used in appropriate equations (see tensile strength) to obtain the cutting stress and energy required. In this report such cutting energy measurements are not reported, but will be subsequently added.

# 2.3.3. Environment Control Chamber (ECC)

# Description:

ECC (Model – ESPEC, BTX-475, 4 ft<sup>3</sup>) is capable of maintaining an environment of constant temperature and relative humidity (RH) with proper controlled air circulation (Fig. 12). Practical range of temperature and RH are approximately 15 to 85 °C and 10% to 95%, respectively (Appendix-II). The unit consists of an airtight chamber, a refrigeration system, a water bath, dry and wet bulb thermometer, trays to hold samples, a circulation fan, and electronic controls.

### Principle of Operation:

ECC continuously conditions air and circulate inside the chamber. Temperature is controlled by electrical heater (increase) as well as refrigeration unit (decrease). RH is controlled by water bath (increase) as well as refrigeration unit (decrease). Water bath pumps moisture into the air (increases RH) and refrigeration unit condenses moisture (decreases RH). The condensing moisture is actually separated from the air thus decreases RH of the air. Purified water at pressure of 20-50 psi supplied to the unit takes care of the humidifying water requirements. Dry and wet bulb (wet wick) thermometers measurements correlate to RH and serves as a means of RH determination. The Watlow F4 controller attached at the front is used to set the limits of the experiment.



Fig. 12. Environmental control chamber showing samples loaded for hydration or storage characteristics determination

# Experimental Procedure:

An overall step-by-step experimental procedure for operation is presented as follows:

- 1. Open the water supply (20 50 psi) to the unit.
- 2. Connect the unit to special electrical supply (2 A, 125 VAC).
- 3. Switch on the Run Switch on the front panel. This will power the circulator fan, the heater and refrigerator circuits that are controlled through Watlow F4 controller. Turning off the Run Switch will shut off these components.
- 4. If a fault occurs when ECC is in the run mode, the fault reset switch is pressed; the chamber may start running again if the Run Switch was left in the run position.
- 5. Set the temperature values of overheat and overcool protector set point thumb wheels to appropriate limits (on the left side bottom of the unit).

6. Watlow F4 controller should be used to set the operating set point (static or profile mode) of temperature and RH (Fig. 13). For through operation of the controller instruction manual may be referred.

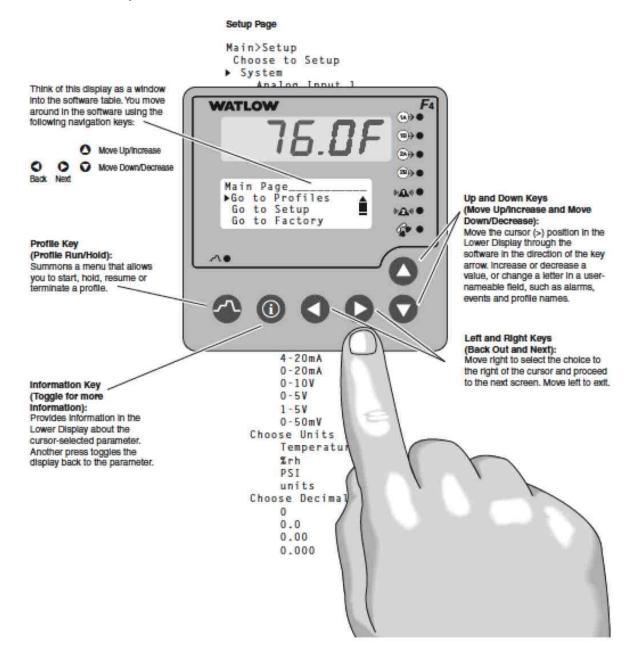


Fig. 13. Keys and navigation features of Watlow F4 controller of ECC

- 7. The navigation keys were used to select and adjust the set points.
- 8. In static mode, the set points are displayed in the Lower Display (small green multi-line text).
- 9. SP1 controls temperature, the Upper Display (single line large red LED) always shows

- the actual temperature in the chamber.
- 10. SP2 controls humidity, the Lower Display shows actual humidity in the chamber.
- 11. Watlow F4 controller has programmable time signals (digital outputs) that were used to activate chamber functions. Digital Output 8 (TS8) enables humidity conditioning. Unless TS8 is enabled, the humidity control will not happen.
- 12. After setting the temperature and RH (e.g. 40 °C, and 90% RH), the chamber should be run so that temperature and RH equilibration occurs.
- 13. Determine the moisture content of the biomass samples separately.
- 14. Load the biomass samples in trays (slotted aluminum foil promotes better moisture exchange) after temperature and RH equilibration.
- 15. Record the mass of biomass sample trays more frequently initially (5 minutes interval) and reducing the measurement frequency gradually later (30 to 60 minutes).
- 16. A top loading digital balance of with necessary accuracy (e.g. 0.1 g) and capacity should be used in mass measurement.
- 17. Quick opening and closing the chamber while withdrawing and loading the trays ensures better air conditions.
- 18. From the gain in mass over the duration of the experiment and the initial moisture content the moisture hydration characteristics (moisture content of the sample with respect to time) was obtained.
- 19. The experiment can be repeated for different crops and air conditions.
- 20. Hydration characteristics data can be mathematically fitted using several hydration models (e.g. Exponential, Page, Modified Page, and Peleg) to derive useful information. Individual hydration models in combination with Arrhenius model can be used to make prediction of hydration characteristics at intermediate temperatures (Appendix-III).

#### 2.3.4. Calorimeter

#### Description:

Calorimeter is an instrument that determines the calorific value of the test sample. The system (Model – IKA, C2000 Basic V2) consists of an airtight decomposition vessel, a temperature controlled cooling water bath, ultra pure oxygen supply, cooling water supply, and display terminal (Fig. 14). After mounting the sample on to the lid, the entire operation was controlled through the display terminal commands and measurement is performed automatically.

# Principle of Operation:

All the samples desired to be tested have a fuel value or energy content. The sample in oxygen rich environment when ignited will completely get combusted and the release of heat based on the heat content of the sample. This combustion happens in a controlled fashion inside a thick walled airtight decomposition vessel. This released heat is captured by a bath of water maintained at a fixed colder temperature. The amount of raise in the temperature of the water is a

measure of the energy value or heating (calorific value) of the sample. Starting of the combustion is triggered electrically and the sample catches fire and burns instantaneously, because of oxygen rich environment, through the cotton thread that serves a wick and connects the ignition wire and the sample held in the crucible.



Fig. 14. Calorimeter installed with high purity oxygen gas (top); sample preparation showing crucible and cotton thread from ignition wire (bottom left); decomposition vessel brought out after completion of measurement yet attached to the lid while display shows calorific value and temperature rise (bottom right)

Higher heating value (HHV) or higher calorific value (HCV) or gross calorific value is determined by bringing all the products of combustion back to the original pre-combustion temperature by condensing any vapor produced (heat of vaporization). Such measurements often use a temperature of 25°C. While the lower heating value (LHV) or lower calorific value (LCV) or net calorific value is determined by subtracting the heat of vaporization from the HHV or HCV. LHV treats any heat involved in vaporizing the moisture is not available to be realized as heat.

# Experimental Procedure:

An overall step-by-step experimental procedure for operation is presented as follows:

- 1. Open the cooling water supply tap that feed to the unit.
- 2. Pressure of cooling water should not exceed 1.5 bar. An inlet water pressure regulator (IKA C 25) can be connected to ensure this.
- 3. Cooling water temperature should be less than 28 °C. If the temperature is < 23 °C the system works in the 25 °C mode, otherwise (cooling water > 23 °C) in 30 °C.
- 4. Open the supply of high purity oxygen (99.95% pure). The oxygen pressure should be 30 bar and should not exceed 40 bar (Fig. 14).
- 5. Switch on the machine. This splash screen appears and the measuring cell opens automatically and the stirrer turns for a few second.
- 6. Then the system checks the temperature of the cooling water for about 150 s and selects the possible setting for experiment. It is also possible to change the options such as isoperibolic or dynamic though settings, but the system should check and allow for such selection.
- 7. After successful temperature check, two dots on both sides within parenthesis across a selected method, the system will be ready for operation indicated by selected mode and X time vs Y temperature graphic display of axes.
- 8. Run calibration tests with samples of known HCV. For the purpose of calibration "Benzoic acid" tablets are used (HCV = 26464 J/g). This HCV will be taken as the reference by the system and it will evaluate unknown samples based on this data.
- 9. Sample preparation is same whether it is for calibration or biomass. About 1 gram of material (the sample size is important so that the temperature increase should not be more than 3 K (Y axis limit of the display) to avoid damaging the system.
- 10. Decomposition vessel is a thick walled screw capped stainless steel vessel that has provision to hold the sample in a crucible, spring loaded valve to pressure oxygen, electrical connection to the ignition wire, and airtight rubber seal.
- 11. About 0.5 g of sample is held in the crucible. The exact mass of the material taken should be measured using a analytical balance (e.g. 0.0001 g resolution).
- 12. Hold the decomposition vessel cover on a preparation stand (Fig. 14 bottom left).

- 13. Fasten the cotton thread onto the middle of the ignition wire with a loop.
- 14. Hold the crucible in the crucible holder bow and pass the loose ends of the thread below the sample to ensure better contact, which is essential for initiating the combustion process.
- 15. Place some amount of distilled water into the decomposition vessel (e.g. 20 ml). This water increases the service life of the parts of the decomposition vessel (O ring, seals, etc.).
- 16. Carefully insert the cover with sample crucible in place into the decomposition vessel and tighten close it using the cap screw.
- 17. Closed decomposition vessel should be inserted into the filler head of the open measuring cell cover unit until it settles in place. When in place the decomposition vessel will be at the center and will not rotate but the spring element makes contact for the electrical ignition. The vertical suspension of the vessel should be checked before proceeding further.
- 18. Using "Sample" command (bottom line), the sample mass was input on "Weighed-in quant" filed, sample proper name using text characters, and 1 for both Bomb and Cell. If the measurement is a calibration procedure then the "Calibration" field should be selected. Press "OK."
- 19. Using "Start" command (bottom line) the measurement can be started and the process continues until completion of the experiment. After completion the decomposition vessel comes out.
- 20. Results of calorific value (C) in J/g, the average calibration value (V) used by the system in J/g, and mass of the sample (m) in g will be displayed.
- 21. Decomposition vessel can be removed and the exhaust gases under pressure should be released using the safety release pin.
- 22. Final mass of the crucible less the crucible mass gives the mass of burnt material (ash).
- 23. Decomposition vessel and the crucible should be cleaned off the brunt deposits with soap and water. After removing the moisture by paper towels the decomposition vessel and crucibles can be used.
- 24. Manual should be referred to set the calibration values.

# 3. RESULTS AND DISCUSSION

#### 3.1. Moisture Content

Moisture contents of some of the samples used in TGA/DSC and calorimeter studies based on ASABE Standards 2008 (S358.2 Moisture Measurement – Forages, ASABE, St. Joseph, MI) were presented in the Table 1.

Table 1. Moisture content of the samples used in thermal properties

			Box+Sample	Box+dry	MC	Avg MC	STD MC
ID	Biomass	Box	(mg)	(mg)	(%w.b.)	(% w.b.)	(% w.b.)
A1	Big bluestem	2200.1	2865.3	2817.5	7.19		
A2		2200.3	2852.5	2805.4	7.22		
A3		2188.3	2860.6	2813.2	7.05	7.15	0.09
C1	Bromegrass	2228.4	3014.8	2948.7	8.41		
C2		2232.3	3082.6	3011.5	8.36		
C3		2230.5	3082.9	3013	8.20	8.32	0.11
D1	Corn stalk	2214.5	2729.5	2687.7	8.12		
D2		2207	2672.8	2635.4	8.03		
D3		2204.9	2750.5	2706	8.16	8.10	0.06
B1	Switchgrass-mature	2208.2	3231.2	3159.2	7.04		
B2		2210.3	3100.4	3037.8	7.03		
В3		2220.2	3113.5	3050.8	7.02	7.03	0.01
F1	Switchgrass-green	2232	3238.4	3164.3	7.36		
F2		2231.8	3254.6	3178.4	7.45		
_F3		2207.1	3260.8	3179.9	7.68	7.50	0.16
E1	Wheat straw (Barlow)	2212.8	2965.7	2906.1	7.92		
E2		2230.5	2840.9	2791	8.17		
E3		2223.3	2885.4	2834.7	7.66	7.92	0.26
G1	Wheat (Barlow) stem*	2212.3	2715.9	2678.2	7.49		
G2		2198.6	2881.9	2830.4	7.54		
G3		2224.1	2851.3	2803.3	7.65	7.56	0.09

<sup>\*</sup> Used only on mechanical strength (shear and tensile) testing.

These were powdered samples and were prepared by grinding them using a Wiley mill fitted with 2 mm mesh. Ground samples allow for distribution of all components of the biomass and they produce a representative sample. Ground samples are easy to handle in TGA/DSC and calorimeter. Ground samples can be pelleted or directly used in powdered form in the calorimeter. We used powdered form and have not noticed any incomplete combustion. As these biomass materials were harvested after maturity last year and stored indoors they were relatively dry. Moisture contents of the samples analyzed have ranged from 7.0% to 8.3% w.b. Fresh samples will be tested in future.

### 3.2. Thermal Analyzer

TGA/DSC characteristics of several selected biomass samples were evaluated. The experimental results were grouped into two categories, such as, thermal analysis only with nitrogen and with nitrogen followed by air. These methods produce different thermal characteristics and help characterize the biomass material.

# 3.2.1. TGA and DSC results with only nitrogen

A thermal characterization with nitrogen represents inert condition when samples were subjected to varying temperatures. In other words, the inert environment produces the pyrolysis characteristics of the tested material. Experimental conditions use for these studies are: Heating rate of 20 K/min (K-Kelvin); temperature limits of 25 to 1100 K; and nitrogen flow rate of 50 ml/min. Initially, a blank (alumina 70 micro gram sample cup without any sample) was run (Fig. 15) and this blank curve was automatically used in the analysis (blank separation) and produced the curves with samples by suitably nullifying the effect of these sample holders (crucible).

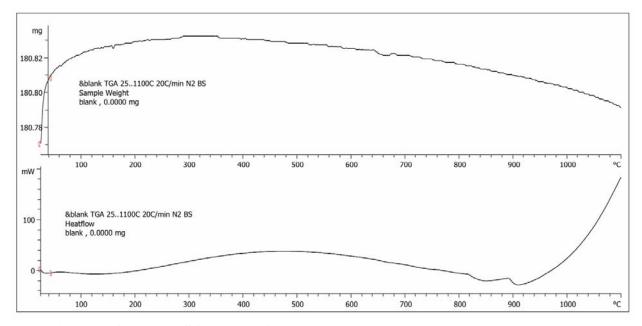


Fig. 15. TGA and DSC curves of alumina sample holder without samples (blank)

It can be seen from the TGA (upper curve) that although a dome shaped (increase then decrease in mass) was observed it is practically a flatter profile, as the total mass variation was about 0.14 mg. DSC curve shows an extended exothermic reaction (heat release) in the range of 200 to 800 K, some noticeable peaks around 800 to 900 K, and steep increase in higher temperature range after 900 K (Fig. 15). This specific characteristics of the alumina crucible was considered as "blank" and was separated from the actual sample experimental run automatically.

Figures 16 through 21 presents the TGA and DSC characteristics of several biomass samples only with nitrogen. Nitrogen used as purge gas makes the environment inert and the heating/combustion process follows the pyrolysis mode of thermal reaction. Biomass samples were ground and a sample size ranging from 5.2 to 5.5 mg was used. Samples were held in the alumina crucible for which the "blank" curves were already determined (Fig. 15).

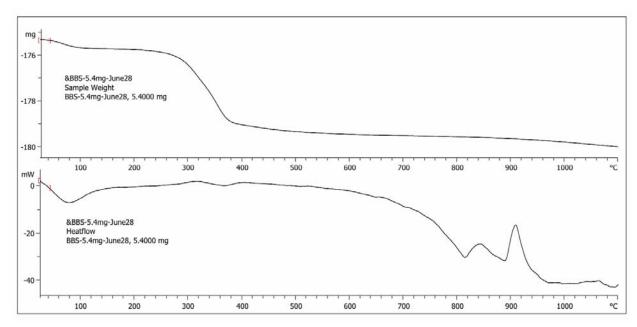


Fig. 16. TGA and DSC curves of big bluestem (7.15±0.09% w.b.)

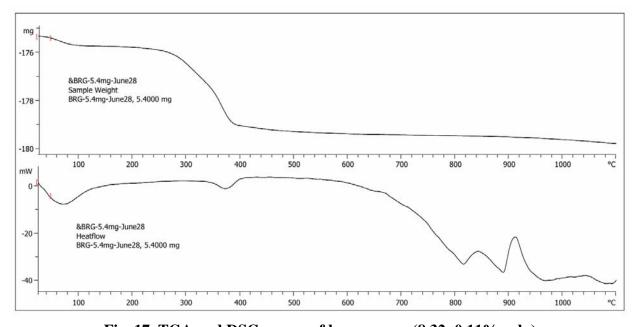


Fig. 17. TGA and DSC curves of bromegrass (8.32±0.11% w.b.)

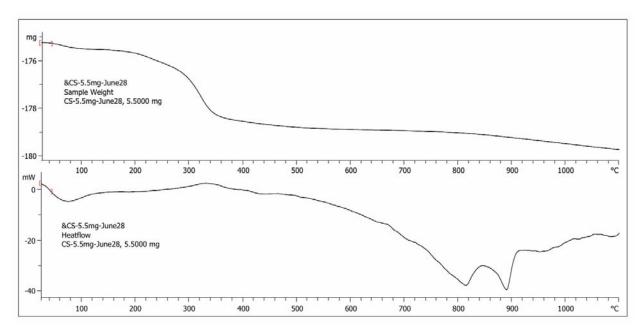


Fig. 18. TGA and DSC curves of corn stalks (8.10±0.06% w.b.)

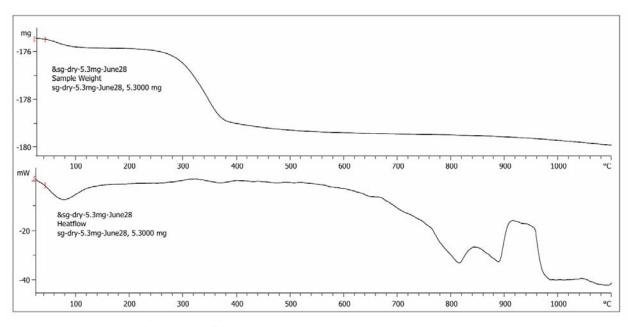


Fig. 19. TGA and DSC curves of switchgrass mature (7.03±0.01% w.b.)

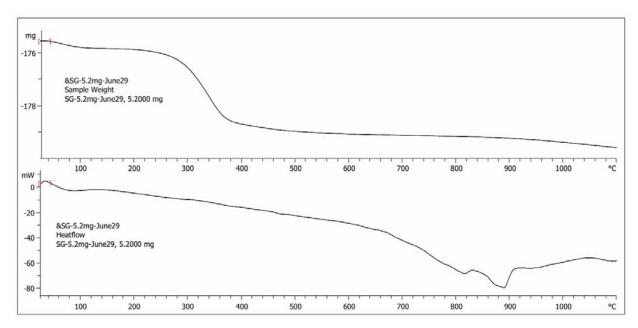


Fig. 20. TGA and DSC curves of switchgrass green (7.50±0.16% w.b.)

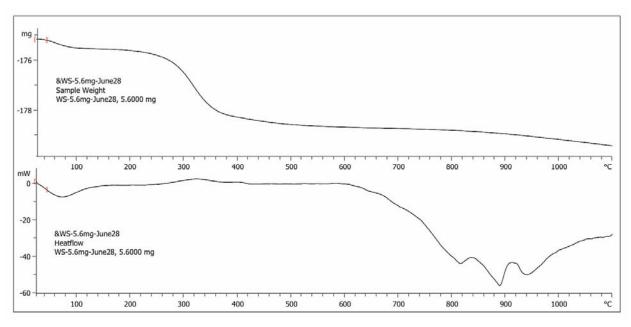


Fig. 21. TGA and DSC curves of wheat (Barlow) straw (7.92±0.26% w.b.)

TGA of all the samples show the following three major regions:

*The initial phase* (upto 250 to 300 K) - where a small reduction in mass was observed that was due to the loss of moisture (7-8% w.b.).

The second phase (300 to 400 K) - where a substantial reduction in mass was observed that was due to the loss of volatiles and pyrolytic charring of samples. At these temperatures much of the volatile chemicals will be released that marks the sudden reduction in mass.

The third phase (beyond 400 K) - where stabilization or a very gradual mass reduction was observed. The material already lost the volatiles was relatively stable and only charring happens and carbon and mineral residues are left. Since the heating occurs in inert (nitrogen) environment carbon is not burned and the heat released. After completion of the experiments, the residues were black in color indicating the unburnt carbon component.

DSC curves plot the relative temperature difference of the sample with respect to the reference. Thus indicating whether the sample receives (endothermic) or gives out heat (exothermic) with respect to the reference (based on the heating variables set by the user). In the DSC, a downward movement means the absorption of heat and upward movement the liberation of heat. DSC curves of all the samples show the following three major regions:

The initial phase (up to 600 K) – where the sample followed the temperature of the heating environment in most biomass samples. But the switchgrass – green showed a gradual decline (endothermic).

*The second phase* (600 to 800 K) – where a sudden drop showing significant heat absorption (endothermic) was observed. This heat absorption will later result in heat release.

The third phase (800 to 1100 K) – where almost two heat release (exothermic) peaks and continuous upward trend were observed. It is interesting that all biomass samples exhibit these two exothermic peaks after 800 K. Further analyses will reveal the thermal reaction mechanism involved in this phase.

# 3.2.2. TGA and DSC results with nitrogen followed by air

Figures 22 through 24 illustrate the thermal characteristics of alfalfa, bromegrass, and miscanthus stems, respectively. Initially the environment was using nitrogen as purge gas up to 600 K and from then on purified air (~21% oxygen) was supplied as the reaction gas. The airflow rate used was 50 ml/min. A blank was run with these specific conditions and subtracted automatically to give the characteristics of the sample. Introduction of the reaction gas (air) supplies oxygen and promote the complete combustion of the sample. This may mean loss of carbon mass as well as significant heat release due to complete combustion.

With the introduction of the air at 600 K, the TGA shows an additional mass loss step (all biomass studied). This represents loss of organic carbon and release of carbon dioxide and carbon monoxide as combustion gas products. After this mass loss, only incombustible ash

minerals remained and the mass of which was not affected by increased temperature from 650 K. This behavior is depicted by an almost straight-line variation for all the biomass studied.

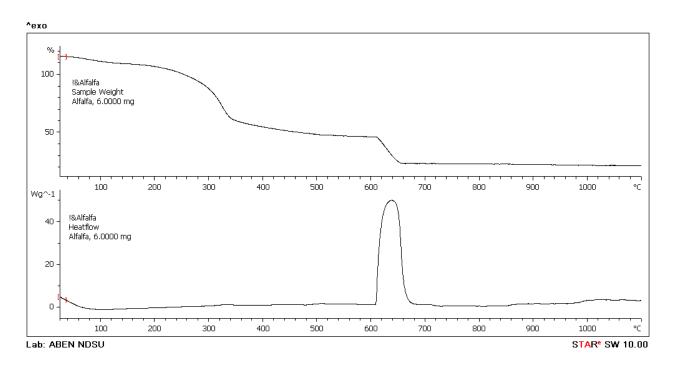


Fig. 22. TGA and DSC curves of alfalfa stem with reaction gas oxygen from 600 K

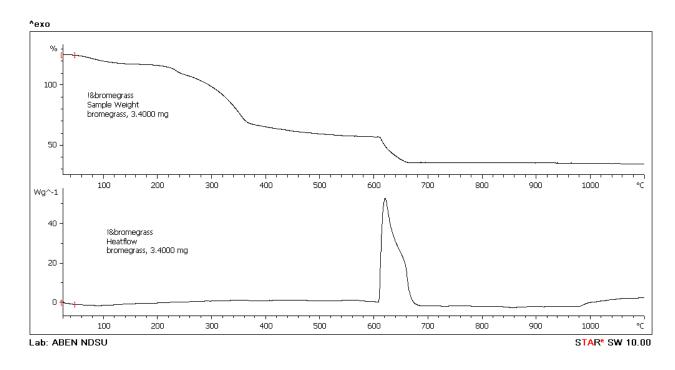


Fig. 23. TGA and DSC curves of bromegrass stem with reaction gas oxygen from 600 K

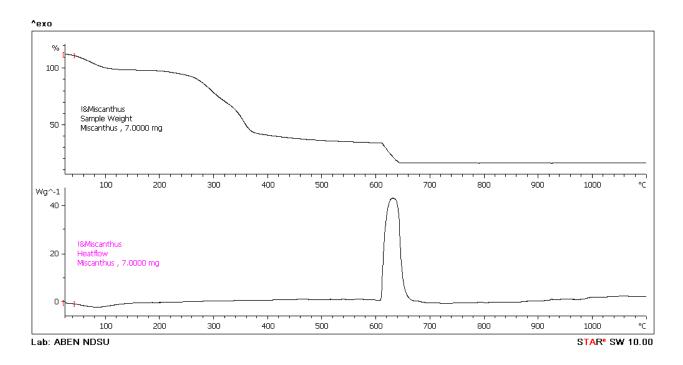


Fig. 24. TGA and DSC curves of miscanthus stem with reaction gas oxygen from 600 K

DSC curves clearly show the exothermic peak after the air introduction at 600 K. This peak represents the complete combustion of the biomass in the presence of oxygen supplied by air. Base of the peak stretched approximately between 610 and 680 K. Other than the peak the DSC curve can be considered flat. Area under the peak represents the net energy released by the biomass sample during combustion. It should be recollected that exothermic combustion was due to supply of air, while supply of nitrogen produce only endothermic pyrolytic charring. Further analyses of these curves reveal more insight into the thermal behavior. It is also possible to use other reaction gases as well as conduct experiments only with air.

Overall, it can be observed that with a small sample size (about 5 mg) the TGA and DSC of biomass samples were different and distinct. In other words, the "thermal characteristics signatures" of biomass are different; and this project is aimed at capturing these "signatures" and catalogs them for a variety of ND biomass. It is also possible to study the effect of moisture content, maturity stage, anatomical component difference, effect of storage, etc., on the thermal characteristics of biomass. When such elaborate studies were performed and analyzed statistically, we can able to conclude "whether or not" there is a significant difference in the thermal properties as affected by the aforementioned differences. These results (whether difference exist or not) will help in making decisions on utilization of these biomass materials and assess their quality.

#### 3.3. Universal Testing Machine

Double shear device and tensile grips were used to obtain the shear and tensile force-displacement characteristics of selected biomass. Typical shear and tensile mechanical characteristic curves of big bluestem (Fig. 25), bromegrass (Fig. 26), and wheat straw (Fig. 27) are presented below. Moisture content of these biomass ranged from 4.4 to 8.2% w.b.

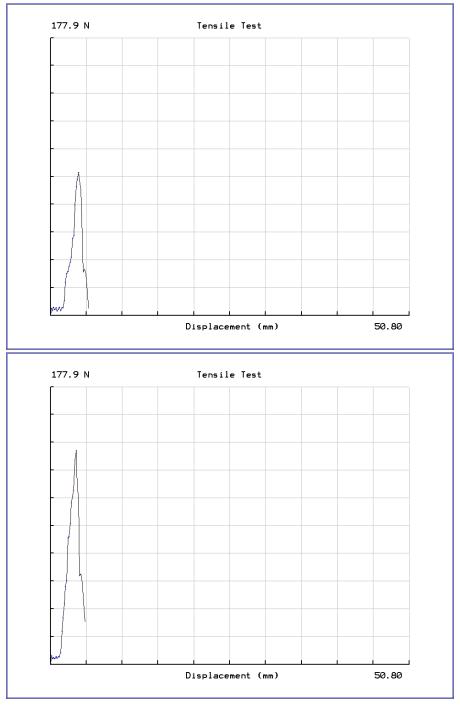


Fig. 25. Force-displacement shear and tensile characteristics of big bluestem

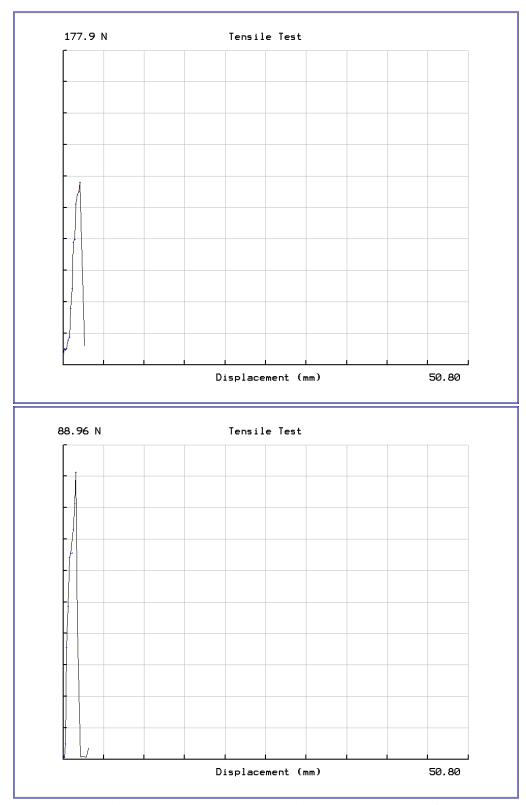


Fig. 26. Force-displacement shear and tensile characteristics of bromegrass

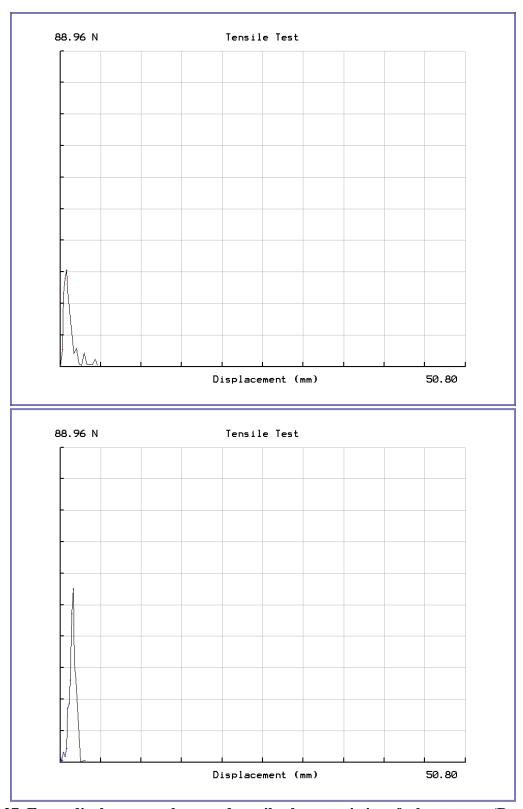


Fig. 27. Force-displacement shear and tensile characteristics of wheat straw (Burlow)

Typical force-deformation characteristics (Figs. 25-27) show an increase in load and after failure of material causing sudden drop in the load. The fall in load is more pronounced in tensile failure (bottom curve) than the shear failure (top curve). Overall the effort involved in causing the failure due to shear was relatively smaller than the failure due to tensile mode. Table 2 presents the various measurements performed replications of the biomass samples and their derived shear and tensile strengths and specific energy, while Table 3 presents the consolidated results.

Table 2. Shear and tensile strengths and specific energy of selected biomass showing replications

			Shearing
	Shear strength	Tensile strength	specific energy
Biomass	(MPa)	(MPa)	(kN/m)
Big bluestem	12.67	33.24	97.51
	15.18	26.69	159.56
	15.46	29.52	105.10
	13.80	31.32	126.91
	11.43	41.44	127.20
	14.23	26.88	152.24
Bromegrass	8.28	31.65	97.18
	10.45	19.75	118.49
	10.15	21.63	133.39
	12.72	18.60	111.62
	9.25	17.33	122.52
	12.27	33.59	98.35
Wheat	7.22	29.23	78.03
	7.23	34.73	76.49
	7.35	21.73	65.47
	9.03	22.12	69.40
	8.02	26.40	60.66
	7.12	35.08	59.27

Table 3. Consolidated shear and tensile strengths and specific energy of selected biomass

	Shear		Tensile		Shearing specific	
	strength	STD	strength	STD	energy	STD
Biomass	(MPa)	(%)	(MPa)	(%)	(kN/m)	(%)
Big bluestem	13.79	1.53	31.51	5.48	128.09	24.66
Bromegrass	10.52	1.71	23.76	7.04	113.59	14.15
Wheat straw	7.66	0.74	28.21	5.88	68.22	7.88

Note: STD – standard deviation

From these results, it can be seen that the strength and energy parameters were different for different biomass species. Further the ratio of tensile to shear strength is about 2.3 both for big bluestem and bromegrass and 3.7 for wheat straw. Shearing specific energies among the crops

were found to different. Cutting characteristics, not reported, will be performed for selected crops as we have developed the device already (Fig. 10). These mechanical properties influence the size reduction (grinding) energy requirement. Effect of moisture content, maturity, and storage of biomass will influence these mechanical properties. Further studies will be addressing the effects of these parameters on the mechanical strength and energy properties of biomass and catalog them.

#### 3.4. Environment Control Chamber

Switchgrass, big bluestem, and bromegrass are a few of the major biomass feedstocks in Midwest. An understanding of hydration characteristics of the selected biomass samples is essential for biomass storage and processing. Biomass storage affects its quality and processing, which in turn influences the biomass final utilization applications. Moisture status in biomass is the most influential factor of biomass storage and influence microbial growth hence the quality. Moisture hydration kinetics explains the dynamic moisture condition of the biomass. ECC simulates the storage environment and allows measuring the moisture hydration characteristics of biomass feedstocks. The objectives of this study were to:

- 1. Determine experimentally the moisture sorption characteristics of switchgrass, big bluestem and bromegrass.
- 2. Model the experimental moisture sorption characteristics using exponential, Page, and Peleg models.
- 3. Determine the effect of temperature on the model parameters.

Following the procedure explained earlier (Sec. 2.3.3) the moisture hydration experiments were performed using the select biomass feedstocks and the observed hydration characteristics are shown in figure 28. A fixed RH of 95% and three levels of the temperatures (20, 40, and 60 °C) were used. Three replications of biomass samples were considered at each condition (temperature and RH) and the average was presented and utilized in modeling. Hydration characteristics among biomass species were found to be different (Fig. 28). An initial quick absorption of moisture followed by gradual increase and finally equilibration were observed on all biomass feedstocks.

As presented in the Appendix-III (Mathematical modeling of hydration characteristics of biomass), the raw data (Fig. 28) was subjected to mathematical model fitting and results obtained are presented. Fitted model results of (1) exponential, (2) Page, and (3) Peleg models are presented subsequently.

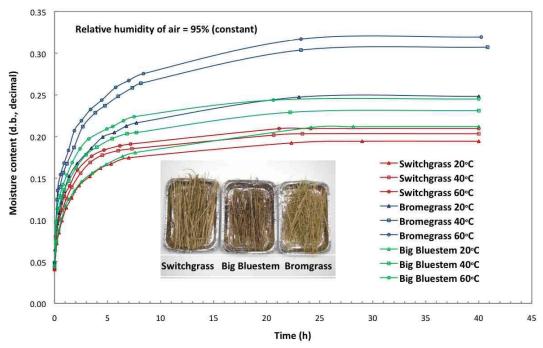


Fig. 28. Observed moisture hydration characteristics of selected biomass at different temperature and fixed relative humidity

# 1. Exponential Model

$$\frac{M_e - M}{M_e - M_0} = \exp(-k_e t)$$

$$M = M_e - (M_o - M_0) \times \exp(-k_e t)$$

Material	$\mathbf{T}(\mathbf{C}^{\circ})$	$K_{\rm e}$ (h <sup>-1</sup> )	$SSD_{-}K_{e}$	$\mathbb{R}^2$
Switchgrass	20	0.4765	0.0416	0.9489
	40	0.6243	0.0706	0.9253
	60	0.6797	0.0853	0.9152
Big Bluestem	20	0.3804	0.0384	0.9183
	40	0.5592	0.0826	0.8312
	60	0.6129	0.0811	0.8571
Bromegrass	20	0.429	0.0511	0.9101
	40	0.3974	0.0513	0.8678
	60	0.4211	0.0564	0.8269

# 2. Page Model

$$\frac{M_e - M}{M_e - M_0} = \exp(-kt^n)$$

$$M = M_e - (M_e - M_0) \times \exp(-kt^n)$$

Materials	T(C°)	<i>K</i> (h <sup>-1</sup> )	SSD_K	n	SSD_n	$\mathbb{R}^2$
Switchgrass	20	0.6050	0.0039	0.639	0.0058	0.9998
	40	0.7430	0.0151	0.5669	0.0183	0.9969
	60	0.7768	0.0116	0.5518	0.0128	0.9984
Big bluestem	20	0.5321	0.0087	0.5893	0.0129	0.9984
	40	0.7358	0.0162	0.4826	0.0174	0.9954
	60	0.7708	0.0117	0.5108	0.0128	0.9979
Bromegrass	20	0.5795	0.0081	0.5843	0.0105	0.9987
	40	0.6021	0.0198	0.532	0.0245	0.9929
	60	0.6341	0.0161	0.4927	0.0202	0.9950

# 3. Peleg Model

$$M = M_0 + \frac{t}{k_1 + k_2 t}$$

			Peleg	Model				
Materials	T(C°)	K,	SSD k <sub>1</sub>	K,	SSD K <sub>2</sub>	R³	R,	M,
	20	7.8925	0.3198	6.3355	0.076	0.996	0.1267	19.8541
Switchgrass	40	5.4227	0.5168	6.056	0.147	0.9739	0.1844	20.5825
	60	4.7516	0.4195	5.8845	0.1273	0.9839	0.2105	21.0638
	20	9.0956	0.6212	5.8724	0.1306	0.9864	0.1099	21.4688
Big bluestem	40	4.6609	0.6197	5.544	0.1991	0.9581	0.2146	22.4775
	60	4.0698	0.3931	5.0792	0.1279	0.9769	0.2457	24.1281
	20	6.0564	0.4498	5.0912	0.1176	0.9877	0.1651	24.5217
Bromegrass	40	5.099	0.614	3.8999	0.1388	0.9626	0.1961	30.5217
	60	4.3322	0.5493	3.8185	0.1524	0.9581	0.2308	31.0683

A plot of observed as well as models results (Fig. 29) shows that Page and Peleg models make very good predictions, which can also be observed from the R<sup>2</sup> values of the models. Exponential model being the most simple was not producing good prediction performance. Since Peleg is more advantageous, we used this model to perform predictions.

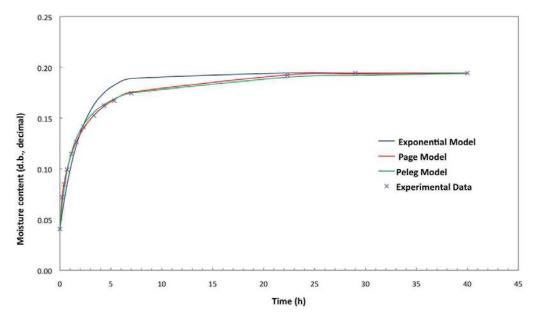


Fig. 29. Comparison of moisture hydration models performance

Arrhenius model fitting results are shown below. Arrhenius model constants (*A* and *E*) estimate the hydration model constants at any intermediate temperatures thereby help predicting the hydration at any time and temperature in the range studied.

#### Arrhenius equation

$$P = Aexp\left[-\frac{E}{RT}\right]$$

		Exponer	itial Model		Page Mode	l k	P	age Model n	
Material	A (h <sup>-1</sup> )	E (kJmol <sup>-1</sup> )	R <sup>2</sup>	A (h <sup>-1</sup> )	E (kJmol <sup>-1</sup> )	R <sup>2</sup>	A (h <sup>-1</sup> )	E (kJmol <sup>-1</sup> )	R²
Switchgrass	8.3484	6,8886	0.9798	4.6379	4,8914	0.9768	0.1774	-3.0964	0.9950
Big Bluestem	15.9879	8.9472	0.9849	9.9701	6.9983	0.9344	0.1505	-3.2506	0.965
Bromegrass	0.3494	-0.4522	0.9551	1.2244	1.8312	0.9997	0.1405	-3.4709	1.000
		Peleg Mo	del 1/k <sub>1</sub>	P	eleg Model	1/k <sub>2</sub>			
Material	A (h <sup>-1</sup> )	E (kJmol <sup>-1</sup> )	R <sup>2</sup>	A (h <sup>-1</sup> )	E (kJmol <sup>-1</sup>	) R <sup>2</sup>			
Switchgrass	7.0588	9.6636	0.9402	0.2924	1.4983	0.9998			
Big Bluestem	39.4448	13,9455	0.8717	0.5713	2.9674	0.9992			
Bromegrass	2.7077	6.8241	0.9996	1.9680	5.4976	0.7683			

Figure 30 illustrates the graphical output of the moisture hydration prediction at intermediate temperatures such as 30 and 50 °C. It should be noted that the prediction can work at any temperature between 20 and 60 °C. It is also possible to directly use the models and generate the values accurately.

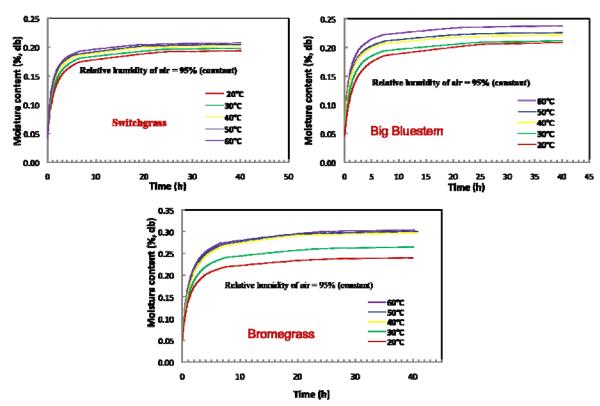


Fig. 30. Predicted moisture hydration characteristics using Peleg model at intermediate temperatures

The conclusions derived from these experiments are:

- Both Page and Peleg models effectively described the observed sorption characteristics of Switchgrass, Big Bluestem and Bromegrass.
- The Peleg model in combination with the Arrhenius equation is recommended for moisture sorption prediction.
- 86.30%, 83.80% and 80.42% water sorption took place within 5 hours for Switchgrass, Big Bluestem and Bromegrass respectively.
- Sorption rate reduce very sharply during initial the 1st. hour and quickly become approximately asymptotic to the time axis.

Similarly, other biomass feedstocks will be studied in future and their results will be cataloged.

#### 3.5. Calorimeter

The calorific value of selected biomass samples were measured based on the procedure outlined earlier (Sec. 2.3.4). Results showing the replications and the consolidated results are presented in Table 4 and 5, respectively.

Table 4. Calorific value and residue content of selected biomass showing replications

Biomass	Sample wt (net)	CV (J/g)	Residue (mg)
Big bluestem	504.3	16910	6.4
	504.4	17135	7.6
	507.1	16950	7.9
Bromegrass	502.6	16213	10.4
	504.8	16289	8.2
	507	16244	11.4
Corn stalk	404.4	16680	6.8
	404.2	16330	7.1
	400.2	16561	6.7
Switchgrass-mature	500.6	17024	7.8
	501	17178	7.5
	501	17083	6.5
Switchgrass-green	502.7	16929	9.4
	505.3	16712	10.3
	504.1	16751	9.4
Wheat straw (Barlow)	500.1	16253	12.9
	506.1	16181	16.5
	506.4	16419	11.4

Table 5. Consolidated calorific value and residue content of selected biomass

Biomass	CV (J/g)	STD	Residue (mg)	STD
Big bluestem	16998	120	7.3	0.8
Bromegrass	16249	38	10.0	1.6
Corn stalk	16524	178	6.9	0.2
Switchgrass-mature	17095	78	7.3	0.7
Switchgrass-green	16797	116	9.7	0.5
Wheat straw (Barlow)	16284	122	13.6	2.6

Note: STD – standard deviation

It can be seen from the results that the calorific values (HCV) are consistent with replications (Table 4). The residue – dark material remaining after combustion amounted to 1.3% to 3.3% among the biomass feedstocks. Observed calorific values of biomass studied (16 to 17 kJ/g) were in the range of reported results. Among the feedstocks, dry switchgrass produced the highest calorific value of about 17.1 kJ/g and bromegrass produced the lowest of about 16.2 kJ/g. Even the switchgrass-green is also ranked high (3<sup>rd</sup>) with 16.8 kJ/g. This result corroborates the idea of

utilizing the switchgrass as the model energy crop by the nation.

#### 3.6. Outreach Activities

The PIs have participated in various outreach activities highlighting the capabilities of the Biomass Testing Laboratory and are outlined below. These activities have brought visibility to the Biomass Testing Laboratory, its capability, and its application for the ND clientele. Similar outreach activities will be carried out by the PIs in the future as well.

#### NDSU Agriculture Communication NEWS (5/11/2010):

Dr. Cole Gustafson has written a column in NDSU Agriculture Communication NEWS on the Biomass Testing Laboratory. This online article is available at:

http://www.ag.ndsu.edu/news/columns/biofuels-economics/new-energy-economics-ndsu-and-usda-ars-partner-to-establish-biomass-testing-lab/



# New Energy Economics: NDSU and USDA/ARS Partner to Establish Biomass Testing Lab

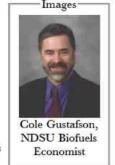
The lab will be designed specifically to test the dimensional, thermal and physical properties of biomass.

By Cole Gustafson, Biofuels Economist

NDSU Extension Service

Igathi Cannayen, an NDSU assistant professor in the Department of Agricultural and Biosystems Engineering, and I have received \$450,000 from the North Dakota Renewable Energy Council and the U.S. Department of Agriculture's Agricultural Research Service in Mandan to establish the first dedicated biomass testing laboratory in North Dakota.

The lab will be designed specifically to test the dimensional, thermal and physical properties of biomass and will be centrally located at the USDA/ARS site in Mandan.



Creation of the lab is timely for several reasons. First, NDSU has established 10-year yield trials using more than 50 different varieties of biomass. Production from these trials will be evaluated for both energy content and densification for shipping. Second, engineers are striving to develop new biomass harvesting, processing and transportation machines. Information on the physical properties of biomass will help the industry design optimal equipment.

... Column incomplete!

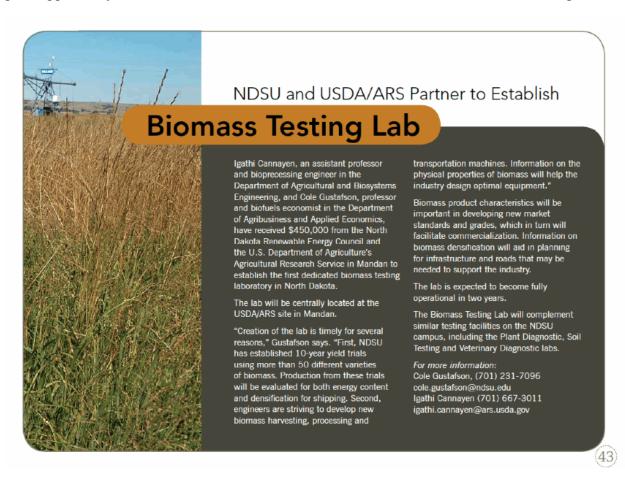
# Customer Focus Group (7/22/2010):

A presentation entitled "NDSU Biomass Testing Laboratory at NGPRL" was made by Dr. Igathinathane Cannayen to the Customer Focus Group held at NGPRL, USDA-ARS, Mandan, ND.



# 2010 Annual Highlights of NDSU North Dakota Agricultural Experiment Station and NDSU Extension Service (January 2011)

A page (shown below) on the "Biomass Testing Lab" has featured as the inner back cover of the NDSU's 2010 Annual Highlights booklet of presentation NDSU North Dakota Agricultural Experiment Station and NDSU Extension Service. NDSU running this note is considered as a great opportunity as this draws state and national attention towards the "Biomass Testing Lab."



#### Renewable Energy Day (2/2/2011)

Drs. Cole Gustafson and Igathinathane Cannayen manned a booth that displayed various activities of the NDSU BioEpic Energy and Product Innovation Center that also depicted the "NDSU Biomass Testing Lab at NGPRL" on the Renewable Energy Day held at the Capitol Building, Bismarck, ND.



#### 2010 Research Results & Technology Conference (2/15/2011)

A short oral presentation and a poster entitled "Biomass Feedstock Process Engineering – Ongoing Research" that depicted "Biomass Testing Lab" was made by Dr. Igathinathane Cannayen on the Research Results & Technology Conference held at Seven Seas, Mandan, ND.



# Friends & Neighbors day 2011 showing the BTL presentation and activities (7/21/2011)

We were involved in various outreach activities increasing the awareness of biomass utilization, quality aspects, and necessity of a testing lab like ours (BTL). We strive to promote the visibility through presentation, demonstration, supporting lab visits, and related events. Pictures of these activities are shown in the following pages:



Presentation to the "Customers Focus Group" and lab visit conducted by Dr. Igathinathane Cannayen and Ms. Manlu Yu – Ph.D. student (July 21, 2011)





# "We Got the Beets! Tour" (8/24/2011)

North Dakota Water Education Foundation's "We Got the Beets! Tour" lead by Jean Schafer, Executive Director, ND Water Coalition, Bismarck, ND. About 30 members attended the BTL .



# "Barley Council Members" (9/7/2011)

A presentation on field about biomass preprocessing and BTL were made to "Barley Council Members" as a part of their NGPRL research field plot tour



# Local school students visit (9/15/2011)

A major local school students of 9th grade biology 20 students visited BTL and were presented a talk on biomass preprocessing and capabilities of BTL.



#### NGPRL on-site review team visit (3/27/2012)

NGPRL on-site review team of experts were presented about the activities of BTL and the collaborative research of NDSU at NGPRL (Mar. 27, 2012)



# Technical paper presented in 2012 ASABE/CSBE North-Central Intersectional Conference from the data generated from BTL (3/30/2012)

Program - Agricultural and Biosystems Engineering (NDSU)

4/29/12 8:33 PM

# NDSU NORTH DAKOTA STATE UNIVERSITY

#### AGRICULTURAL AND BIOSYSTEMS ENGINEERING

#### 2012 ASABE/CSBE North-Central Intersectional Conference

Friday, March 30, 2012

8:15 - 8:40 a.m. - Registration, Great Plains Ballroom Lobby

8:40 - 10:00 a.m. - Session 1 - Biofuels - Hidatsa Room - Moderator: dr. Igathinathane Cannayen

Change in Fermentable Sugars in Sugar Beets Stored Anaerobically, Juan M. Vargas-Ramirez, North Dakota State University, oral plus paper. RRV12100.

**Hydration Characteristics of Switchgrass, Big Bluestem, and Bromegrass**, Manlu Yu, North Dakota State University, oral plus paper. RRV12101.

Fermentation of Sugarbeets with E. coli K011: Impact of Buffer Strengthand Hydrolyzate Composition, Nurun Nahar, North Dakota State University, oral, no paper. RRV12126.

10:40 - noon - Session 5 - Soil and Water II - Rose room - moderator: dr. dean steele

Comparison of Evapotranspiration Estimates from Remote Sensing and Eddy Covariance Measurements in the Devils Lake Basin, Dean Steele, North Dakota State University, oral no paper. RRV12114.

 $http://www.ndsu.edu/aben/department/newscalendar/2012\_north\_central\_intersectional\_conference/papers/$ 

Page 1 of 2

# "Sustainable Bioenergy" online training module inclusion (4/9/2012)

Dr. Cole Gustafson about to present in the "Sustainable Bioenergy" Train-the-trainer workshop where in the online course module the activity of BTL was indicated in his contributed chapter. This gives nationwide visibility to the extension specialists, agents, researchers, and general public; Dr. Igathinathane Cannayen attended this training held at University of Missouri, Columbia.



# Presentation to Empower ND Commission (6/21/2012)

Dr. Igathi Cannayen made a presentation entitle "Biomass Testing Laboratory for Physical and Thermal Characteristics of Feedstock of North Dakota" and "Energy Beet Research-Phase II"



"Friends & Neighbors Day 2012" showing the BTL activities (7/19/2012)



# Technical paper to be presented in 2012 ASABE International Annual Meeting at Dallas Texas from the data generated from BTL (7/29-8/1/2012)



An ASABE Meeting Presentation

Paper Number: 121337358

2950 Niles Road, St. Joseph, MI 49085-9659, USA 269.429.0300 fax 269.429.3852 hq@asabe.org www.asabe.org

# Mechanical shear and tensile characteristics of selected biomass stems

#### M. Yu, C. Igathinathane\*

Department of Agricultural and Biosystems Engineering, North Dakota State University; 1221 Albrecht Blvd, Fargo, ND 58102, USA, \* Igathinathane.Cannayen@ndsu.edu

#### C. Gustafson

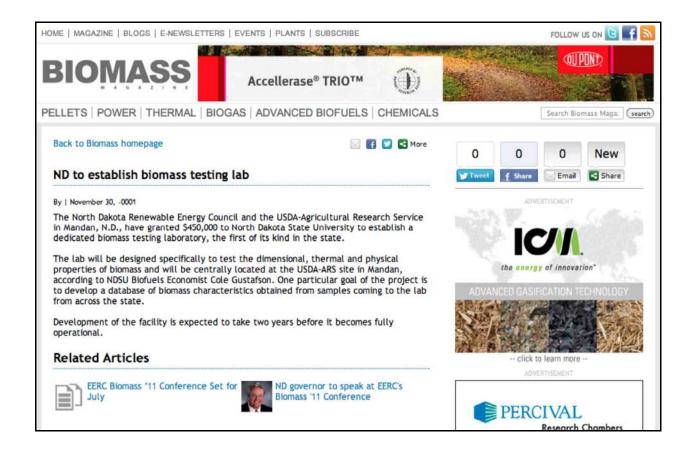
Department of Agribusiness and Applied Economics, North Dakota State University, 500 Barry Hall, PO Box 6050, Fargo, ND 58108, USA

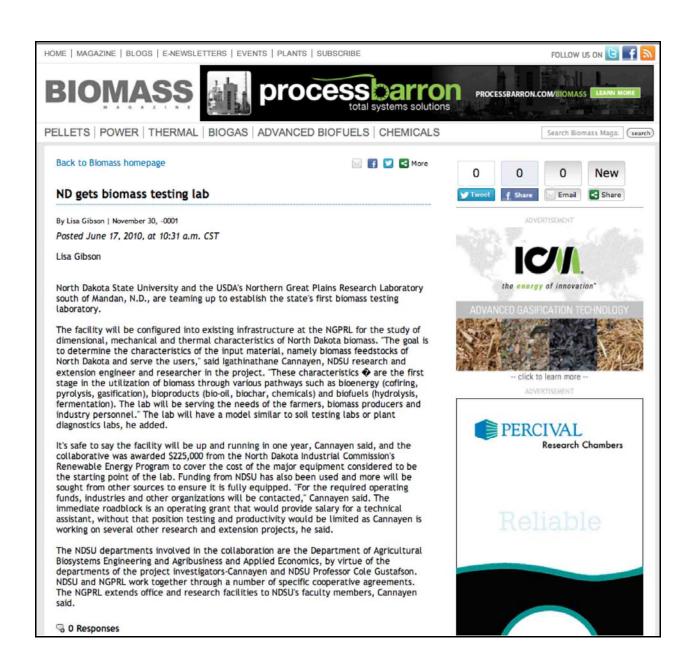
#### J. Hendrickson, M. Sanderson

Northern Great Plains Research Laboratory, USDA-ARS, 1701 10th Avenue SW, Mandan, ND, 58554, USA

Written for presentation at the 2012 ASABE Annual International Meeting Sponsored by ASABE Hilton Anatole Dallas Dallas, Texas July 29 – August 1, 2012

#### 3.7 News items on BTL





#### **NDSU News**

North Dakota State University

# NDSUNEWS

Home

First presidential candidate visits NDSU campus

NDSU shows record growth in doctoral degrees awarded in a decade --

#### Biomass Testing Lab established

Posted on April 28, 2010 by NDSU News

Cole Gustafson, NDSU biofuels economist, and Igathi Cannayen, Northern Great Plains Research Laboratory assistant professor, have received \$450,000 from the North Dakota Renewable Energy Council and U.S. Department of Agriculture's Agricultural Research Service in Mandan to establish the first dedicated biomass-testing laboratory in North Dakota

The lab will be designed to test dimensional, thermal and physical properties of biomass. Creation of the lab aligns with the NDSU Bio Energy and Product Innovation Center's (BioEPIC) goal of fostering development of a biomass industry in North Dakota. BioEPIC already has created a searchable biomass inventory so prospective investors can evaluate potential biomass supplies in different geographic locations across the state. In addition, a decision aide, Biomass Compare, has been developed to help farmers and ranchers compare the profitability of biomass production with traditional farm enterprises.

Additional funding is being sought from the U.S. Department of Agriculture to develop biomass market standards, assist agricultural producers in forming a biomass supply network and developing a hands-on mobile biomass processing display to educate potential biomass suppliers on differing harvest and processing methods.

The Biomass Testing Lab will contain four primary machines. A universal testing machine will measure the force needed to compress, shear or cut biomass. A second machine will monitor the mass and temperature of biomass as it is heated in a controlled inert environment. A bomb calorimeter measures the energy content of various biomass samples. Finally, an environmental control chamber will enable storage studies of biomass in a controlled environment of constant temperature and humidity.

The Biomass Testing Lab will be located at the U.S. Department of Agriculture – Agricultural Research Service site in Mandan.

This entry was posted in Uncategorized and tagged biomass, biomass testing, Cole Gustafson, Grant, NDSU, ndsu biofuels, northern great plains research. Bookmark the permalink.

First presidential candidate visits NDSU campus

NDSU shows record growth in doctoral degrees awarded in a decade -- (Saush)

#### Recent Posts

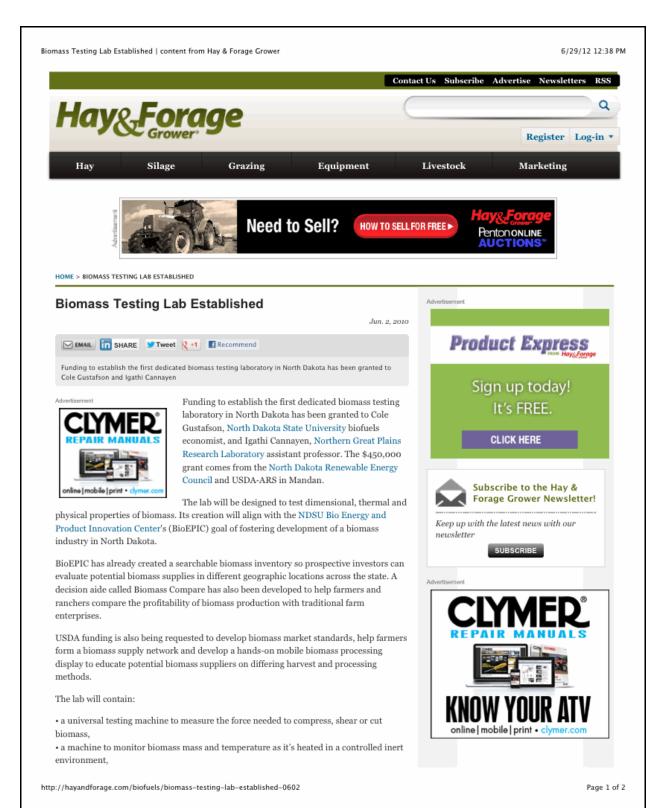
- NDSU announces spring commencement ceremonies
- Run/walk scheduled to honor fallen NDSU students, faculty and staff
- Sanford Health to be NDSU's first Campus Community Partner
- 750 NDSÚ students, alumni volunteer for The BIG Event on April 24
- NDSU Center for Child Development collects toys for children undergoing chemotherapy

Tags

agriculture arts athletics barry hall Bison Bison athletics bison mens basketball bollinger bresciani bruce bollinger construction Dean Bresciani education enrollment entertainment events family flood flood 09 flood 2009 football Hanson higher education klai hall minard collapse minard hall moms NDSU ndsu athletics NDSU Bison ndsu enrollment NDSU football ndsu president ndsu presidential search ndsu research ndus north dakota state university president President Bresciani presidential search road construction Science Cafe sports state of university volunteers

#### Archives

- May 2012
- April 2012
- March 2012





Caught In The Middle

6/29/12 12:38 PM

- $\bullet$  a bomb calorimeter measuring the energy content of various biomass samples,
- an environmental-control chamber enabling biomass storage studies in a controlledtemperature and humidity environment. The lab will be located at the USDA-ARS site in Mandan.



Round Hay Bales: What Horse Owners Should Know
5 people recommend this.

High Hay Prices Will Persist
111 people recommend this.

Weather Changes Likely In Upcoming Hay Season
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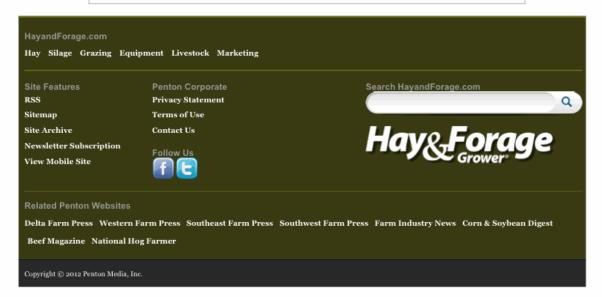
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http://hayandforage.com/biofuels/biomass-testing-lab-established-0602

Page 2 of 2

### 4. FUTURE WORK AND RECOMMENDATION

Having established the NDSU Biomass Testing Laboratory at NGPRL with four major pieces of equipment and demonstrated the measurement, analysis, modeling, and interpretation, the future work will relatively simple but more productive. Biomass samples from the immediate fall harvest from NGPRL and other samples from NDSU research stations will be subsequently studied and results will be cataloged. Future studies will include the effect of moisture content, maturity stage, anatomical component difference, effect of storage, etc., on the various physical, mechanical, and thermal properties of biomass that the BTL specializes. The results on the quality of the biomass along with statistical analysis will enable to rank and select the suitable biomass feedstocks for industrial applications.

Dr. Igathinathane Cannayen with other researchers from NDSU and "Green Vision Group" obtained a grant from NDIC for the "Energy beet Research – Phase II." Dr. Cannayen will be heading the "front-end processing of beets" component, and this work has funded position for a technician (22 months). Another three year project that is in the final stages of funding entitled "Flood Affected Wood Biomass Utilization Opportunities in North Dakota" in which Dr. Cannayen is involved and will be collaborating the ND Forest Service will have a technician/postdoc researcher. During downtime opportunities, the service of these technicians could be sought with BTL activities without affecting the primary objective of their hire. In distant future, a nominal amount will be charged to the customers that desire to use the services of the BLT just to cover the cost to make it sustainable. There is also a loud thinking and background action to get a hard funded technician attached to Dr. Igathinathane Cannayen's position from the ABEN, NDSU. If this happens the services of the BTL will be quick and more affordable.

The activities and capabilities of BTL is a strong point for ABEN to support the extramural funding proposal initiatives. The role of BTL is quite significant in bringing more visibility to the NDSU presence in NGPRL and our research activities provide us opportunity to participate in multi-university and multi-state project funding proposals. Research results generated from BTL have tremendous potential to be published as peer-reviewed articles, technical presentations, factsheets, extension activities, demonstrations, training, and popular articles. The results could be appealing to state and nationwide as well as worldwide audience, as the experiments being engineering in nature they could be easily repeated and verified. Experiences gained during the course of this project will help in future better productivity of the BTL. Significant additional results will be presented in future as an addendum to this final report.

We would like to record our gratitude and appreciation to NDIC for seeing the need and funding this significant proposal.

#### 5. SUMMARY

This comprehensive final report on the project entitled "Biomass Testing Laboratory (BTL) for Physical and Thermal Characteristics of Feedstock of North Dakota" presents background of the need; methodology followed; test material processing; description and operation principle of four major pieces of equipment; results, analysis, discussion, and interpretation; outreach activities highlighting the BTL capability; news items on the BTL; future work and recommendations; and appendices for more detailed information.

A substantial void exists among producers and processors of North Dakota biomass regarding its quality, suitability for densification, and energy applications. Evaluation of the physical and thermal characteristics of raw and processed biomass forms the important phase of evaluation of baseline data. This information guides various efficient operations of biomass processing and handling as well as aiding in development of new processes.

This two year, \$450,000 (total with 1:1 cost match) equipment grant project established a "Biomass Testing Laboratory" (BTL). Four major pieces of equipment (thermal analyzer TGA/DSC, universal testing machine, environment control chamber, and calorimeter) were purchased, installed and tested successfully. Although the installation process has faced with some unforeseen installation issues and delayed the progress, the equipments are now in order and are ready for experiments and production of research results.

Results generated from numerous experiments that were presented on appropriate sections provide good insight on the quality (physical, mechanical and thermal) characteristics of the ND biomass feedstocks. Future studies will include the effect of moisture content, maturity stage, anatomical component difference, effect of storage, etc., on the various physical, mechanical, and thermal properties of several ND biomass that the BTL specializes. The BTL has become fully functional from June 2012.

#### **APPENDICES**

# Appendix – I: Award contract and processing through NDSU

#### The Award

The Principal Investigators (PI) express their gratitude to the North Dakota Industrial Commission (NDIC), Bismarck, ND for granting the award and to the Northern Grate Plains Research Laboratory (NGPRL), USDA-ARS, Mandan, ND for providing the matching fund and the laboratory space for establishing the "Biomass Testing Laboratory." The award information and the contract (# R-008-018) released by NDIC dated 11/22/2010 is shown in Fig. A1-1.

#### Contract No. R-008-018

#### "Biomass Testing Laboratory for Physical & Thermal Characteristics of ND Feedstock"

Submitted by North Dakota State University
Principal Investigators: Gustafson and Cannayen

#### **PARTICIPANTS**

Sponsor	Cost Share
NGPRL USDA-ARS	\$125,053
NGPRL USDA-ARS	\$ 99,947 (in-kind)
North Dakota Industrial Commission	\$225,000
Total Project Cost	\$450,000

Project Schedule – 24 months Project Deliverables:

Contract Date – November 23, 2010 Status Report: November 30, 2010 Start Date – June 1, 2010 Status Report: May 31, 2011 Completion Date – May 31, 2012 Final Report: May 31, 2012

#### **OBJECTIVE/STATEMENT OF WORK:**

The goal of this project is to establish a Biomass Testing Laboratory to evaluate physical and thermal characteristics of diverse ND feedstock and the densified biomass products. The lab will be a joint venture between NDSU and USDA-ARS in Mandan. The lab will provide North Dakota with a way to measure quality of biomass feedstocks, producing a unique industry and market database of performance characteristics. The funding provided by the North Dakota Industrial Commission/Renewable Energy Program will be utilized to purchase four pieces of equipment: Thermo Gravimetric Analyzer; MTI Universal Testing Machine; Environmental Control Chamber; Bomb Calorimeter.

#### STATUS

Contract has been executed and the matching funds have been verified.

11/22/2010

Fig. A1-1. NDIC award contract R-008-018 on biomass testing laboratory

#### **Award Processing**

The award was then processed by the North Dakota State University (NDSU) through the offices of North Dakota Agricultural Experiment Station and through Sponsored Programs Administration (SPA). These offices made further inquires about the various aspects of the project administration and the PIs responded to inquiries satisfactorily. The Office of Grant and Contract Accounting of SPA processed the award, made an initial approval, and then released a revised version dated 12/16/2010 (Fig. 2. The allotted the following important information for operating the funds are, Fund Number: 46000, Control Dept: 7620, Program #: 01463, and Project#: FAR0016577.

Grantor: ND Industrial Commission Project # FAR0016  Renews Old	Project # FAR0016577
Principal Investigator:  Cole Gustafson & Igathinathane Cannayen Ag & Blosystem Engineering  Effective Date: From  06/01/2010 to 05/31/2012  Title:  Biomass Testing Laboratory for Physical and Thermal Characteristics of Feedstock in North Dakota.  Billing Type: Cost Reimburseable Fixed Price  Equipment: TGA Analyzer	Log Number   FAR0016577
### Effective Date: From 06/01/2010 to 05/31/2012   (BT) Level 2 (TD) Level 3 (BC) Level 4 (CD) Level 4 (CD) Level 5 (BC) Level 4 (CD) Level 5 (BC) Level 5 (CD) Level 6 (CD) Level 6 (CD) Level 7 (CD) Level 8 (CD) Level 8 (CD) Level 8 (CD) Level 9 (CD)	(BT) Level 2 (TD) Level 3 (BC) Level 4 (BL) Level 5 n North Dakota.    Billing Type:   Cost Reimburseable   X     Fixed Price       74,000.00     ne   60,000.00     ber   65,000.00
Bidmass Testing Laboratory for Physical and Thermal Characteristics of Feedstock in North Dakota.   Billing Type: Cost Reimburseable Fixed Price   X	Content   Cont
Cost Reimburseable   X	Cost Reimburseable X Fixed Price  74,000.00 ne 60,000.00 ber 65,000.00
BUDGET ACCOUNT #  Equipment:  TGA Analyzer 74,000.00  MTI Universal Testing Machine 60,000.00  Environmental Control Chamber 65,000.00  Bomb Calorimeter 26,000.00	74,000.00 ne 60,000.00 ber 65,000.00
Equipment:  TGA Analyzer 74,000.00  MTI Universal Testing Machine 60,000.00  Environmental Control Chamber 65,000.00  Bomb Calorimeter 26,000.00	ne 60,000.00 ber 65,000.00
TGA Analyzer 74,000.00  MTI Universal Testing Machine 60,000.00  Environmental Control Chamber 65,000.00  Bomb Calorimeter 26,000.00	ne 60,000.00 ber 65,000.00
MTI Universal Testing Machine         60,000.00           Environmental Control Chamber         65,000.00           Bomb Calorimeter         26,000.00	ne 60,000.00 ber 65,000.00
Environmental Control Chamber 65,000.00  Bomb Calorimeter 26,000.00	ber 65,000.00
Bomb Calorimeter 26,000.00	
Revised	icad
	/15EU
TOTAL DIRECT COST BUDGETED: \$ 225,000.00	\$ 225,000.00
FACILITIES & ADMINISTRATIVE COSTS 0.00% of 225,000,00 0.00	
	0.00
	0.00 VAPD \$ 225,000.00
Cost Sharing of this award required? Yes	
If yes, requirements below:	
Personnel:         Other:         Project FAR0014211 - ARS         48,053.00	
	VARD \$
USDA-ARS In-kind Match (time & space) 99,947.00	VARD \$ 225,000.00 48,053.00
	VARD \$ 225.000.00 48,053.00 77,000.00
	VARD \$ 225.000.00 48,053.00 77,000.00

Fig. A1-2. NDSU's approval of NDIC's award on biomass testing laboratory

#### **Equipment Purchase Processing**

After receiving the approval from the NDSU's SPA, we have processed the purchase of the four piece of equipment. The equipment supply quotes were obtained from the leading manufacturers of these equipment. As our requirement is specific, related to biomass, the manufacturers were meticulously identified based on the criteria such as suitability with the test material, accuracy of measurement, and cost effectiveness. Based on our selection criteria, only single supplier matched the requirements on each equipment and we have not obtained multiple quotes. The NDSU purchase procedure needs justification to this effect, and we have filed the "Sole Source Purchase Justification" forms, giving the necessary justifications and highlighting the uniqueness of the equipment. The first three equipment are unique in their configuration and were supplied by specific manufacturers, while the calorimeter was selected based on the cost effectiveness without compromising the features. Copy of the quotes and a two-page graphical description of the equipment ordered are given in Appendix-II.

#### **List of Equipment and Services Ordered**

Table A1 shows the list of equipment, spares, and services ordered so far costing \$177,306. The purchase documents for these items are being processed by the NDSU's Purchasing Department. Other supply items that will be required during testing and experiments will be purchased as required.

Table A1. List and the cost of equipment, supplies, and services ordered for the BTL

ltem	Cost
Thermal Analyzer	
TGA/DSC1/SF - High Temperature Thermogravimetric Analyzer (TGA) with	\$46,426
Differential Scanning Calorimetry (DSC) – Basic unit	
TA-SW Silver - SRARe Silver Package Ver. 10.0	9,800
Two boxes of Aluminum oxide crucibles	\$1,018
XS105DU - Analytical balance 120 g capacity, 0.1mg/0.01mg accuracy	\$4,348
EQ-TAZ2- 1year service for DSC &TGA	\$4,096
TA/FP - 1 TA Standard training for DSC & TGA	\$1,455
TGA-EWPZ2 - 12 Month extended warranty plus	\$5,486
Subtotal	\$72,631
Universal testing machine	60,000
MTI-100K Universal Testing System - 100,000 lb capacity	\$55,000
One set of 30,000 lb capacity tensile specimen grips	\$4,800
One set of Compression loading platens	\$1,800
One 20,000 lb force capacity tensile/compression load cell	\$900
On- site installation and training	\$900
Subtotal	\$63,400
Environmental chamber	
BTX-475 4cu. ft. Benchtop Temperature/Humidity Chamber	\$12,910
Heated widow with interior light	\$820
Independent overcool limit protection	\$260
Emergency stop	\$190
Mobile cart with cabinet door and built in shelf	\$1,170
Extended 2 year warranty 10% of system and options	\$1,535
Freight to ND	\$620
Start-up and training for one unit	\$2,550
Subtotal	\$20,055
Bomb calorimeter	
IKA Calorimeter C 2000 Basic V2 115; Model No: EW-50905-30	\$21,220
Total	\$177,306

The balance of funds as of June 08 is shown in figure A1-3. Additional items such as specialty gases for TGA/DSC and calorimeter, regulator, UTM moving expenses, fabrication charges for UTM attachments (double shear device and modified Warner-Bratzler cutting device), ECC trays, inlet cooling water pressure regulator for calorimeter, pellet press, and calorimeter supplies worth about \$11,265 was also purchased.

			Spons As o	PI Report or Funding Repo f June 08, 2012	ort			1 06/20/2012 20:01:55
AWARD: AWARD NAME: AWARD DEPT: AGENCY: AWARD PI: AWARD START DA:	7620 AES A ND Industr Gustafson,	esting Laborato ag & Biosystems rial Commission Cole R	s Eng	al and Thermal	PROJECT: PROJECT NAME: PROJECT DEPT: PROJECT STATUS: F&A RATE: PROJECT PI: PROJ START DATE	7620 AES Ag ACTIVE 0.000 Gustafson,Co	ing Laborator & Biosystems	y for Eng
AWARD END DATE					PROJ END DATE: FUND CODE:		ch-level 4	
EXPENSE DESCRI	PTION	BUDGET	CURRENT MO EXPENSES	CUMULATIVE EXPENSES		OUTSTANDING ENCUMBRANCE	AVAILABLE BUDGET	PERCENT AVAILABLE
Salaries-Reglr Salaries-Other Salaries-Temp Salaries-OT Salaries-Fac Salaries-Grad <i>I</i> Benefits	Asst	0.00 0.00 0.00 0.00 0.00	0.00 0.00 0.00 0.00 0.00	0.00 0.00 0.00 0.00 0.00 0.00	0.00 0.00 0.00 0.00 0.00 0.00	0.00 0.00 0.00 0.00 0.00	0.00 0.00 0.00 0.00 0.00 0.00	0.00 0.00 0.00 0.00 0.00 0.00
Benefits TOTAL PERSONNEI	L EXPENSE	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Travel Supplies Food and Cloth: Bldg,Grounds,V Miscellaneous : Office Supplies	ehicle Supply Supplies	0.00 0.00 0.00 0.00 0.00	0.00 0.00 0.00 0.00 0.00	0.00 0.00 0.00 0.00 0.00	0.00 0.00 0.00 0.00 0.00	0.00 0.00 0.00 0.00 0.00	0.00 0.00 0.00 0.00 0.00	0.00 0.00 0.00 0.00 0.00
Postage Printing Minor Equipment Utilities Insurance Rents & Leases	Ŀ	0.00 0.00 0.00 0.00 0.00 0.00	0.00 0.00 0.00 0.00 0.00	0.00 0.00 0.00 0.00 0.00	0.00 0.00 0.00 0.00 0.00	0.00 0.00 0.00 0.00 0.00	0.00 0.00 0.00 0.00 0.00	0.00 0.00 0.00 0.00 0.00
Repairs IT Communication Professional De Operating Fees Professional Fe	evelopment and Services se & Services	0.00 0.00 0.00 0.00 0.00	0.00 0.00 0.00 0.00 0.00	0.00 0.00 0.00 0.00 0.00	0.00 0.00 0.00 0.00 0.00	0.00 0.00 0.00 0.00 0.00	0.00 0.00 0.00 0.00 0.00	0.00 0.00 0.00 0.00
Medical, Dental Miscellaneous B Subcontracts Interest Expens Cost of Goods	Expenses	0.00 0.00 0.00 0.00	0.00 0.00 0.00 0.00	0.00 0.00 0.00 0.00	0.00 0.00 0.00 0.00 0.00	0.00 0.00 0.00 0.00	0.00 0.00 0.00 0.00 0.00	0.00 0.00 0.00 0.00
TOTAL OPERATING	G EXPENSE	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Waivers, Schola		0.00	0.00	0.00	0.00	0.00	0.00	0.00
TOTAL WAIVERS,	SCH & FELLOW	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Capital Asset E Equipment	Expense	0.00 225,000.00	0.00	0.00 188,571.41	0.00	0.00	0.00 36,428.59	0.00 16.19
TOTAL EQUIPMENT	r expense	225,000.00	0.00	188,571.41	0.00	0.00	36,428.59	16.19
TOTAL DIRECT CO	OST	225,000.00	0.00	188,571.41	0.00	0.00	36,428.59	16.19
F&A		0.00	0.00	0.00	0.00	0.00	0.00	0.00
TOTAL F&A EXPE	NSE	0.00	0.00	0.00	0.00	0.00	0.00	0.00
GRAND TOTAL		225,000.00	0.00	188,571.41	0.00	0.00	36,428.59	16.19
		Report ID: SH	FRNDU101PC					

Fig. A1-3. NDSU sponsor funding report showing the expenditure incurred and balance available as of June 08, 2012

Representation to utilize to balance funds \$36,428 (Fig. A1-3), originally meant for equipment (> \$5000), for purchase of "supplies" (items <\$2500) was sent to NDIC. Also an extension of three months – up to September, 2012, was also requested. These permissions will allow to test the various biomass samples that will be made available during the fall of 2012 and beyond. We are hopeful that this permission will be received.

Second Amendment to Contract No. R-008-018

This amendment is between the State of North Dakota acting by and through its Industrial Commission, hereafter called Commission, and the North Dakota State University, hereafter called Contractor.

This amendment is to Contract No. R-008-018 which was originally executed by the Commission on November 23, 2010 and then further amended on April 13, 2012. Requests have been made to allow the filing of an additional report to include test results for work conducted in the laboratory utilizing the equipment purchased with these dollars; flexibility in expending \$36,428.59 for additional supplies and equipment related to the equipment previously purchased and the naming of a new Principal Investigator due to the untimely death of Dr. Cole Gustafson. Contract No. R-008-018 Paragraphs 2 and 3 are amended to read as follows:

#### 2. Scope of Work

- a. The Contractor agrees to purchase the following four pieces of equipment:
  - · Thermo-Gravimetric Analyzer;
  - · Universal Testing Machine (high capacity MTI model)
  - Environmental Control Chamber
  - Bomb Calorimeter

in order to facilitate the establishment of a testing laboratory to conduct the work described in the amended Exhibit A, entitled Biomass Testing Laboratory for Physical and Thermal Characteristics of Feedstock of North Dakota, which is attached to this Agreement and is made a part of it. The Contractor is further authorized to utilize funds in the amount of \$36,428.59 for supplies and materials including additional replacement parts for the purchased equipment related to the work being conducted on the four pieces of equipment listed above.\* Exhibit A is amended to name Igathinathane Cannayen as the Principal Investigator due to the untimely death of Dr. Cole Gustafson.\*

b. Contractor agrees to provide Reports for the work mentioned in Paragraph 2a as follows:

Status Report:

November 30, 2010
Status Report:

Final Report:

April 30, 2012
June 30, 2012
Amended Final Report

October 31, 2012\*

The Reports shall be in compliance with 5.11 of the Industrial Commission/Renewable Energy Development Program Policies. If requested, the Contractor will provide a tour of the facility where the work is being conducted to the Council and/or staff. Each Report must provide documentation verifying the receipt of the private matching funds. The project data and reports shall be provided to the Department of Commerce & Renewable Energy Council in both electronic and hard-copy formats with permission for unrestricted distribution. The electronic versions shall be in a suitable format for hosting on the Department of Commerce and Renewable Energy Council websites. Specifically, the Final Report must include a single page project summary describing the purpose of the project, the work accomplished, the project's results, and the potential applications of the project.

#### 3. Consideration

a. For performing the work the Commission agrees to grant to Contractor an amount not to exceed \$225,000 according to the following schedule:

Upon execution of the contract and verification that matching funds of \$225,000 have been identified \$22,500.00

Upon receipt and consideration of receipts for purchased

equipment identified above \$166,071.41\*
Upon filing of amended final report \$36,428.59\*

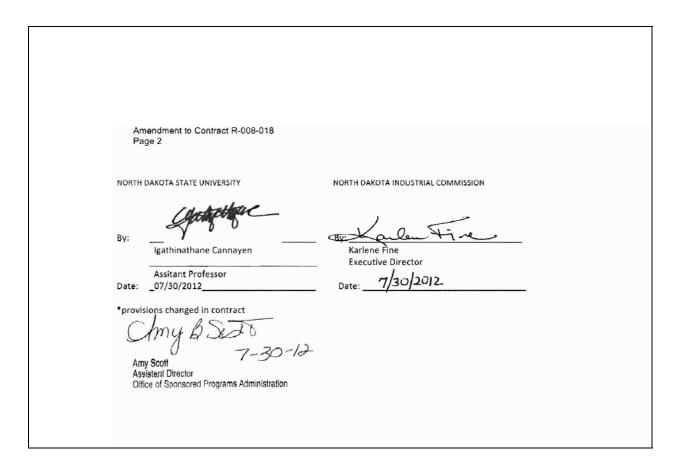


Fig. A1-4. Received amendment from NDIC for spending the balance funds for supply purchase

We are thankful to NDIC for approving this amendment (Fig. A1-4) towards the purchase of supply items of BTL that will help producing results efficiently.

# North Dakota State University FI Report Sponsor Funding Report As of October 22, 2012.

Fage No. 1 Bun Date 10/22/2012 Bun Time 08:34:17

AGENT: ED Industrial Commission
ANAID FI: Cannayer, Igathinathane
ANAID END DATE: 06/01/2010
ANAID END DATE: 10/31/2012

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Biomass Testing Leboratory for Physical and Thermal
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0.00	0.00	0.00	0.00	00.0	0.00
0.00	0.00	0.00	0.00	00.00	0.00
9 2,492.93	2,492.93	0.00	7,872.08	26,063.58	71.55
a c.aa	a.aa	a.aa	0.00	0.00	a_aa
a 6.40	0.00	0.00	<b>a.</b> aa	0.00	a.aa
a c.aa	0.00	0.00	0.00	0.00	0.00
i 6.00	188,571.41	0.00	0.00	0.00	a.aa
L C.QQ	188,571.41	a.aa	0.00	0.00	a.aa
2,452.53	191,064.34	Q.00	7,672.98	26,063.58	11.58
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	191,064.34	0.00	7,872.08	26,063.58	11.58
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Report IB: SFREWIGIPC

Fig. A1-5. NDSU sponsor funding report showing the expenditure incurred and balance available as of October 22, 2012

This report is not showing some of the funds being utilized in supply purchases (shown below) that are being processed.

Outstanding invoices	Details	Amount
Lowe's, Fastenal, Staples	Office and fabrication supplies	\$102.14
Cole Parmer	Laboratory supplies	\$8,388.13
Rubbermaid	Laboratory trolley	\$178.66
Fisher Scientific	Laboratory supplies	\$38.48
Old Will Knotts Scale	Moisture measuring balance	\$1756.00
Praxair	Specialty gases refill	\$624.54
Instrumart	Electrical energy measurement	\$2,187.00
	Total	\$13,274.95

# Appendix – II: Equipment quote and literature

# A. Thermal Analyzer – TGA/DSC

# METTLER TOLEDO

QUOTE

Quote #: BW1-11-01-0007

Date: 01/31/2011

1900 Polaris Parkway Columbus, OH 43240 Phone (800) 523-5123 Fax (614) 438-4900 www.mt.com Blaine Weddle MatChar Instrument Sales Spec Phone: (847) 608-1355 Fax: (614) 985-9091 Blaine, Weddle@mt.com

Quote To: Ship To:

Created By:

North Dakota State University Igathi Cannayen 1221 Albrecht Blvd Fargo, ND 58102 Phone: (701) 667-3011 Fax: (701) 667-3054

Phone: (701) 667-3011
Fax: (701) 667-3054
Email: Igathinathane.Cannayen@ndsu

North Dakota State University Igathi Cannayen 1221 Albrecht Blvd Fargo, ND 58102 Phone: (701) 667-3011 Fax: (701) 667-3054

Email: Igathinathane.Cannayen@ndsu.edu

# Item# Qty. Description Unit Disc. Extended TGA/DSC1/SF 1 TGA/DSC1/SF \$54,619.00 15.00% \$46,426.15

HIGH TEMPERATURE THERMOGRAVIMETRIC ANALYZER WITH DSC Part of Metitor-Tolodo's new Thermal Analysis Excellence line of instruments, the TGA/SSCT incorporates the island technology, with our traditional medium design along with high-quality Swiss precision mechanics. This unit includes our standard temperature famiciae with a temperature range from ambient up to 1100° C and can accommodate sample volumes up to 1500L. See below for your choice of sample holders and possible balance configurations. In addition to measuring the mass change of your samples using the most advanced balance technology in any TGA instrument, this model see adds the depactify to measure the hear flow (DSC signal) from transitions in your samples as well. This means that with this instrument, it is possible to easily measure endoffermed and exothermic transitions that do not result in a loss of mass (e.g. polymorphism, melling, crystalization, etc.) flus revealing considerably more information about your sample than a possible in a standard TGA-only instrument. The DSC signal sits allows to the use of meeting point standards for easiler and more accounted temperature and heat flow calibration than is possible using the magnetic Currie-point standards that are used in simple TGA-doty instruments. The

Instrument Includes: Operating instructions, Calibration Rt. Indium, Abminum, and Gold for temperature calibration, Crucitile handling set, 70 L. Alominum Oxide crudibles (20 pcs.) with removable list, TGA demo crudible set, power cable and a LNA cuteff to institute it commissionation.

Optional items include. MaxRes, robotic sample changer (with lid pieceling and removal), various balance configurations, and various gas flow control options. Please inquire for more details.

#### 51142095

#### SDTA® Small Furnace

METILER TOLEDO Patented Single Differential Thormal Analysis (SDTA) technology provides the best simultaneous performance at an attractive price. The SDTA enteror provides an ordine heat flow simultaneously with your typical mass change information which can be useful to help interpret results and to calibrate the system using metting point standards. Bocause there is only one sample, you do not need to awary about thermal gradients or eccentricity of finaling. The single sample also allows the furnace to be smaller for better temperature control. The SDTA sensor provides an on-line heat flow signal with a resolution of 0.5 mW.

#### MX1-TGA

MX1-TGA MICRO BALANCE

#### "METTLER TOLEDO Inside"

Integrated patented METTLER TOLEDO micro balance with a 1000 mg sample capacity and confinuous resolution of 1 ug over the entire weight range without range switching. Parallel guided technology for position independent weighing eliminates weighing errors, due to the thermal expansion of the sample support. Readability 1ug/ Repostability 0.9ug/ Linearity +1-4ug

#### F25MC

#### 200 WATT CRYOSTAT

Constant temperature recirculation baths are used with the TGA, TMA, and optionally for the high pressure DSC to cool the furnace and maintain a constant temperature of the balance (TGA) or measuring cell (TMA). Maintains temperature at set temperature +1-0.01°C.

#### 51142671

#### GC200 DUAL MASS FLOW CONTROLLER (4 GAS)

The GC200 MASS FLOW CONTROLLER allows you to switch and control the flow rate of up to three different purge gases for your experiment. Also include a second mass flow controller which controls the protective gas for the balance or low temperature DSC cell. The Mettler-Toledo FlexCali® software ensures that your calibration constants are automatically changed during your measurement to match the purge gas that is being used in each segment of your measurement to match the purge gas that seeking used in each segment of your measurement to control calibration constants, even if your purge gas or heating rate happen to change during your temperature program.

#### TA-SW Silver

#### 1 TA-SW Silver

#### 51143646

#### STARe Silver Package Version 10.0

The STARe Silver is a comprehensive software package which includes everything you need to get started with the STARe software. Whether you are a Research & Development scientist or a Quality Control Technician, the STARe Silver package has the vensetility to meet all of your current and future software needs. Specific software options are included and quoted as needed.

The Silver software package includes the capability to perform the following evaluations: onset, endest, pask integration, peak temperature, step calcutations, data tables, curve min and max, and normalization to sample size (Wig or 56). Curves can be picted vs. time, reference temp, or sample temp.

#### Includes

- \* Base Software
- Routine Experiment and Method Editor
- Advanced Mathematics
- Advanced Method Window
- Advanced Experiment Window
- Install Plus\*
- \* User Rights
- Automatic Result Evaluation with Pass/Fail determination
- Conditional Experiment Termination
- Advanced Report Generator

#### PC Requirements

-Recommended: Windows XP, 500MHz CPU, 256MB RAM, 2.0GB Hand disk

-Required: Windows XP, 233MHz, 128MB RAM, 1.2GB Hard Disk

#### Includes FlexCal®

FlexCel is offered only by Mettler-Toledo and ensures that your results are always accurate regardless of experimental conditions such as pan type, purge gas, or heating rate. Since factors such as the heat capacity and thermal conductivity of your pans and purges gases will affect your results, other manufactures require that you must calibrate your instrument under the same conditions that you will be running your experiments, every time you change one of these parameters. At Mettler-Toledo, we have measured the effects of using different crucibles & purge gases and our completely automatic Total Calibration method measures the effect of the heating rate. The tesult is that your results are always accurate no matter what crucible, purge gas or heating rate you are using. FlexCel saves you the time of heaving to perform and maintain different calibrations for your instrument and its very important when using different heating rates or purge gases in the same experiment.

#### 119459

#### SW-ADVANCED TGA EVALUATIONS

Adds evaluations for content and stoichiometric calculations.

\$19,601.00 50.00% \$9,800.50

24123	Standard aluminum oxide TGA 70ut, crucibles.	\$355.00 TS.00 /S	\$1,010.00
XS105DU	1 XS105DU	\$5,436.00 20.00%	\$4,348.80
	Analytical Balance 120g/41g x. 1mg/ 01mg. The new XS105DU uses innovative technology to increase productivity and accuracy in the laboratory. The new SmartGrid weigh pan is suspended from the back of the balance to promote repid and accurate analytical weighing by allowing air to pass feety through the slotted grid. Like any weigh pan, the SmartGrid lifts out for easy cleaning- but XS takes it to a new level. All weigh cell connection points are above the weigh pan, any spilled sample fails directly onto the fully sealed base plate, not interfering with the weighing. Simply remove the SmartGrid and wise down the belance.		
EQ-TAZ2	1 IQ/QQ of DSC, TGA or TMA	\$4,096.00	\$4,096.00
	Service includes:  * All travel and labor for the installation and certification of your new instruments.  * Your system will be unpacked, setup and installed by a factory-trained METTLER TOLEDO service engineer.  *instrument checks and calibrations will be performed as necessary to ensure that the system is performing to METTLER TOLEDO specifications and your log book will be completed per our SOP's.		
	In addition, please note:  *An EQ typically takes an additional 1/2 day compared to a normal installision.  *The Pre-installation Checklist needs to be signed and completed prior to installation, or any additional time required for the installation will be billed at our standard labor rate.  *IGNOS on and existing installed unit requires a PM Maintenance Contract to insure the instrument will pass the qualification.		
	**Any part or extended labor required to get the instrument to pass may require		
TRAINING TA/FP	additional charges.  1 TA STANDARD TRAINING (Includes DSC, TGA, TMA, FP)	\$2,911.00 50.00%	\$1,455.50
	Following the installation there will be one day of training depending on the customer's specific needs and outrent proficiency with the technique. The service engineer of a thermal analysis specialist will spend time on-site training the users (maximum 5) how to use and take care of the system. Typically you will neceive one day of training immediately after installation and the training will be completed at a later date to allow the users to become familiar with the systems, items to be covered are:		
	-Operation of instrument(s)		
	Sample preparation     Analysis and interpretation of data     Calibration of Instrument     Routine Maintenance of hardware and software		
	This training will also include on-site consulting time to run any desired samples and help with method development for typical samples that are to be analyzed on the instrument.		
Optional Items (N	(OT Included in Quote Total)		
TGA_EWPZ2	1 12-Month Extended Warranty Plus	\$5,486.00	\$5,486.00
	Protect your investment with METTLER TOLEDO's Exclusive Extended Warranty		
	*Includes one (1) Preventative Maintenance visit *This economical program covers all repair costs during your 2nd year of ownership *Parts and labor are included at no additional charge *Onsite repair of instrument at no additional charge		
	*Covers defects in material and workmanship	W	
TMA_EWP2Z2	1 24-Month Extended Warranty Plus 2 PMs Protect your investment with METTLER TOLEDO's Exclusive Extended Warranty Plus	\$6,738.00	\$6,738.00
	*Includes Two Preventative Maintenances  *This economical program covers all repair costs during your 2nd and 3rd year of ownership  *Parts and labor are included at no additional charge  *Onsite or Service Center repair based on customer location  *Covers defects in material and workmanship		
	The same and the s	Quote Total	\$67,145.25
		water i trust	407,170,20

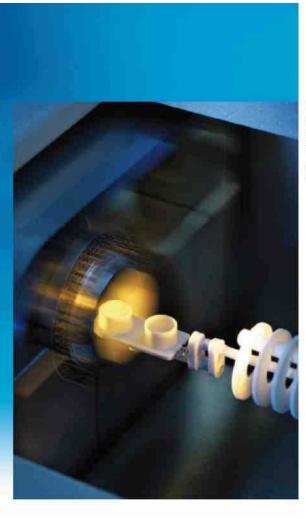
\$599.00 15.00%

\$1,018.30

2 ALUMINUM OXIDE CRUCIBLES, 70 UL (20 PCS.)

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TGA/DSC 1 STAR\* System Innovative Technology Versatile Modularity Swiss Quality



# Thermogravimetry

for Unmatched Performance



# **Excellent Performance**

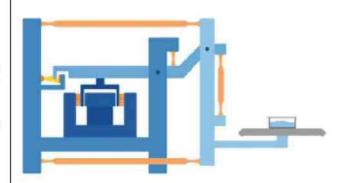
Over the Whole Temperature Range

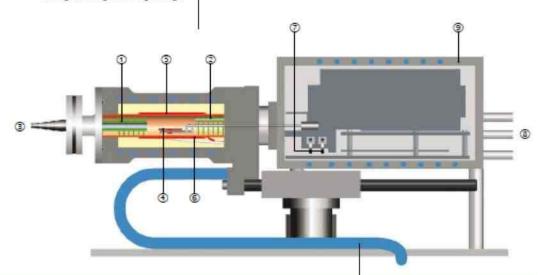
# Parallel-guided balance

The parallel-guided balance ensures that the position of the sample does not influence the weight measurement, if the position of the sample changes during meiting, no change in weight occurs.

#### Outstanding weighing performance

No other TGA can measure up to 50 million resolution points continuously - weight changes of a 5-gram sample are determined to 0.1 µg. This means you can measure small and large samples with the same high resolution without having to change the weight range.





#### Thermostating

The balance cell is thermostated to minimize environmental influences. The cryostat is also used to rapidly cool the turnace.

#### Key

- 1 Baffles
- 2 Reactive gas capillary
- 3 Gas outlet
- 4 Temperature sensors
- 5 Furnace heater
- 6 Furnace temperature sensor
- 7 Adjustment ring weights
- 8 Protective and purge gas connector
- 9 Thermostated balance chamber

### **Universal Testing Machine**

# Measurements Technology, Inc.

4240 Loch Highland Pkwy. Roswell, GA 30075 770-587-2222 FAX: 770-592-3385

North Dakota State University NDSU Dept. 7620 Fargo, ND 58108

Quotation No. 110201 February 2, 2011 until May 2, 2011

#### Attn: Prof. Igathinathane Cannayen

Measurements Technology Inc. is pleased to offer for consideration for purchase the MTI-100K Universal Testing System

as priced and described in the following cost and technical proposal.

#### MTI-100K Universal Testing System:

\$ 55,000.00

#### Included Equipment, Accessories, Features and Support Equipment:

- ⇒ 100,000 pounds capacity high-stiffness Floor Model test frame with calibrations.
- ⇒ MTI C-DAS Pentium IV Data-Acquisition and Analysis and Control System.
- ⇒ One tensile/compression Universal Load Cell up to full frame capacity.
- ⇒ One tensile universal joint and linkage rods to adapt to tensile grips.
- ⇒ One MTI AD-24 Automatic 24-bit Load signal conditioner subsystem.
- ⇒ One MTI AD-24 Automatic 24-bit Deflectometer/Strain Extensometer signal conditioner subsystem.
- ⇒ Crosshead displacement measurement and reporting to a resolution of 0.00001 inches.
- ⇒ Installation and operation training manual. (on-site training is optional)
- ⇒ Testing and Analysis software to support all testing as later described.
- ⇒ All MTI data acquisition and control software, including source code, is furnished.

(The list of standard test programs follows later. Included accessories are described in detail in the following sections of this quotation.)

#### Available Accessories and Options:

Set of 15,000 pound capacity tensile specimen Grips \$ 3,920,00 or, one set of 30,000 pound capacity tensile specimen Grips 4,800.00 or, one set of 60,000 pound capacity tensile specimen Grips 6,250.00 One set of Compression loading platens \$ 1,800.00 20,000 pounds force capacity tensile/compression load cell 900.00

(100,000 pounds force load cell is included in the system price)

S 900.00 per day plus travel expenses On-site Installation and Training

#### Terms:

FOB Point is Roswell, Georgia, USA. Shipment shall be 6 - 8 weeks ARO. All duties, taxes and shipping charges are paid by the customer. Warranty period is two years from date of installation.

35% down payment at placement of order. Remaining balance net 30 days after system shipment.

# MEASUREMENTS TECHNOLOGY, INC.

### M. Duggan

Michael F. Duggan President

www.mti-atlanta.com

meastech@mindspring.com

# MTI-100K



450 kN (100,000 Pounds) Force Capacity Automated Universal Testing System

# Optional Equipment:

#### Tensile Wedge Grips:

A set of 15,000 or 30,000 or 60,000 pound capacity Wedge Grips can be provided. A set of cross-hatch file-face flat inserts is included with the grips. Optional inserts can be furnished for griping circular specimens. 15K grips weigh 7 lbs. each. 30K grips weigh 18 lbs. 60K grips weigh 42 lbs.



#### Compression Platens with Spherical Seat

A set of 6-inch diameter hardened compression platens can be provided. The upper platen is attached directly to the load cell mounted on the bottom of the traveling crosshead. The lower platen, with spherical alignment seat, rests on the bottom fixed crosshead. Platen surfaces are 2-inch thick surface finished steel.



#### Load Cells:

Additional load cells of 20,000 pounds capacity can be provided.

#### **Environment Control Chamber**



ESPEC NORTH AMERICA, INC. 4141 Central Pertwey, Hudsonville, MI 49426 977-00-ESPEC 618-896-6100 - fax 616-896-6150 www.mspec.com



SALES REP#: QUOTATION #: REVISION #:

WTM-MN MP020311-04

SHIPPING TERMS DATE Feb. 3, 11 FOB Ori FOB Origin, Hudsonville, MI 30 Days Net 30 Days (Subject to credit approval)

Igathinathane (Igathi) Cannayen, Ph.D. North Dakota State University NDSU Dept. 7620 PO Box 6050 Fargo, ND 58108-6050

(701)667-3011



#### Quotation

#### BTX-475 4 CU. FT. BENCHTOP TEMPERATURE/HUMIDITY CHAMBER

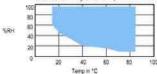
\$12,910.00 Freight to ND ... \$620.00

#### **GENERAL SPECIFICATIONS**

Temperature range: Temperature cycling rate:

Humidity range:

-70°C to 180°C (±0.5°C fluctuation at control sensor after stabilization) 2.75°C/m (empty chamber, per IEC 80088 3-5, at supply sensor) Actual change rate depends on testing range and test sample load 10 to 95% RH per chart (without live load)



Interior volume:

19.6" x 15" x 23.6" (W x D x H) 30" x 33.5" x 47" (W x D x H) 208/230V 1Ø 60Hz 12A full-load (NEMA 6-20 plug) Interior dimensions: Exterior dimensions:

Power supply source: Filtered water supply:

4 cubic feet

0.2 to 10 µS/cm resistivity Filtered to 5 microns, max 2mg/L free chlorine

3/8" NPT connection (Connection and water testing by buyer) 3/8" NPT connection (gravity strain) Condensate Drain:

10 to 25°C for assured performance, 5 to 35°C allowable and free Room Temperature:

of sharp temperature fluctuations. (0.1°C/min or 3°C/30 mins.)

#### STANDARD FEATURES

#### **CHAMBER DESIGN FEATURES:**

- · High performance and quiet operation (65 dBA)
- Space-saving footprint, designed for flush installation to wall
   Stainless steel exterior and durable thermoformed plastic door
- · Full thermal break around doorframe
- . One 2\* diameter cable port on left, with flexible silicone plug
- Three levels of overheat protection
- Detachable eight-foot power cord with plug
   Accurate wet/dry-bulb humidity measurement system is easy to maintain (solid-state sensor may be substituted at no charge)

#### WATLOW F4 PROGRAMMER/CONTROLLER

- · Popular PtD controller for test chambers
- · Stores 40 different profiles (total 256 steps)
- · Step types include: ramp, soak, jump, auto-start, and end
- RS-232C Modbus interface (free standard 'configurator' software via download)

Not for use with specimens which are explosive or inflammable, or which contain such substances. To do so could be hazardous, as this may lead to fire or explosion. Chambers are not designed to auto defrost. Based on test conditions and duration of testing, periodic manual defrosting maybe required. Does not apply to humidity mode.

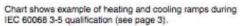
MP020311-04 February 3, 2011

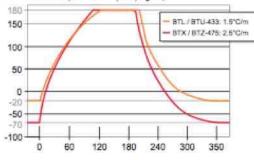




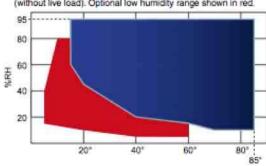
# SPECIFICATIONS - 4 cu. ft. MODELS

Model	BTL-433	BTX-475	BTZ-475	BTU-433		
Temperature Flange	-20 to 180°C (-4 to 354°F)	-70 to 180°C (-100 to 354°F)		-20 to 180°C (-4 to 354°F)		
Change Rate (per IEC 60068 3-5)	1.5°C/m heat-up 1.5°C/m cool-down	2.75°C/m heat-up 2.5°C/m cool-down				1.5°C/m heat-up 1.5°C/m cool-down
Temperature Fluctuation		± 0.5°C, per IEC 60068 3-5				
Temperature Gradient		± 1.5°C (up to 100°C)	per IEC 60068 3-6			
Live Load Capacity	190 W to 0°C	300 W to	-40°C	190 W to 0°C		
Humidity Range	10 to 95% RH, p	er chart below		÷		
Humidity Fluctuation	±5% FIH per IEC 60068 3-5 eair	Constitution of the Consti		ě		
Interior Volume		4 cu. ft (	110 L)			
Interior (W x D x H)		19.6" x 15" x 23.6" (5	00 x 380 x 600 mm)			
Exterior (W x D x H)		30" x 33.5" x 47" (76	0 x 850 x 1150 mm)			
Heaters	500 W	1000 W		500 W		
Refrigeration	1/3-hp x1 (single-stage)	3/4-hp x2 (	cascade)	1/3-hp x1 (single-stage		
Air Circulation		200 cu. ft./min.	(5.6 m3/min.)			
		Site Requirements				
Power	120V 1ph 60Hz, 16A max.	208/230V 1ph 6	OHz 12A max.	120V 1ph 60Hz, 16A ma		
Weight	350 lbs. (160 kg)	550 lbs. (	250 kg)	350 lbs. (160 kg)		
Sound Level		65 dBA (1 meter	away from door)			
Heat to Room	2000 BTU/Hr	3000 B	TU/Hr	2000 BTU/Hr		
Gravity Drain		3/8*1	IPT			
Humidity Water Supply	Supply: 3/8" NP Typical usage: 0 Purity required: 0.2 µ	.5 gailons/day		-		
Room Temperature	Performa	nce assured when room terr	p. is steady, 18 to 27°C	(65 to 80°F)		





Controlled humidity range for BTL-433 & BTX-475 models (without live load). Optional low humidity range shown in red.



7

#### Calorimeter

U.S.A.



NORTH DAKOTA STATE UNIV 1221 ALBRECHT BLVD FARGO ND 58105

# Quotation

Phone: 1-800-323-4340
Fax: 1-847-247-2929
E-mail: SALES@COLEPARMER.COM

WWW.COLEPARMER.COM

#### Page 1 of 2

Quote#	Customer RFQ
102030283	TELECON 2/03/11
Created	Validity
02/03/2011	ALL PRICES VALID 60 DAYS

#### Shipping Address 537136-01

Billing Address 057793-38

NORTHERN GREAT PLAINS RESEARCH LABS 1701 10TH AVE SW MANDAN ND 58554 U.S.A.

# Prepared For

IGATHINATHANE CANNAYEN PHONE: 701-867-3011 EMAIL: IGATHINATHANE,CANNAYEN@NDSU.EDU

#### Thank you for the opportunity to serve you!

Payment Terms: NET 30 DAYS. FREIGHT CHARGES ARE NOT INCLUDED IN THIS QUOTE BUT WILL BE ADDED TO YOUR INVOICE, IF APPLICABLE.

1-3 DAYS AFTER RECEIPT OF ORDER IF ITEMS ARE IN STOCK. SPECIFIC DELIVERY INFORMATION IS LISTED IN THE LINE ITEM DETAIL BELOW. **Delivery Terms:** 

Shipment Method: FEDEX-GROUND

	Line	Qty	MOU	Item#	Description	Unit Weight	Current Lead Time	Unit Price		Extended Price
1	1	1	EA	EW-50905-30	CALORIMTR C2000 BASIC V2 115V	185.000 LBS	17 Days	\$ 21,220.00	S	21,220.00



ESTIMATED PRODUCT NET WEIGHT (LBS): 185 FOB: SHIPPING POINT US\$ 21,220.00

ESTIMATED PRODUCT NET WEIGHT (KGS): 84 ESTIMATED PRODUCT GROSS WEIGHT (LBS): 213 ESTIMATED PRODUCT GROSS WEIGHT (KGS): 96 ESTIMATED PRODUCT GROSS SIZE (CUBIC FEET): 32.03

ORDER NOW

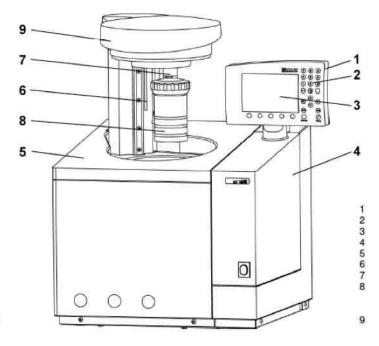
#### NOTES:

TO PLACE AN ORDER FOR THE ITEMS ON THIS QUOTATION, PLEASE CONTACT A CUSTOMER SERVICE REPRESENTATIVE AT 1-800-323-4340 OR YOU MAY USE THIS QUOTATION TO PLACE AN ORDER AT WWW.COLEPARMER.COM

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# 4.1 C 2000 basic



Control panel
Keyboard
Display
Electronics unit
Measuring cell
Temperature sensor
Oxygen filling device
Decomposition vessel
(Accessory, please
order seperately)
Measuring cell cover

C 2000 basic

# C 2000 basic Version 2

For determination of calorific values of solid and liquid fuels. Halogen resistant and catalytic activated decomposition vessel for simultaneous decomposition of halogens and sulfur included.

# Consisting of:



C 5012 Decomposition vessel, halogen resistant

Consumables for installation, calibration and unit start up are also included with delivery.

C 2000 basic Version 2	Ident. No	
230 V / 50-60 Hz	8801900	
115 V / 50-60 Hz	8801901	

# Appendix – III: Mathematical modeling of hydration characteristics of biomass

Storage characteristics of biomass are strongly connected with the moisture content, as dry material store well and moist will have shorter shelf life. Thus study of hydration (the rate at which moisture absorbed by the material) when biomass exposed to an environment of specified temperature and RH relates to the storage characteristics. Moisture absorption hydration modeling is fitting the observed moisture absorption characteristics (moisture vs time using the environment control chamber studies) in mathematical models that were meant to simulate the observed characteristics. This section presents some of the sorption kinetics hydration models studied.

# **Exponential Model**

$$\begin{split} \frac{M_e - M}{M_e - M_0} &= \exp(-k_e t); \\ M &= M_e \cdot (M_e - M_0) \times \exp(-k_e t) \end{split}$$

Where  $M_e$ = predicted equilibrium moisture content of material selected in dry basis (d.b.), M = instantaneous moisture content of material (d.b.) at any time t,  $M_0$  = initial moisture content of material selected (d.b.),  $k_e$  = sorption rate constant (h<sup>-1</sup>) of exponential model, and t = time of sorption (h).

# Page Model

$$\frac{M_e - M}{M_e - M_0} = \exp(-kt^n);$$

$$M = M_e - (M_e - M_0) \times \exp(-kt^n)$$

Where  $k = \text{sorption rate constant (h}^{-1})$  of Page model, and n = exponent (dimensionless) of Page model.

# Peleg Model

Peleg (1988) proposed a simple non-exponential equation to describe sorption behavior of food materials:

$$M = M_0 + \frac{t}{k_1 + k_2 t}$$

Where M is moisture content at time t (% d.b.);  $M_0$  is initial moisture content (d.b.); t is socking time (h);  $k_1$  and  $k_2$  are constants. The rate of sorption R can be obtained from first derivative of the Peleg equation

$$R = \frac{dM}{dt} = \frac{k_1}{(k_1 + k_2 t)^2}$$

The Peleg rate constant  $k_1$  relates to the initial sorption rate  $R_0$ , i.e. R at  $t = t_0$ 

$$R_0 = \frac{dM}{dt} \Big| \ell_0 = \frac{1}{k_1}$$

$$M_{Pe} = \, M \, | \, t_{\infty} = M_e = M_0 + \frac{1}{k_2}$$

Where  $M_e$  = equilibrium moisture content (% d.b.).

Thus, the Peleg model has the advantage of estimating the equilibrium moisture content and initial sorption rate. It can predict the model based on initial moisture content rather than Page model based on both initial moisture content and equilibrium moisture content.

Using SAS procedure NLIN the various model constants of these models can be evaluated with the observed characteristics (M, t) as inputs. The performance of the model can be compared with the original characteristics using performance parameters such as coefficient of determination  $(R^2)$  as well as mean square deviation (MSD).

# Temperature Dependence of Model Parameters – Arrhenius Model

$$P = Aexp \begin{bmatrix} E \\ RT \end{bmatrix}$$

Where P = parameter modeled and takes the parameters  $k_e$ , k, n,  $1/k_1$ ,  $1/k_2$  with consistent units, A = frequency factor of the initial hydration rate with consistent unites of selected parameter P, E = activation energy (kJ mol<sup>-1</sup>) of model considered, R = universal gas constant (8.314 × 10-3 kJ mol<sup>-1</sup> K<sup>-1</sup>), and T = absolute temperature (K).

Again using SAS procedure NLIN the Arrhenius model constants (E and A) can be evaluated with the other hydration model constants as inputs. Using this model the temperature dependence of the hydration model constants was evaluated. The hydration model constants at any temperature in the studied range can estimated from the Arrhenius model results. Thus the combination of hydration kinetics and Arrhenius models it is possible to predict the moisture condition of biomass at any time and temperature in the range of variables studied.

# References

- 1. Peleg, M. 1988. An empirical model for description of moisture sorption curves. Journal of Food Science 53(4): 1216-1219.
- 2. Igathinathane, C., Pordesimo, L. O., Womac, A. R., and Sokhansanj, S. 2009. Hygroscopic Moisture Sorption Kinetics Modeling of Corn Stover and Its Fractions. Applied Engineering in Agriculture, 25(1): 65-73.

# Appendix – IV: Installation issues and solution

#### A. Environmental control chamber installation

The environmental control chamber was received and placed at BTL (Fig. A4-1). Meanwhile, we ordered the shelves from the suppliers, which were not the part of the basic unit. Also we wanted to make the utilities from the lab side to be in compliance with the requirements of the equipment.



Fig. A4-1. Environmental control chamber as received and ready to be installed

The equipment (ESPEC - Environmental Control Chamber: BTX-475) needs a special voltage of 208/230 V, 60 Hz, and 12A (NEMA 60-20 plug), which is not common in laboratory setting. A local electrical specialist was brought and this electrical supply arrangement was made. The equipment also needs filtered water to be supplied at a pressure of 30 PSI. While arranging these specialized utilities, a request for training and installation was sent to the supplier in July 2011. Suppliers scheduled the October 3-4, 2011 for installation and demonstration. This long time gap

was because of the time needed to obtain the optional items and necessary utilities. Before the technician arrived we gave the water connection and prepped the chamber. A special bladder air pressure device was attached to the equipment to supply the water (Fig. A4-2). Later this arrangement was dismantled and lab-wide unit providing RO water was installed at the end of November, 2011; and the equipment was now connected directly to the water supply tap.



Fig. A4-2. Bladder air pressure arrangement for pressurized water supply to equipment

The technician found that the chamber was flooded with water (Fig. A4-3 - top) and suspected that it would have affected the electronics (Fig. A4-3 – bottom-left) and recommended a fan should be run continuously for 3-4 days and we have followed that suggestion (Fig. A4-3 – bottom-right).

The technician indicated that the flooding was due to not removing a packing in the water input line that was in place when equipment was shipped. The technician left without installing the equipment as he suspected the electronics would have flooded and installation would have caused serious damage to the equipment if proceeded. When we contacted the supplier for second installation attempt they said the fault is ours and will cost another fresh installation charge. Even though we requested for some consideration, they said that it is not possible without additional charges as the technician is too busy and transportation cost the most part.

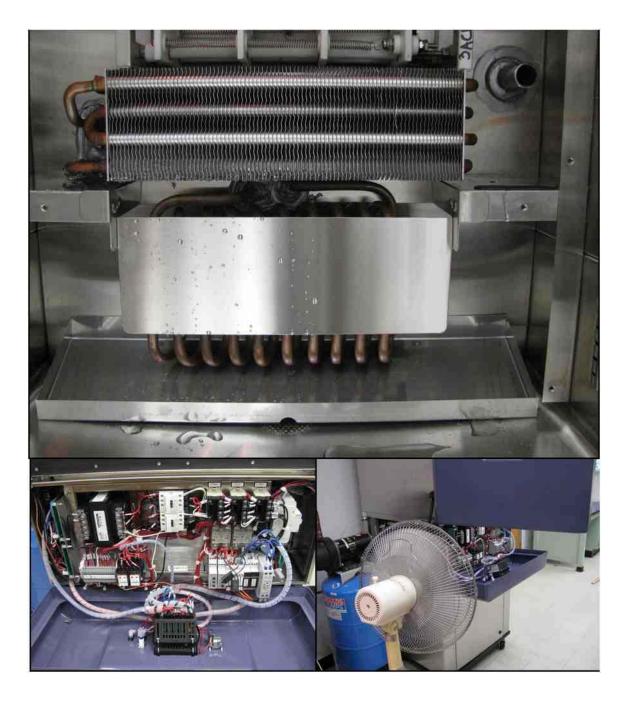


Fig. A4-3. Water flooding issue inside equipment (top), electronics (bottom-left), and moisture removal by pedestal fan (bottom-right)

Also we were told if the technician is around ND for other assignments, we will be contacted and offered a reduction in installation charges. As this was uncertain, we proceeded with the installation on our own and successfully completed it using the online technical support. In fact, the flooding did not affect the equipment the electronic circuits. We used the equipment to study the hydration characteristics of biomass samples successfully as presented in results.

# B. Universal testing machine installation

The UTM is a heavy and high capacity machine (Fig. A4-4). The equipment was received and kept in the ND state shop, and need to be moved to the BTL located at the main administrative building of NGPRL, USDA-ARS, Mandan, ND.



Fig. A4-4. UTM received and kept in NDSU State shop

This equipment weigh about 2500 lbs, and is beyond the capacity of the technical crew of the NGPRL. So we went for professional movers competitive quotes. Two of the companies quoted more than \$3500, and fortunately another company called "Action Movers" quoted was \$727 and we have chosen them and ordered for the move. The quote would have cost us extra if they brought their forklift. However, with NGPRL forklift and the crew helping we saved some expense. A series of activities of transporting with proper packing and moving into BTL are

depicted in figure A4-5. The professional movers with a crew of six members did a good job and the safely moved the equipment to the BTL. The move took about 2.5 hours and was performed on September 6, 2011.



Fig. A4-5. Sequence of moving the UTM from the State shop to BTL by professional movers

When the equipment was powered on after giving the necessary connections it worked once and stopped unfortunately. This was reported to the supplier, who is expected to perform the installation and training, requested us to check a few things before he comes for the official installation and training. So we started following their instructions and did some checking. It was suspected that some component was defective but we were told before shipping everything was checked OK. The equipment is operated by computer control thus there are two major systems namely (i) computer control and (ii) equipment control. As several components, controls, and

circuit boards were involved and the faulty component was not diagnosed correctly, they asked us to dismantle first computer control chords (PCB) and then equipment control components and chords. They checked the components and chords we sent and shipped it back. Some of the components sent/received and tested at supplier and our facilities are shown in figure A4-6.



Fig. A4-6. Various components of the computer and UTM controls shipped and received after testing in a fault finding mission

Thus we sent and received the following components during this fault finding mission (i) Computer chords sent and tested OK, (ii) UTM frame control chords sent and tested OK, (iii) UTM frame control box received and tested, (iv) Power chords sent and tested OK, (v) Hand held motor was received to check, (vi) Servo drive unit with amplifier was received and worked well (Fig. 16), and finally (vii) shipping back all the borrowed components.

In these sequences of shipping and testing, it was found that most of the components were working alright. This means that the faulty component was not to be detected until some trials and errors were made. It is unfortunate that the fault-finding mission took this long as only a single component was defective (Fig. A4-7). Successful replacement of this component was performed on March 5, 2012 and the equipment is working well now. Supplier paid all the shipping charges. Since we have performed most of the work in this fault-finding, we negotiated with supplier and we were promised their digi-cal calipers with direct computer interface that allows the operator to push a button on the caliper and have dimension transmitted automatically into the test program (or) extend the their warranty to the equipment for one additional year. After assessment more negotiation or a decision will be made.

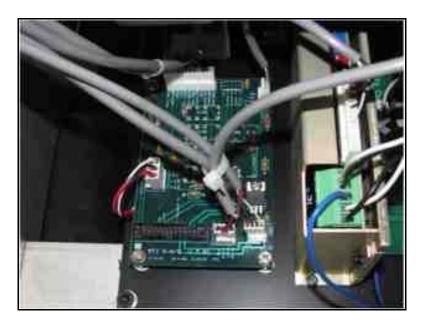


Fig. A4-7. Servo drive unit with amplifier (gold enclosure – right to PCB) was the faulty component that was detected finally and replaced

For actual testing of the samples special attachments (e.g. shearing, cutting, etc.) need to be designed and fabricated. It is necessary to design our own attachment and fabricate them, as these attachments are specialized pieces designed to suit our needs and are usually not available for direct purchase or supplied with the basic UTM (Fig. A4-4). Therefore for the mechanical properties determination of selected biomass we designed a double shear device (Fig. 7) and modified Warner Bratzler device (Fig. 10).

With the designed drawings, we approached local mechanical fabricators of Mandan and Bismarck, ND. We could get only two firms that can able to make these items and their quotes were very expensive (> \$3000). We then explored the possibility with NDSU, and technicians from the Agricultural Service Center, NDSU, Fargo informed us to fabricate these attachments for about \$500 or less (final amount will be known after fabrication). So we made the work order and the fabrication of these attachments were in the final stages.

Experiments will start after receiving the fabricated UTM attachments. An ASABE Annual Meeting, at Dallas, Texas in August 2012 was submitted on the mechanical strength characteristics of selected biomass that will be using the data generated from this equipment and specialized attachment. More information and results will presented and published in regular outlets in the future.

Installation of the other equipment went on smoothly. Now all of the equipment are successfully installed and tested. Results were obtained using the equipment were presented and more results will be generated in the future.

# Appendix – V: Samples Used in the Study - Switchgrass Under Different Water Stress Treatments and Other Biomass Species

# A. Specification of switchgrass samples used in the study

2006-6-3-1 CONTROL	2007-6-3-1 CONTROL	2008-6-3-1 CONTROL
2006-12-3-1 CONTROL	2007-12-3-1 CONTROL	2008-12-3-1 CONTROL
2006-3-2-1 JUL-AUG DRY	2007-3-2-1 JUL-AUG DRY	2008-3-2-1 JUL-AUG DRY
2006-13-3-1 JUL-AUG DRY	2007-13-3-1 JUL-AUG DRY	2008-13-3-1 JUL-AUG DRY
2006-8-3-1 MAY-JUN DRY	2007-8-3-1 MAY-JUN DRY	2008-8-3-1 MAY-JUN DRY
2006-16-4-1 MAY-JUN DRY	2007-16-4-1 MAY-JUN DRY	2008-16-4-1 MAY-JUN DRY

Note: The ID (e.g. 2006-6-3-1) refers to 2006 – year, 6 – plot number, 3 – treatment number, 1 – bag number

CONTROL This treatment with long-term (1913 – 2004) precipitation.

MAY-JUN DRY

This treatment is water stress during May and June.

JUL-AUG DR

This treatment is water stress during July and August.

The above treatments were initiated in 2006. In 2007, all watering treatments were back at the long-term average (Control). The control was also used in 2008. Samples are collected from the NGPRL, USDA-ARS, Mandan, ND.

### Other biomass feedstocks used are:

Bromegrass, big bluestem, corn stover, intermediate wheat grass, pea stems, and wheat straw. Note that some of the properties of these crops were already presented.



Fig. A5-1. Picture showing ground switchgrass biomass on aluminum dishes being held in desiccator for cooling after the oven drying procedure to obtain the bone dry mass in moisture content determination. The moisture content of the samples used in the study varied from 4.2% to 7.4% wet basis.

# Appendix – VI: Calorific Values of Switchgrass Under Different Water Stress Treatments and Other Biomass Feedstocks

Table A2. Calorific value and residue content of selected biomass showing replications

Biomass	Sample mass (mg)	CV (J/g)	Residue (mg)
Switchgrass			
2006-3-2-1 JUL-AUG DRY	503.3	17107	12.8
	519.1	16892	11.4
	508.6	17098	11.4
2006-6-3-1 CONTROL	506.7	17173	10.0
	504.1	17195	13.4
	503.7	17277	13.8
2006-8-3-1 MAY-JUN DRY	503.6	17154	10.5
	504.6	17056	9.9
	503.0	16975	10.9
2006-12-3-1 CONTROL	505.9	16993	15.1
	510.0	17089	12.7
	508.7	17045	15.6
2006-13-3-1 JUL-AUG DRY	500.7	17377	9.0
	500.1	17329	12.4
	508.5	17449	10.0
2006-16-4-1 MAY-JUN DRY	502.8	16980	10.0
	500.6	17406	8.8
	506.9	17271	9.2
2007-3-2-1 JUL-AUG DRY	507.6	16806	11.9
	510.6	16928	12.0
	501.3	17170	13.6
2007-6-3-1 CONTROL	506.1	17221	14.4
	508.5	16892	11.8
	509.0	17117	13.1
2007-8-3-1 MAY-JUN DRY	504.2	17151	12.5
	501.5	17195	10.5
	505.1	16795	12.0
2007-12-3-1 CONTROL	508.5	16958	14.1
	504.1	16906	12.0
	509.4	16809	11.0
2007-13-3-1 JUL-AUG DRY	506.9	16835	15.1
	503.7	17421	9.6
	508.8	17205	11.0
2007-16-4-1 MAY-JUN DRY	509.1	16960	11.3
	503.3	17007	11.1
	503.1	17003	9.6

2008-3-2-1 JUL-AUG DRY	504.0	17059	11.4
	506.9	17085	11.4
	508.3	17080	11.3
2008-6-3-1 CONTROL	508.4	16694	14.5
	502.8	16907	13.7
	501.8	16917	12.4
2008-8-3-1 MAY-JUN DRY	508.7	17190	10.2
	500.9	17124	9.7
	505.9	17139	11.4
2008-12-3-1 CONTROL	509.1	17202	11.3
	508.2	16942	10.2
	504.7	16818	17.7
2008-13-3-1 JUL-AUG DRY	507.0	17113	9.7
	503.8	16911	12.1
	508.5	17266	11.3
2008-16-4-1 MAY-JUN DRY	500.7	17077	9.7
	502.4	17153	14.7
	509.4	17095	12.7
Pea stalks	501.6	15204	8.0
	510.3	15312	5.8
	519.1	15423	5.1
Intermediate wheat grass	506.5	17178	9.2
	503.4	17163	7.4
	514.0	17290	8.7

Note: Refer to Appendix V for explanation of the switchgrass biomass labels

Table A3. Consolidated calorific value and residue content of selected biomass

Biomass	CV (J/g)	STD (J/g)	Residue (mg)	STD (mg)
Switchgrass				_
2006-6-3-1 CONTROL	17215	55	12.4	2.1
2006-12-3-1 CONTROL	17042	48	14.5	1.6
2006-3-2-1 JUL-AUG DRY	17032	122	11.9	8.0
2006-13-3-1 JUL-AUG DRY	17385	60	10.5	1.7
2006-8-3-1 MAY-JUN DRY	17062	90	10.4	0.5
2006-16-4-1 MAY-JUN DRY	17219	218	15.5	10.6
2007-6-3-1 CONTROL	17077	168	13.1	1.3
2007-12-3-1 CONTROL	16891	76	12.4	1.6
2007-3-2-1 JUL-AUG DRY	16968	185	12.5	1.0
2007-13-3-1 JUL-AUG DRY	17154	296	11.9	2.9
2007-8-3-1 MAY-JUN DRY	17047	219	11.7	1.0
2007-16-4-1 MAY-JUN DRY	16990	26	10.7	0.9
2008-6-3-1 CONTROL	16839	126	13.5	1.1
2008-12-3-1 CONTROL	16987	196	13.1	4.1
2008-3-2-1 JUL-AUG DRY	17075	14	11.4	0.1
2008-13-3-1 JUL-AUG DRY	17097	178	11.0	1.2
2008-8-3-1 MAY-JUN DRY	17151	35	10.4	0.9
2008-16-4-1 MAY-JUN DRY	17108	40	12.4	2.5
Pea stalks	15313	110	6.3	1.5
Intermediate wheat grass	17210	69	8.4	0.9

Note: STD – standard deviation

**Discussion:** The CVs of pea stalks were much higher than that of switchgrass and intermediate wheat grass. With switchgrass, the irrigated and water stressed crops in 2006 and 2007 did not have difference in CVs; however, in 2008 the water stress switchgrass had higher CVs. Residues of switchgrass (2.4%) were greater than pea stalks and intermediate wheat grass (1.4%), which indicates higher concentration of ash or silica in switchgrass. It is interesting to note that the energy values of switchgrass were not affected by water stress conditions during the crop growth.

# Appendix – VII: Thermal Analysis of Switchgrass Under Different Irrigation Treatments and Other Biomass Feedstocks

# A. Pyrolytic combustion in inert environment (nitrogen as purge gas)

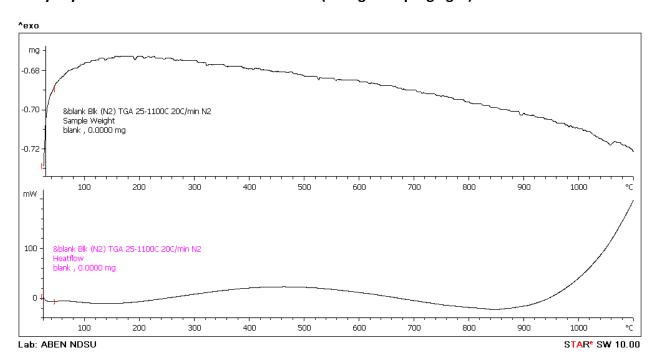


Fig. A7-1. TGA and DSC blank curves with nitrogen as purge gas



Fig. A7-2. Picture showing alumina sample holder and pyrolyzed ground switchgrass when nitrogen was used as purge gas. Note that in inert environment such as nitrogen only charring occurs and carbonaceous material remains; however, with air (presence of oxygen) only white ash remains

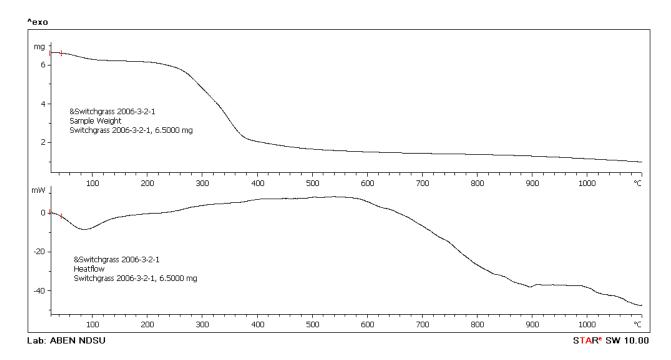


Fig. A7-3. TGA and DSC curves of switchgrass (ID: 2006-3-2-1 Jul-Aug Dry)

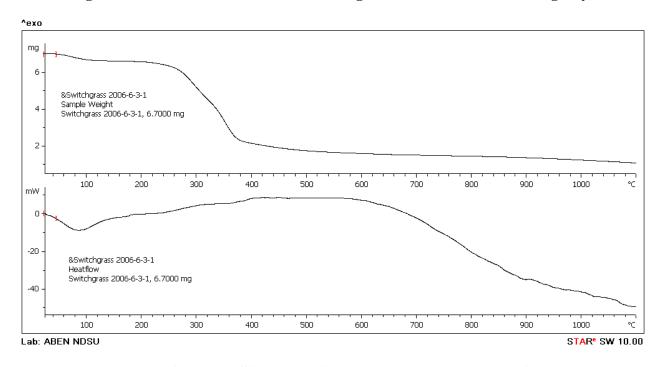


Fig. A7-4. TGA and DSC curves of switchgrass (ID: 2006-6-3-1 Control)

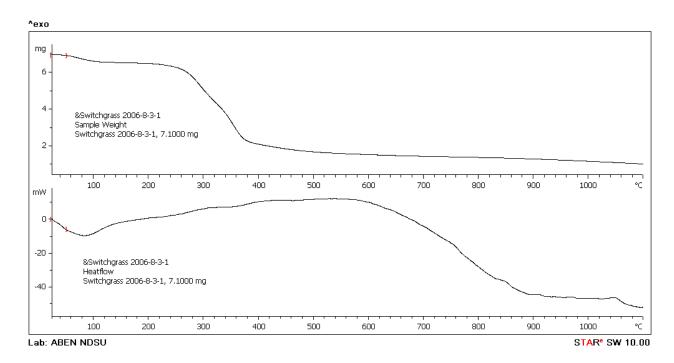


Fig. A7-5. TGA and DSC curves of switchgrass (ID: 2006-8-3-1 May-Jun Dry)

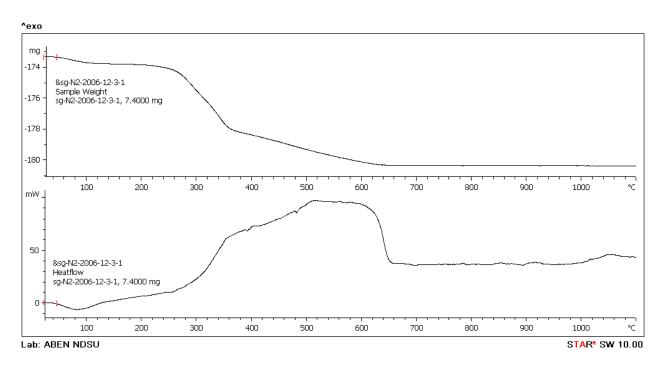


Fig. A7-6. TGA and DSC curves of switchgrass (ID: 2006-12-3-1 Control)

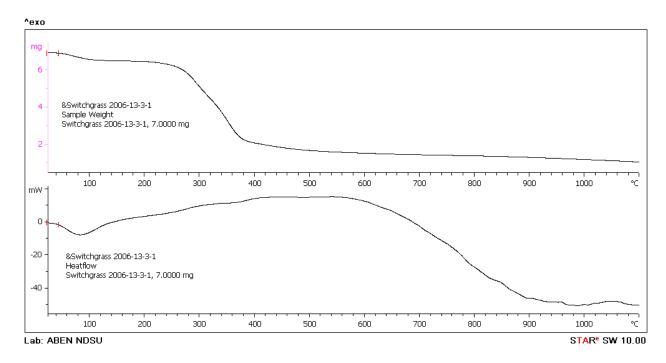


Fig. A7-7. TGA and DSC curves of switchgrass (ID: 2006-13-3-1 Jul-Aug Dry)

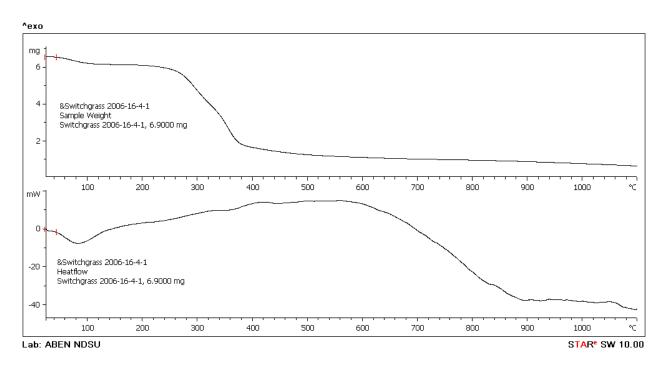


Fig. A7-8. TGA and DSC curves of switchgrass (ID: 2006-16-4-1 May-Jun Dry)

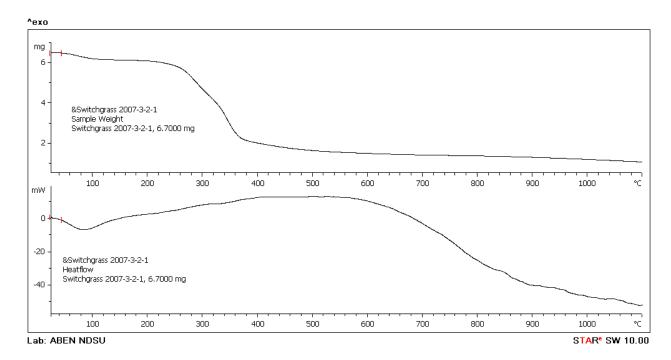


Fig. A7-9. TGA and DSC curves of switchgrass (ID: 2007-3-2-1 Jul-Aug Dry)

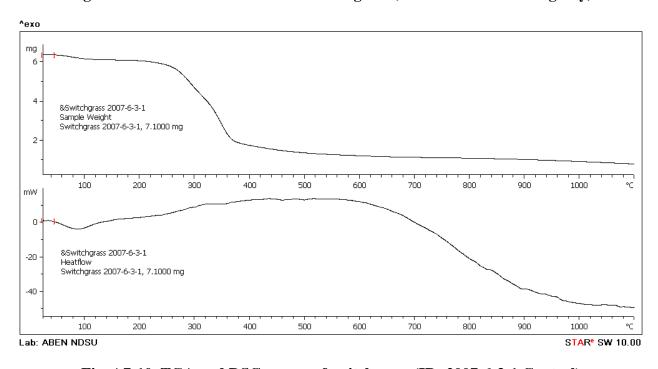


Fig. A7-10. TGA and DSC curves of switchgrass (ID: 2007-6-3-1 Control)

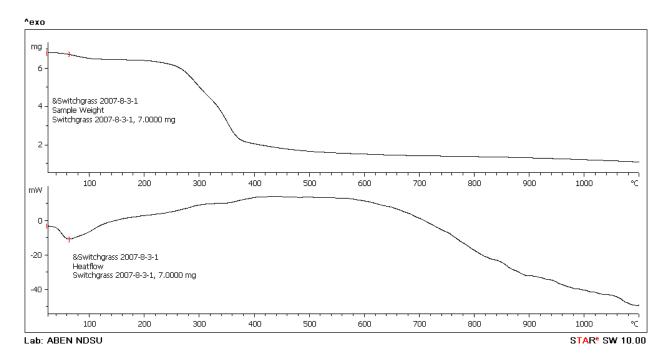


Fig. A7-11. TGA and DSC curves of switchgrass (ID: 2007-8-3-1 May-Jun Dry)

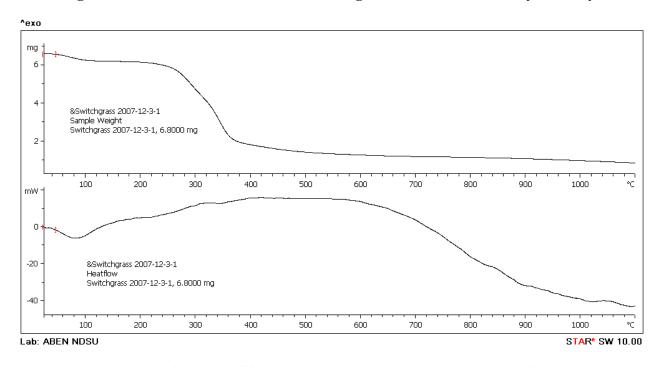


Fig. A7-12. TGA and DSC curves of switchgrass (ID: 2007-12-3-1 Control)

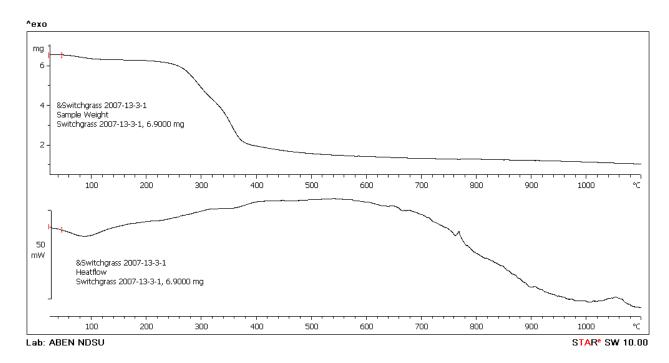


Fig. A7-13. TGA and DSC curves of switchgrass (ID: 2007-13-3-1 Jul-Aug Dry)

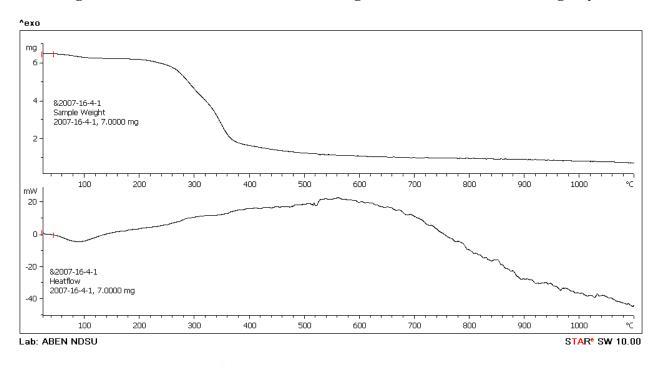


Fig. A7-14. TGA and DSC curves of switchgrass (ID: 2007-16-4-1 May-Jun Dry)

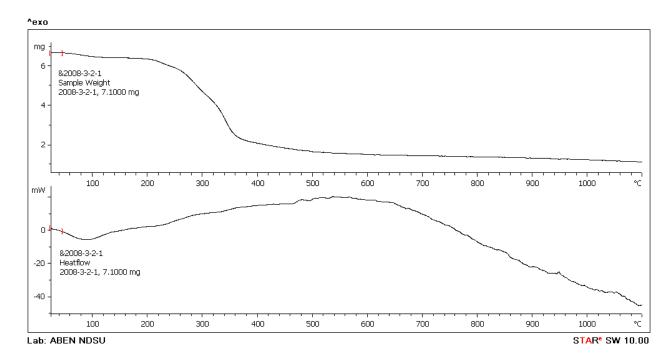


Fig. A7-15. TGA and DSC curves of switchgrass (ID: 2008-3-2-1 Jul-Aug Dry)

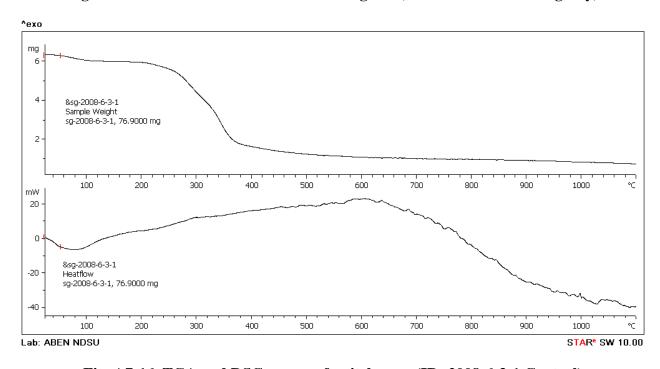


Fig. A7-16. TGA and DSC curves of switchgrass (ID: 2008-6-3-1 Control)

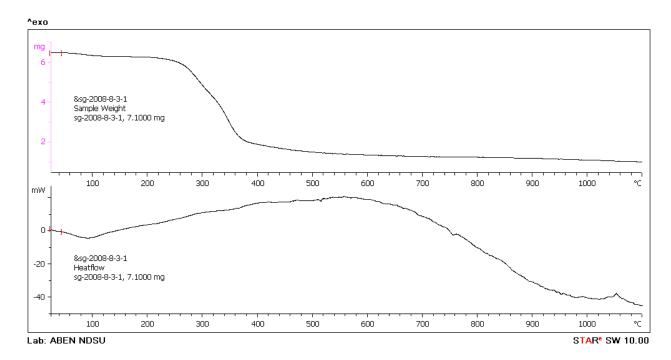


Fig. A7-17. TGA and DSC curves of switchgrass (ID: 2008-8-3-1 May-Jun Dry)

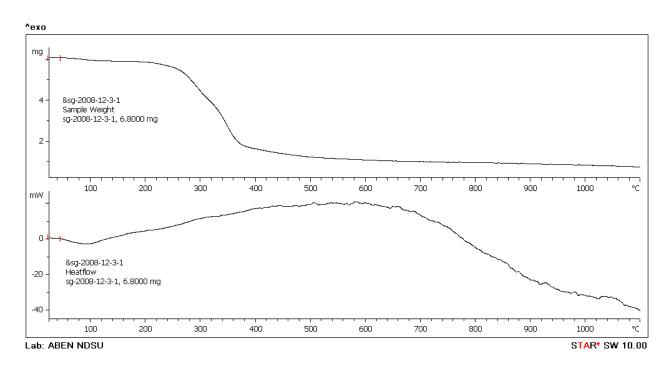


Fig. A7-18. TGA and DSC curves of switchgrass (ID: 2008-12-3-1 Control)

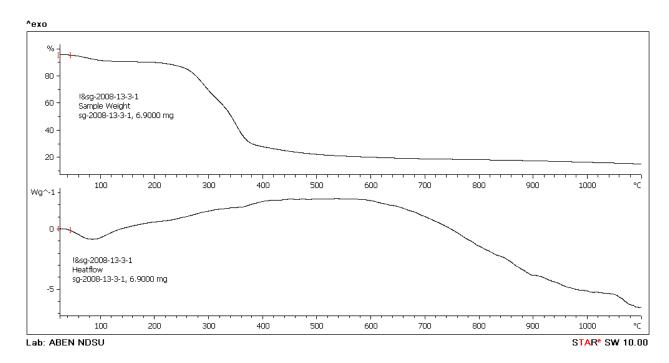


Fig. A7-19. TGA and DSC curves of switchgrass (ID: 2008-13-3-1 Jul-Aug Dry)

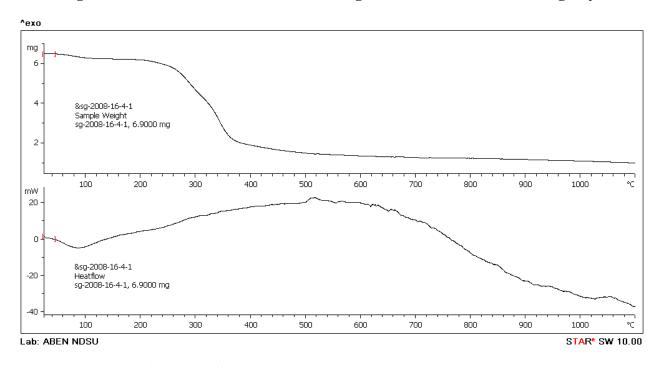


Fig. A7-20. TGA and DSC curves of switchgrass (ID: 2008-16-4-1 May-June Dry)

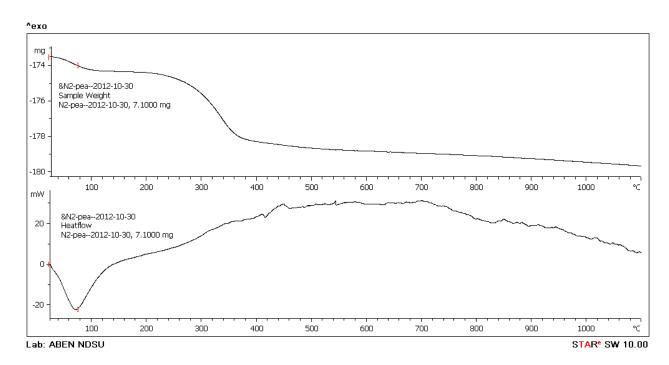


Fig. A7-21. TGA and DSC curves of pea stalks

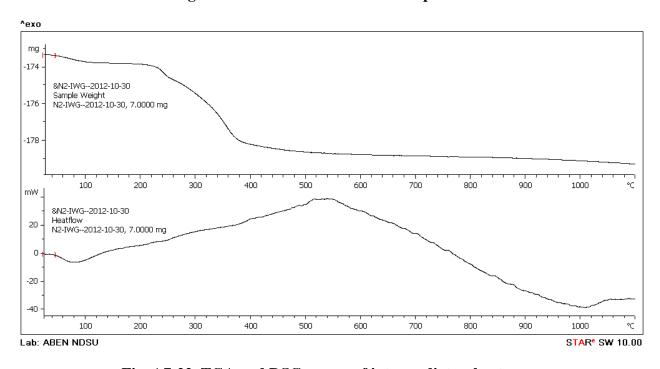


Fig. A7-22. TGA and DSC curves of intermediate wheat grass

**Discussion:** With nitrogen as purge gas the biomass goes through pyrolytic combustion, which produces pyrolysis gas that was vented out and a solid residue as combustion product. The combustion product is biochar, which is black and consisted mostly of carbon (Fig. A7.2). TGA curves of the selected biomass showed a sudden mass loss in the range of 200 to 400°C

representing major mass loss due to volatiles loss and charring. After the 400°C the mass reached a minimum level and gradually reduced or remained constant in the rest of 1100°C. DSC characteristics showed mostly endothermic reaction (-ve mW values) as the combustion happened in inert environment. Flattened peak representing the exothermic reaction (+ve mW) were found in a wide temperature range of 200 to 800°C. This means gradual release of heat under the inert environment with nitrogen gas. TGA and DSC curves with blank subtraction of all biomass studied (switchgrass, pea stalks, and intermediate wheat grass) were similar but show their characteristic thermal signature.

#### B. Complete combustion in presence of oxygen (air as reaction gas)

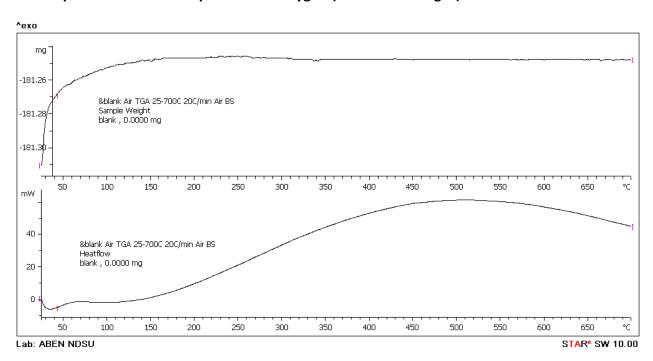


Fig. A7-23. TGA and DSC blank curves with air as reaction gas

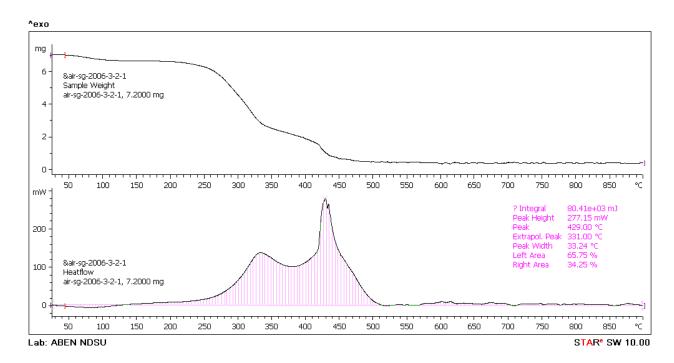


Fig. A7-24. TGA and DSC curves of switchgrass (ID: 2006-3-2-1 Jul-Aug Dry)

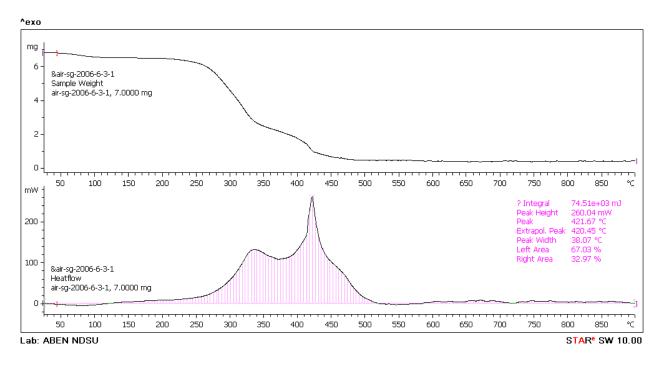


Fig. A7-25. TGA and DSC curves of switchgrass (ID: 2006-6-3-1 Control)

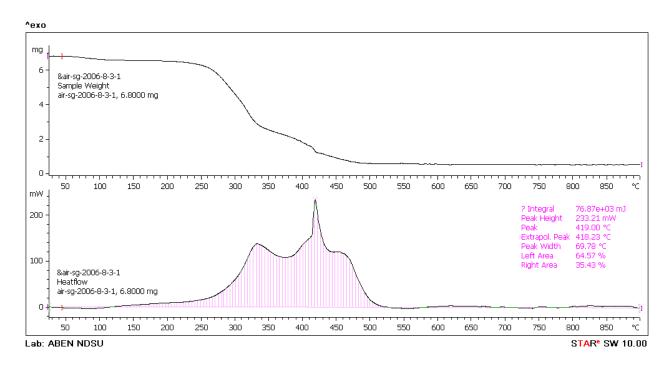


Fig. A7-26. TGA and DSC curves of switchgrass (ID: 2006-8-3-1 May-Jun Dry)

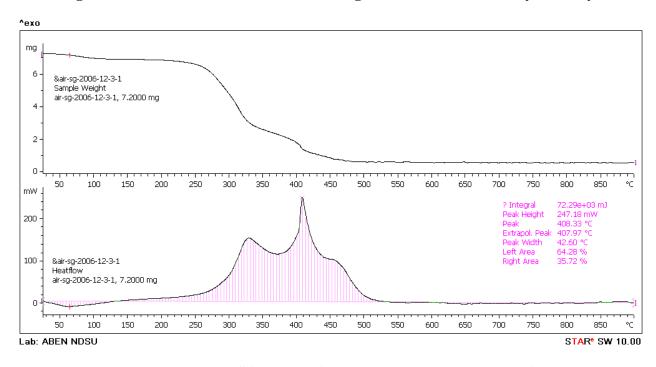


Fig. A7-27. TGA and DSC curves of switchgrass (ID: 2006-12-3-1 Control)

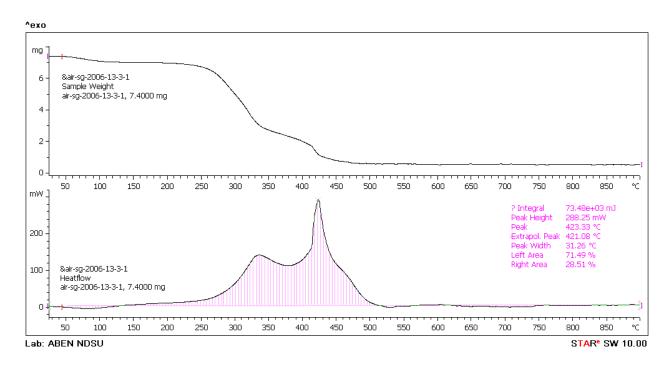


Fig. A7-28. TGA and DSC curves of switchgrass (ID: 2006-13-3-1 Jul-Aug Dry)

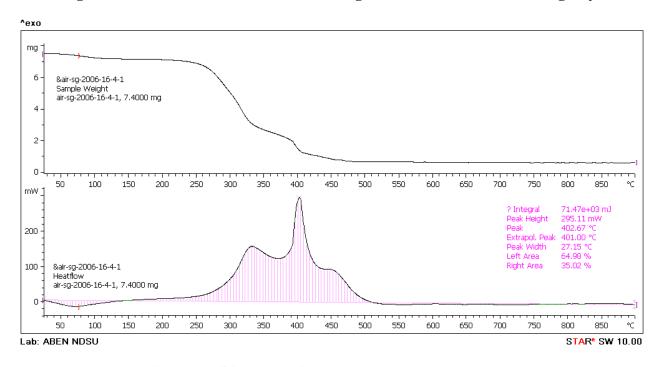


Fig. A7-29. TGA and DSC curves of switchgrass (ID: 2006-16-4-1 May-Jun Dry)

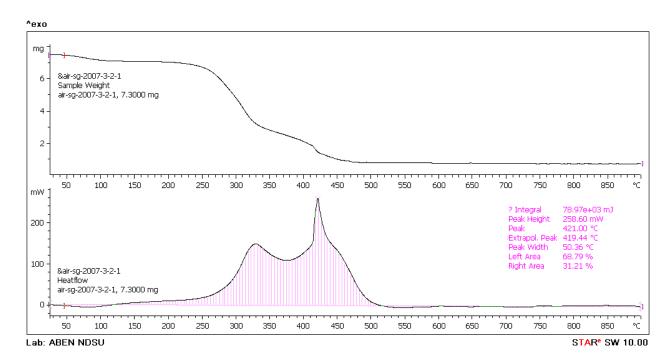


Fig. A7-30. TGA and DSC curves of switchgrass (ID: 2007-3-2-1 Jul-Aug Dry)

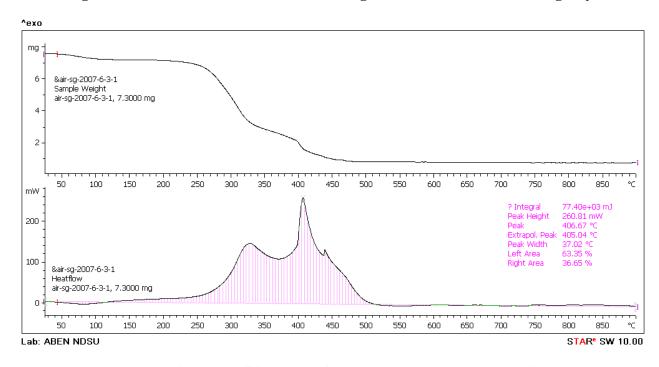


Fig. A7-31. TGA and DSC curves of switchgrass (ID: 2007-6-3-1 Control)

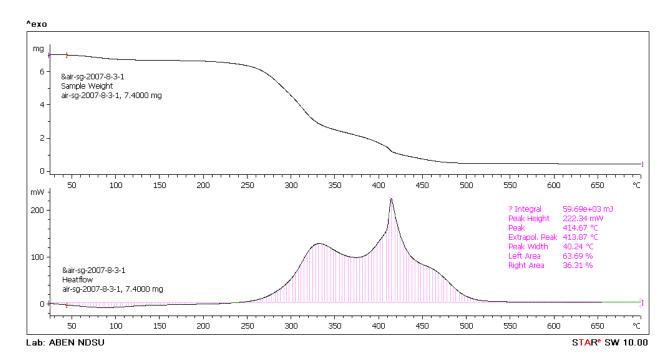


Fig. A7-32. TGA and DSC curves of switchgrass (ID: 2007-8-3-1 May-Jun Dry)

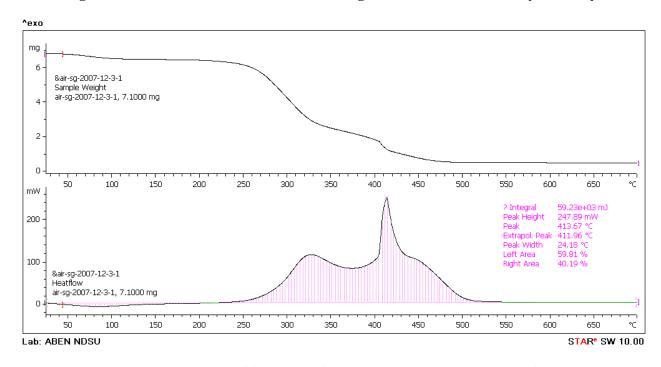


Fig. A7-33. TGA and DSC curves of switchgrass (ID: 2007-12-3-1 Control)

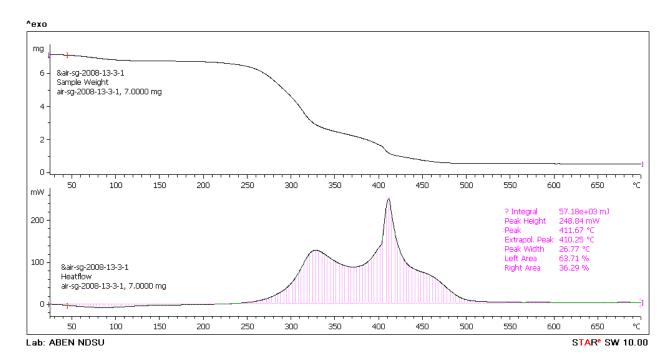


Fig. A7-34. TGA and DSC curves of switchgrass (ID: 2007-13-3-1 Jul-Aug Dry)

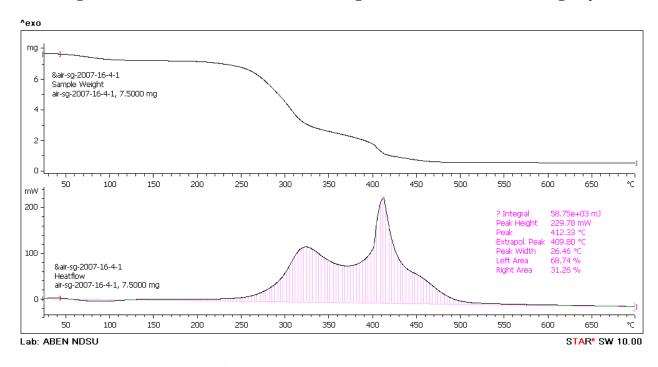


Fig. A7-35. TGA and DSC curves of switchgrass (ID: 2007-16-4-1 May-Jun Dry)

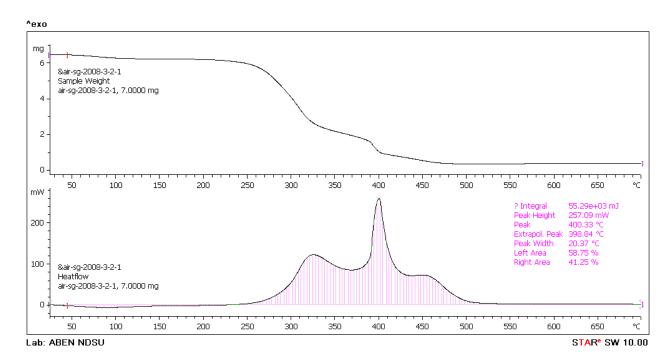


Fig. A7-36. TGA and DSC curves of switchgrass (ID: 2008-3-2-1 Jul-Aug Dry)

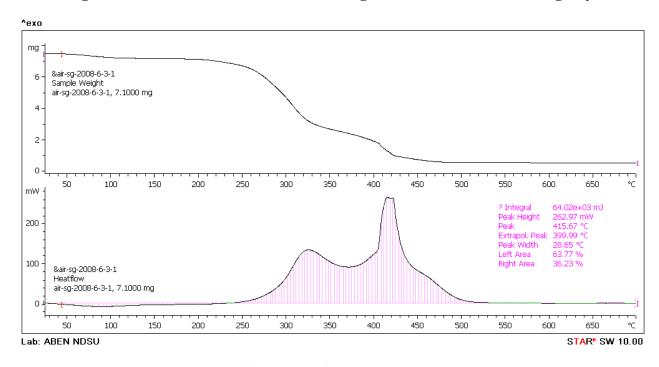


Fig. A7-37. TGA and DSC curves of switchgrass (ID: 2008-6-3-1 Control)

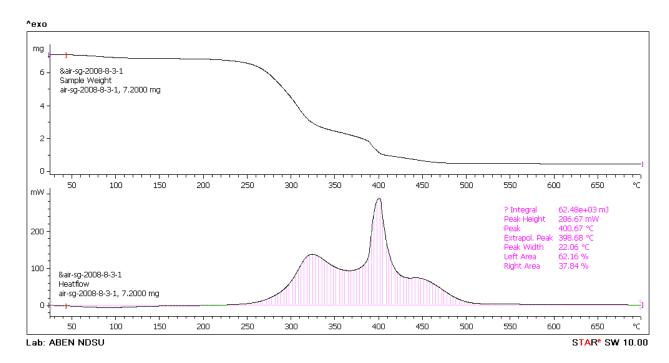


Fig. A7-38. TGA and DSC curves of switchgrass (ID: 2008-8-3-1 May-Jun Dry)

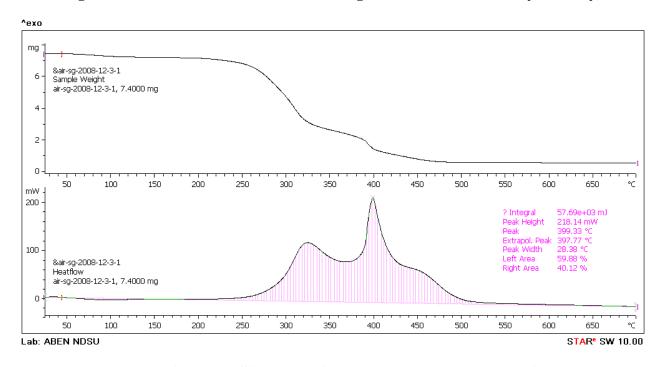


Fig. A7-39. TGA and DSC curves of switchgrass (ID: 2008-12-3-1 Control)

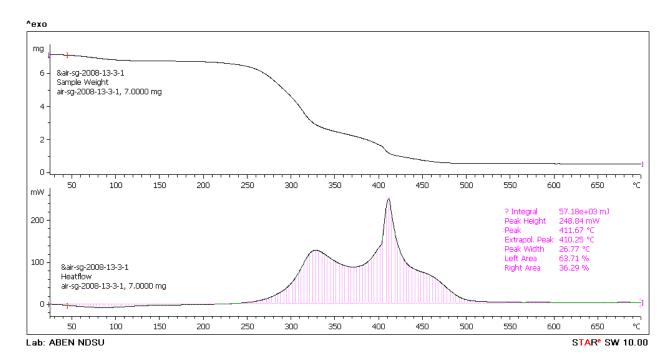


Fig. A7-40. TGA and DSC curves of switchgrass (ID: 2008-13-3-1 Jul-Aug Dry)

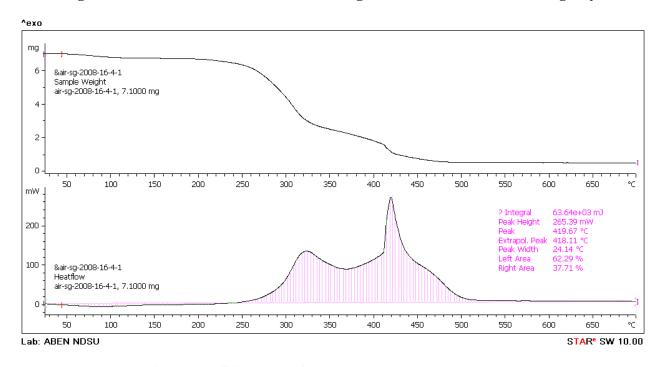


Fig. A7-41. TGA and DSC curves of switchgrass (ID: 2008-16-4-1 May-Jun Dry)

### Other crops

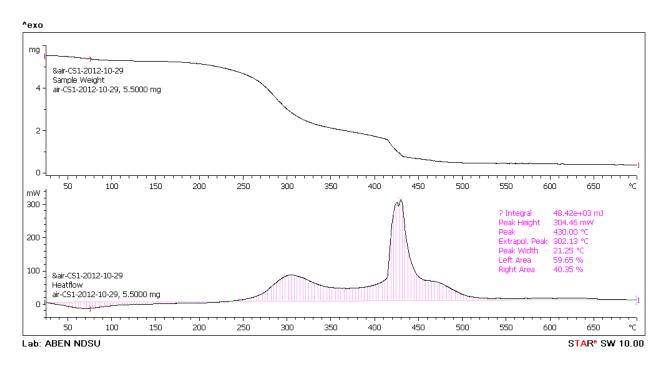


Fig. A7-42. TGA and DSC curves of corn stover

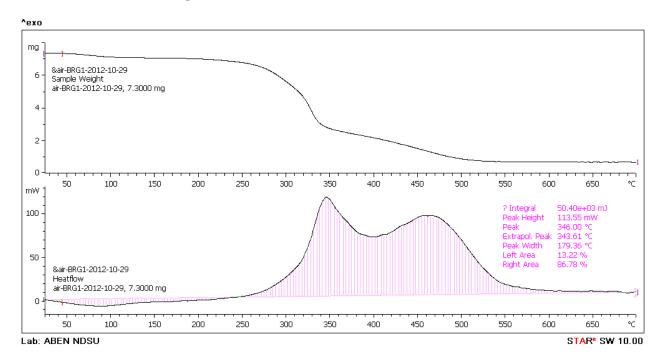


Fig. A7-43. TGA and DSC curves of bromegrass

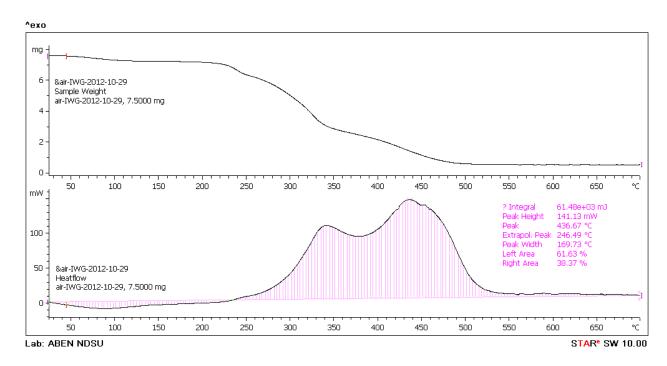


Fig. A7-44. TGA and DSC curves of intermediate wheat grass

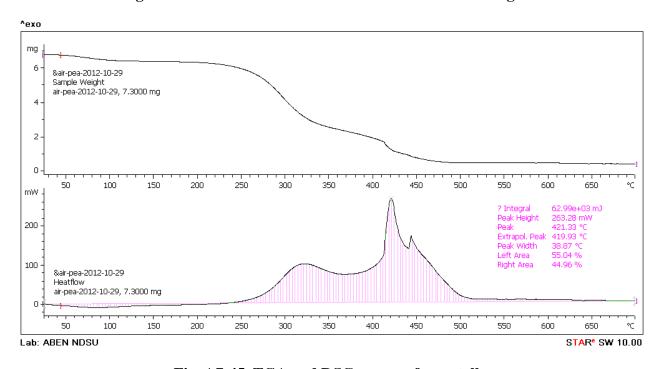


Fig. A7-45. TGA and DSC curves of pea stalks

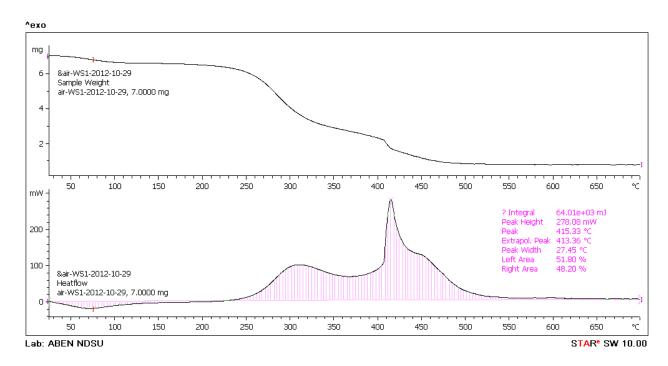


Fig. A7-46. TGA and DSC curves of wheat straw

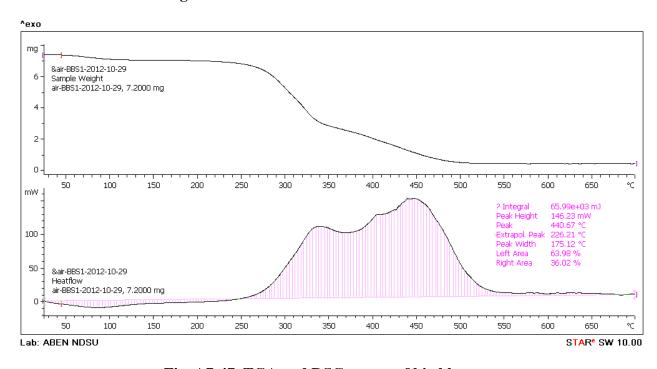


Fig. A7-47. TGA and DSC curves of big bluestem

Table A4. Consolidated calorific value and residue content of selected biomass

Biomass	Integral area - Energy	Peak height	Peak
Civitalemana	(J)	(mW)	(C)
Switchgrass 2006-6-3-1 CONTROL	74.54	260.04	404 7
	74.51	260.04	421.7
2006-12-3-1 CONTROL	72.29	247.18	408.3
2006-3-2-1 JUL-AUG DRY	80.41	277.15	429.0
2006-13-3-1 JUL-AUG DRY	73.48	288.25	423.3
2006-8-3-1 MAY-JUN DRY	76.87	233.21	419.0
2006-16-4-1 MAY-JUN DRY	71.47	295.11	402.7
2007-6-3-1 CONTROL	77.40	260.81	406.7
2007-12-3-1 CONTROL	59.23	247.89	413.7
2007-3-2-1 JUL-AUG DRY	78.97	258.60	421.0
2007-13-3-1 JUL-AUG DRY	57.18	248.84	411.7
2007-8-3-1 MAY-JUN DRY	59.69	222.34	414.7
2007-16-4-1 MAY-JUN DRY	58.75	229.78	412.3
2008-6-3-1 CONTROL	64.02	262.97	415.7
2008-12-3-1 CONTROL	57.69	218.14	399.3
2008-3-2-1 JUL-AUG DRY	55.29	257.09	400.3
2008-13-3-1 JUL-AUG DRY	57.18	248.84	411.7
2008-8-3-1 MAY-JUN DRY	62.48	286.67	400.7
2008-16-4-1 MAY-JUN DRY	63.64	265.39	419.7
Corn stover	48.42	304.46	430.0
Bromegrass	50.40	113.55	346.0
Intermediate wheat grass	61.48	141.13	436.7
Pea stalks	62.99	263.28	421.33
Wheat straw	64.01	278.08	415.3
Big bluestem	65.99	146.23	440.7

**Discussion:** Complete combustion of biomass was achieved in the presence of oxygen that was supplied by air as reaction gas. TGA and DSC curves of biomass feedstocks were similar but have their characteristic thermal signature distinguishing them. TGA curves show a rapid reduction of mass in the approximate temperature range of 250 to 500°C in two stages. DSC curve shows two clear exothermic peaks in this temperature range. Opposed to nitrogen as purge gas, with air the combustion (DSC) is predominantly exothermic (+ve mW).

Analysis of the DSC curve produces the area under the DSC curve (integral), peak height (mW), temperature of the peak, width of peak, and left and right area of peak. These outputs are characteristics of biomass tested and can be used to distinguish or differentiate biomass feedstocks. Although DSC curves of switchgrass were similar, the various derived parameters help to differentiate the biomass feedstocks. It can be observed that the DSC curves of other biomasses were clearly different from the switchgrass. Bromegrass, intermediate wheat grass,

and big bluestem were broader than switchgrass that had a narrow second peak. The initial endothermic region from 25 to 250°C signified the warming or excitation of biomass before releasing the useful energy. However, the endothermic area (-ve mW) was negligible. Table A4 presents integral area of DSC curves and indicates that the other biomass crops (corn stover, bromegrass, intermediate wheat grass, pea stalks, wheat straw, and big bluestem) produce lesser energy than switchgrass. A temperature of about 400°C divides the exothermic peak (left and right portions), and these data can also be applied to compare thermal properties of biomass.

# Appendix – VIII: Mechanical Properties (Cutting and shearing) of Selected Biomass Feedstocks

### A. Cutting characteristics of selected biomass



Fig. A7-46. Figure showing indigenously developed and fabricated (Figs. 9 and 10) modified Warner-Bratzler device being used to measure the cutting strength and energy of biomass stems (e.g. corn stalk). The load cell is at the bottom fixed to the bed of UTM and knife is moved down by the crosshead in compression mode. Load cell is loaded through compressing platens.

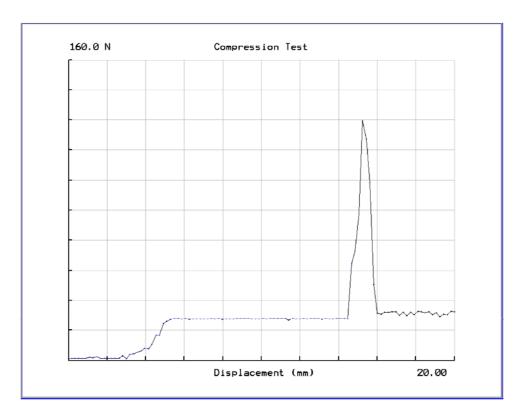


Fig. A7-49. Force-displacement cutting characteristics of big bluestem

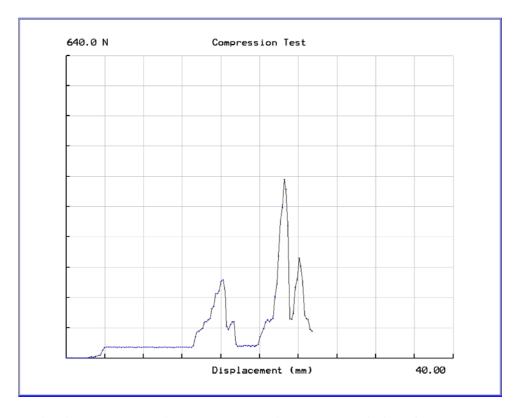


Fig. A7-50. Force-displacement cutting characteristics of pea stalks

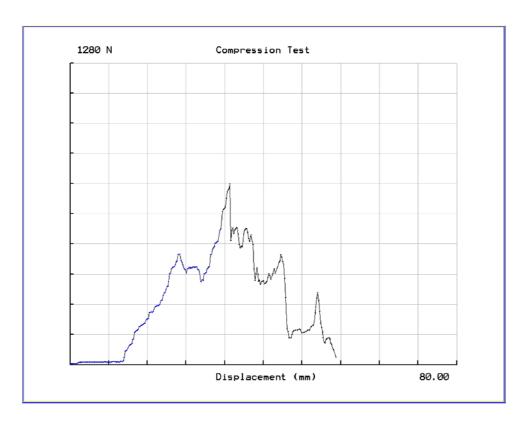


Fig. A7-51. Force-displacement cutting characteristics of fresh corn stover

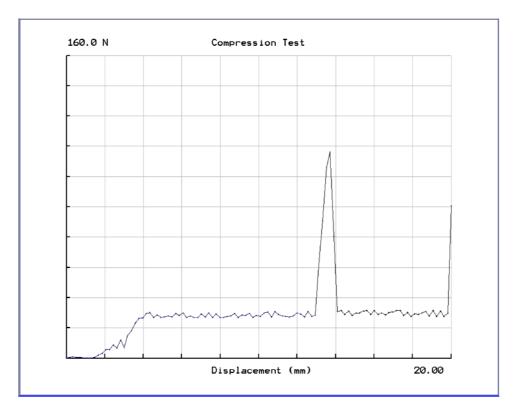


Fig. A7-52. Force-displacement cutting characteristics of intermediate wheat grass

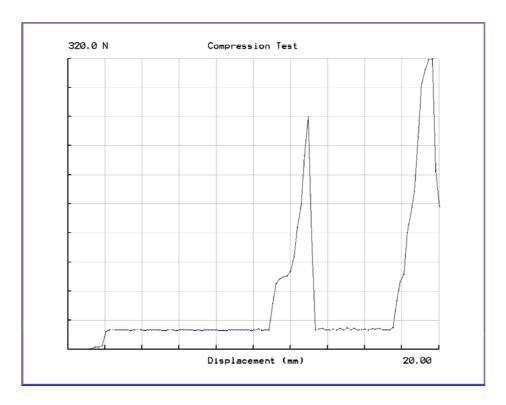


Fig. A7-53. Force-displacement cutting characteristics of switchgrass

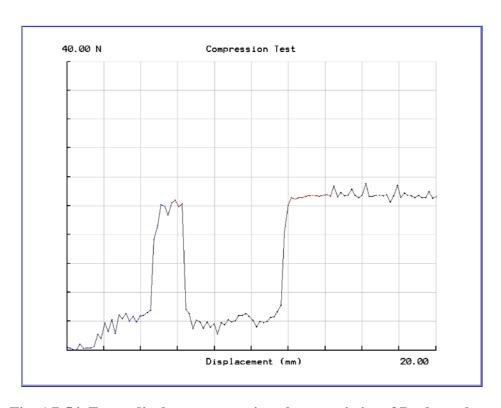


Fig. A7-54. Force-displacement cutting characteristics of Barlow wheat

Table A5. Cutting tensile strengths and specific energy of selected biomass showing replications

Biomass	Cutting strength (MPa)	Cutting specific energy (kN/m)
Big bluestem	8.38	41.90
(7.2% w.b.)	13.72	53.18
	14.35	39.12
Pea stalks	10.55	242.74
(16.0% w.b.)	6.06	220.53
	17.49	316.32
Corn stover fresh	13.01	92.02
(70.3% w.b.)	19.88	87.73
	10.24	98.75
Intermediate wheat grass	23.79	74.91
(4.0% w.b.)	25.92	57.30
	21.52	56.19
Switchgrass	18.72	86.96
(7.5% w.b.)	21.35	39.19
	18.72	59.62
Barlow wheat	5.76	14.77
(7.9% w.b.)	7.89	15.82
	7.24	15.86

Table A6. Consolidated cutting strengths and specific energy of selected biomass

Biomass	Cutting strength (MPa)	STD (MPa)	Cutting specific energy (kN/m)	STD (kN/m)
Pea stalks				
(16.0% w.b.)	11.37	5.76	259.86	50.14
Corn stover fresh				
(70.3% w.b.)	14.38	4.96	92.83	5.56
Intermediate wheat grass				
(4.0% w.b.)	23.74	2.20	62.80	10.50
Switchgrass				
(7.5% w.b.)	19.60	1.52	61.92	23.97
Wheat Barlow				
(7.9% w.b.)	6.96	1.09	15.48	0.62

## **B.** Shearing characteristics of selected biomass

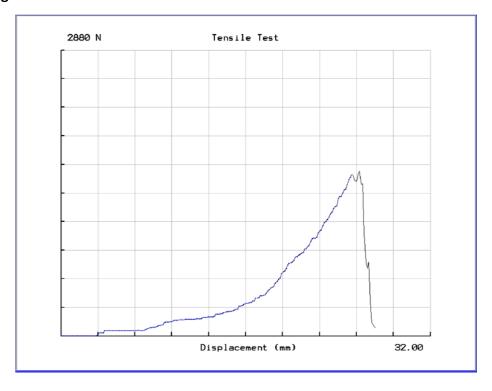


Fig. A7-55. Force-displacement shearing characteristics of fresh corn stover

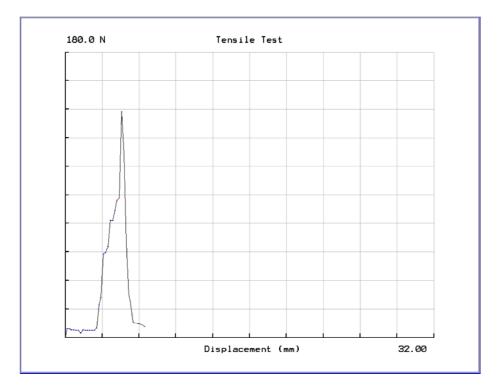


Fig. A7-56. Force-displacement shearing characteristics of intermediate wheat grass

Table A7. Shear strengths and specific energy of selected biomass showing replications

Biomass	Shear strength (MPa)	Shearing specific energy (kN/m)
Corn stover fresh	12.40	62.25
(70.3% w.b.)	12.60	80.84
	13.46	65.87
Intermediate wheat grass	10.87	90.78
(4.0% w.b.)	11.29	169.13
	11.90	118.94

Table A8. Consolidated shear strengths and specific energy of selected biomass

Biomass	Shear strength (MPa)	STD (MPa)	Shearing specific energy (kN/m)	STD (kN/m)
Corn stover fresh				
(70.3% w.b.)	12.82	0.57	69.66	9.85
Intermediate wheat grass				
(4.0% w.b.)	11.35	0.52	126.28	39.69

**Discussion:** Results of mechanical properties through cutting and shearing tests were presented in the form of graphs and tables. Force-displacement curves of cutting (Figs. 49-54) sometimes showed two peaks; the first represented the actual cutting of the stems, and the second represented cut parts or pealed skins getting entangled and rubs between the knife and support block. Cutting stress and energy calculated herein was based only on the first peak while discarding rest of the curves. High moisture in biomass aided in such binding of knife leading to double or more peaks. Dry stalks get cut cleanly leading to single peaks. Bevel given on the knife blade caused the stem to lift from the base and get bound between the knife and base. Force-displacement curves of shearing (Figs. A755-56) were simple and characterized by single peak. Comparing the cutting and shearing modes with common biomass (corn stalks and intermediate wheat grass), failure stress was less in shearing mode than in cutting mode. Shearing and cutting were different mechanisms and resulted in different cut products (double shear device producing three pieces; cutting device producing two pieces).

Biomass testing laboratory being well equipped with equipment capable of determining the biomass feedstocks and their products quality through physical, thermal, mechanical, and storage properties is poised to produce results and serve the local, industrial, and academic needs of ND.