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

Design of a Biomass Scale Cubical Triaxial Tester

Christopher J. Lanning[a], Hojae Yi[b], James H. Dooley[a], Virendra Puri[b], Jordan M. Whitt[a]

[a] Forest Concepts, LLC, 3320 West Valley Hwy N Suite D-110

Auburn, WA 98001

[b] Agricultural and Biological Engineering Department,

The Pennsylvania State University, University Park, PA 16802

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ABSTRACT. *The bioenergy industry is dependent on predictable and reliable feedstock handling equipment. High profile failures in the industry demonstrate the need for better modeling tools for feedstock handling, specifically tools which can model the bulk flow of biomass feedstock materials in handling equipment such as hoppers or screw conveyors. Measurement of bulk material flow characteristics is a critical component to inform modeling tools. Following examples in the soils and pharmaceuticals industries, a Cubical Triaxial Tester (CTT) was selected as a critical component to study relationships between applied stress on a bulk sample and the resulting strain on the bulk material. In cooperation with The Pennsylvania State University, Forest Concepts designed a CTT specifically to measure bulk biomass flowability characteristics. Several mechanical design constraints were identified to ensure proper scaling and repeatability. Major design constraints included sample chamber size versus particle length, volumetric strain sensing accuracy, independent axial pressure control, and sample chamber handling. This report explains how major design constraints were determined.*

Keywords. Biomass, Biomass Flow, Flowability, Integrated Biorefinery, Cubical Triaxial Tester, Constitutive Flow Model

Introduction to the Problem

Consistent handling and conveying of bulk biomass materials are critical in reliable operations of facilities using size-reduced bulk biomass as a raw material. Unfortunately, poor flowability and highly variable physical properties are facts of life with biomass feedstocks. Reliable feedstock handling and conveying are keys to consistent operation of advanced biofuel and bioproducts production facilities.

One of key challenges of scaling-up of biomass processes is feedstock handling. The lack of a systematic tool to characterize and model biomass flow behavior results in excessive downtime due to irregular or problematic flow characteristics of the feed material. In other words, traditional flowability metrics, such as compression ratio, Hausner Index, angle of repose, etc., give qualitative indication of relative flowability among feedstocks but provide little-to-no value for the quantitative engineering design of hoppers and feeders. Therefore, there is a need to develop an engineering tool set that enables an accurate prediction of physical and mechanical conditions of biomass feedstocks and their interaction with feeding systems.

Biomaterials respond to compression and handling differently than metals and minerals. Biomaterials such as biomass feedstocks are considered viscoelastic materials. The viscoelastic nature of biomass helps explain why feedstocks sometimes stick in hoppers after a weekend of non-flow, and augers and feeders have exceptionally high starting torque, even after just a few minutes of stoppage.
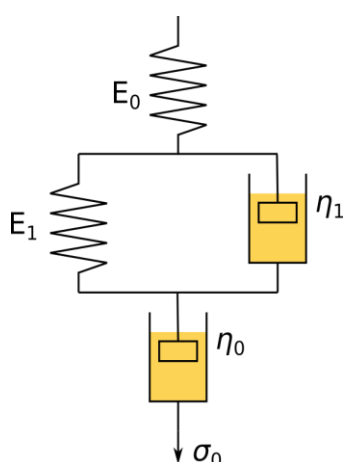
Figure 1. Simplified mechanical analog of biomass particles using springs and dashpots where, E_0 and E_1 are elastic modulus, η_1 and η_0 are damping factors, and σ_0 is applied stress (Mohsenin 1970).

As presented in the diagram (Figure 1), biomass particles (and volumes of particles) respond to loading through elastic, plastic, and viscous deformation. Particles change shape and compress due to loading and then continue to deform and change state over time through stress relaxation and creep. These mechanical properties have been known and studied in other bio-based materials (Mohsenin 1970) but have not been rigorously studied in comminuted biomass feedstocks. Even in traditional biomaterials, such as foodstuffs, the mechanical properties have typically been studied at only one temperature and moisture content of interest to the needs of a user. In the case of the biomass feedstocks industry, biomass materials are subjected to varying temperature and moisture content, as well as forces/pressures.

Friction between particles and/or at the contact points between biomass and materials of construction is also strongly affected by temperature and moisture as well as originating species, unit processes involved, etc. Static and kinetic friction values are highly non-linear as moisture changes. For example, very dry woody material has a coefficient of friction with smooth steel of about 0.4, but as the moisture increases the static friction can increase as high as 0.9 near fiber saturation and then decreases again in the presence of free surface water. In general, kinetic friction is about 20% less than static friction. However, kinetic friction is also a function of sliding speed, especially when surface water is present (Forest Products Laboratory 2010). Temperature effects are much less until either the biomass or the surface temperature approaches the glass transition/melt points of cellulose, hemicellulose, and lignin. At or near those points catastrophe can occur if a small downward temperature change, velocity change, or other factor triggers bio-glue to form between the particles and the materials of construction.

Nuri Mohsenin at The Pennsylvania State University is generally considered the patriarch of the field for study of physical and mechanical properties of biomaterials (Mohsenin 1965). It was clear by 1970 that materials we currently consider to be among the biomass feedstocks had much more troublesome physical and mechanical properties than grains with respect to handling and feeding. Unfortunately, very little research related to physical properties happened until the work of Brian Jenkins at UC Davis specific to biomass materials (Jenkins and Sumner 1986, Jenkins 1989). In his 1989 book chapter (Jenkins 1989), Jenkins admonished biomass engineers to consider the infeed into conversion processes as part of the feedstock system. Jenkins also commented on the lack of organized physical and mechanical properties data for important biomass feedstocks.

A common device used for determining some aspect of bulk biomass mechanical behavior is a tool called a ring shear. Other papers discuss the limitations of ring shears and why a different tool is needed (Schwedes 2003, Ittershagen, Schwedes et al. 2011). Nevertheless, a review of the scaling of ring shears is relevant to determining an appropriate scale for a biomass bulk properties measurement tool since several studies of the applicability of ring shear testers to large particles have been published. The Institution of Chemical Engineers' evaluation of the Jenike ring shear tester recommended that the standard Jenike 95.25 mm ring diameter was useful for particles up to 5 mm size (Institution of Chemical Engineers 1989). Grima

(Grima, Mills et al. 2010) observed that accurate results were increasingly challenging to achieve with particles larger than 4 mm.

A ring shear tester appropriately sized for coal was developed by Scott (Scott and Keys 1992). The device had a 300 mm ring diameter to accommodate 30 mm particles. A large-scale wall friction tester was designed and built in Australia by the University of Wollongong (Grima, Mills et al. 2010) using guidelines from earlier evaluations of the Jenike ring shear tester (Institution of Chemical Engineers 1989). The Grima ring shear tester had a 300 mm diameter and provided for a 50 mm sample depth. It was found to be effective for testing bauxite particles with a 5 mm average particle diameter and 16 mm top size. Thus, Grima concluded that the 300 mm ring shear unit should be useful for particles in the 15-20 mm top-size range. It is useful to note that these programs were interested in industrial bulk materials such as plastic pellets, coal, bauxite, and the like. The term "top size" (also called nominal top size) is defined as the aperture size of the sieve where at least 95% by mass of the material passes (ISO 2016).

From these prior efforts to relate particle size to ring shear device diameter, we can conclude that the device diameter ranged from 10 to 20 times the particle top-size. We applaud the authors of the few reports that discussed how they approached the relationship between test device sample chamber size and particle size for the materials of interest. The literature reinforces our belief that we will need to experimentally derive a particle-size-test-chamber-size relationship appropriate for non-densified bulk biomass materials.

This paper provides an overview of the process and considerations of designing a laboratory toolset for collecting data directly related to quantifying biomass feedstock mechanical properties. The collected data is processed through a series of analysis stages to extract mechanical properties. Ultimately the properties are resolved into a set of continuum scale constitutive models appropriate to model bulk biomass flow reflecting the pressure dependent strength and volume change of bulk solids.

Relevant Standards

ASTM standard E973-82(2013) for measuring bulk density of densified solid biofuels requires a 305 mm cubic test box (ASTM 2013). It has a limitation of applicability to particles of less than 1 cubic inch size to accommodate pellets and cubes.

ASTM has several standard methods for use of triaxial compression testers with cylindrical soil specimens (ASTM 2007, ASTM 2011, ASTM 2011, ASTM 2015). The standards use common language to limit applicability to samples where the "largest particle" is smaller than one-sixth of the specimen diameter. The largest particle is measured post-test by spreading the sample on a table and selecting the largest particle for measurement.

ASTM standard methods for use of ring shear testers also make recommendations of the maximum particle size as a function of the ring diameter. Standard D6128-16 (ASTM 2016) relates to the Jenike shear cell and requires that the shear cell diameter must be at least 20 times the maximum particle size of the bulk solid being tested. Standard D6682-08 (ASTM 2017) applies to the Peschl rotational split-level shear tester. It requires that the shear cell diameter be at least 25 times the average particle diameter for powders. The text further comments that the standard 60 mm shear cell should not be used with particles larger than 2,400 microns (2.4 mm).

The standards clearly were developed in the context of soils and industrial granular materials that are not analogous to bulk biomass materials. While the content of the standards suggests that test cells be six times the top diameter for soils and up to 25 times the mean diameter for powders, it is clear that we will need to experimentally derive such relationships specific to bulk biomass materials.

Materials of interest to flowabilty measurement

The range of materials of interest to this project are bulk woody and herbaceous biomass that are handled within biorefineries and off-site depots that produce reactor-ready feedstocks for just-in-time delivery to biorefineries. We identified a list of bulk materials that we are defining as outside the scope of the project and hence, not required to be accommodated by the biomass flowability measurement tool.

Included in this exclusion are baled agricultural residues and energy crops such as corn stover, miscanthus, and switchgrass. Once the material is debaled and initially processed into a particulate, as opposed to whole-stalk form, the material will be considered inside the scope of the project. Likewise, coarsely ground, unscreened forest residuals, land clearing and storm debris, and the like are excluded due to the high content of very large chunks and pieces up to 500 mm long. After screening to remove overs or milling at a biorefinery or depot, the material will be considered inside the scope of the project.

Most of the other materials in the table below are excluded because they are not currently relevant as feedstocks for cellulosic biorefineries. That said, it will likely be possible to utilize the tool to measure the bulk mechanical properties of many of these materials. They are just not specifically included in the tool design criteria.

Table 1. Materials NOT of intrest to this project

Raw Biomass	**Solid Wastes**	**Industrial Materials**
• Baled ag. residues and crops • Ground unscreened forest residuals • Chipped unscreened whole trees	• Municipal solid waste • C&D[1] debris • Storm debris	• Industrial powders • Soil • Gravel, sand, etc.
Very High Moisture Biomass	**Agricultural Commodities**	**Consumer Products**
• Anaerobic digestate • HTL/HTC[2] aqueous suspension feedstocks • Liquid manure slurries • Algae, pond scum, etc.	• Grain • Forage • Feed • Densified feed and forage (pellets) • Food	• Fertilizers • Pesticides • Absorbents • Animal bedding

As a practical matter, biomass materials that are categorically non-flowable, such as mats of agave fiber and hemp, are also excluded from consideration. Very large format materials such as sugar cane billets (200-300 mm length), chunkwood (100-150 mm diameter), and ISO P65 and larger wood chips (65-100 mm length) are also excluded. Despite these exclusions, nearly all biomass feedstocks and intermediates that are conveyed, stored in bins, fed into reactors, and otherwise handled as bulk materials at biorefineries and associated biomass processing depots remain included for design consideration. A selection of included biomass materials is shown in Figure 2 below.

Figure 2. A selection of materials included in design considerations. Top) wood chips of various sizes and shapes; Middle) 1st stage processed herbaceous crop residues; Bottom) Reactor-ready feedstocks

Introduction to the Solution

Modeling of mechanical behavior of biomass feedstock requires a deep fundamental understanding of its highly nonlinear dependence on environmental conditions. It is not possible to conduct a uniaxial mechanical test and superimpose it to construct the three-dimensional mechanical properties because biomass feedstock is in the form of particulates. Measurement of particulate materials is complicated by the confounding effect of the boundary conditions and non-linear response in orthogonal directions of the sample.

The mathematical foundation for understanding particulate mechanics were being laid at least as early as Charles-Augustine de Coulomb's 1776 essay separating the strength of soil into friction and cohesion components (Heyman 1997).

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[1] Construction and Demolition
[2] Hydrothermal liquefaction / Hydrothermal carbonization

Rankine (Rankine 1856) continued to build out soil mechanics theories by relating the strength back to stress rather than total pressure, and in 1936 Kjellman proposed a true triaxial device (Kjellman 1936) as a way to measure the response to principle stresses on a soil sample (Bell 1965). However, likely due to the complexity of such a device, the idea was not widely realized until the mid-1960s. Since then, several variations of triaxial testers have been designed in search of a better way to understand soil mechanics (Hoyos, Laikram et al. 2009).

Other industries have adapted triaxial testers as well. Work at The Pennsylvania State University (PSU) by Mohsenin and others mentioned above, and extended by Puri's group, applied the theory in the form of a Cubical Triaxial Tester (CTT) (Kamath, Puri et al. 1993, Kamath and Puri 1997). The CTT shown in Figure 3. Left) A photograph of the medium pressure CTT that was fabricated and had been successfully used in other studies by The Pennsylvania State University. Right) An exploded view of the CTT core including one column that applies pressure on one side of the cubical sample. was developed and successfully used to characterize fundamental mechanical properties of particulate materials by Puri's research group. They showed that fundamental bulk mechanical (elastic, elasto-plastic) and rheological (viscoelastic, visco-plastic) properties could be determined and implemented in a reliable flow model.

Cubical Triaxial Tester

The PSU CTT (Figure 3. Left) A photograph of the medium pressure CTT that was fabricated and had been successfully used in other studies by The Pennsylvania State University. Right) An exploded view of the CTT core including one column that applies pressure on one side of the cubical sample.) is a flexible membrane triaxial tester that can apply pressure on three principal orthogonal directions and measure the deformation of six faces of a cubical bulk material sample. Owing to the flexible membrane, a particulate sample can deform purely in response to the applied pressure. The CTT has been used successfully to characterize and model many types of small granular materials, including pharmaceutical, cosmetic, and agricultural products (Mittal and Puri 2002, Mittal and Puri 2005, Karamchandani, Yi et al. 2014, Karamchandani, Yi et al. 2015). The sample size of this tester is 5 cm (2 in) by 5 cm by 5 cm.
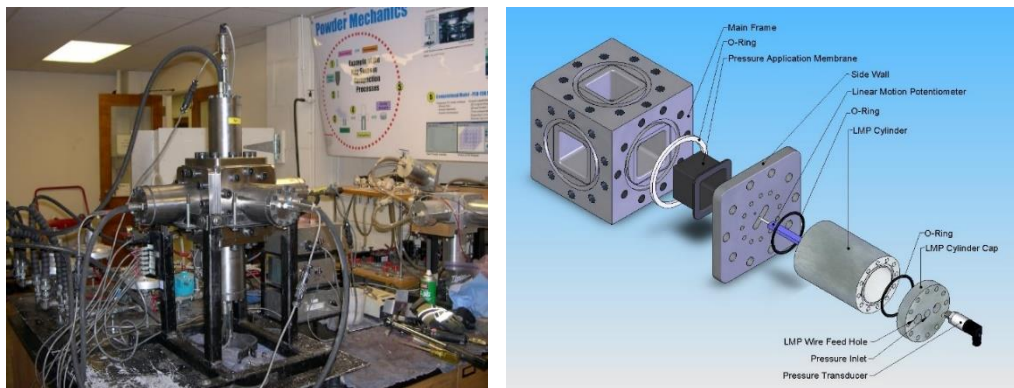
Figure 3. Left) A photograph of the medium pressure CTT that was fabricated and had been successfully used in other studies by The Pennsylvania State University. Right) An exploded view of the CTT core including one column that applies pressure on one side of the cubical sample.

The CTT is particularly useful for quantifying compressive and shear behavior. Figure 4A shows a compressive behavior where an equal amount of pressure is applied to all six faces of a cubical test specimen at the same rate. Resulting deformation of the sample gives us an idea on how much compression there will be, how much more difficult it will become under a specified pressure level, and a history of pressure for further compression. This test is called a hydrostatic triaxial compression (HTC) test.

Figure 4B illustrates the stress path for shear or flow behavior. As mentioned before, when bulk biomass consolidates at a certain pressure it will become harder to shear. This can be characterized with a conventional triaxial compression (CTC) test. In this test a pre-determined consolidation pressure is applied. Then additional pressure is applied only to one axis while pressures on other four faces are maintained. This condition introduces shear in the bulk biomass sample, and eventually there will be a failure of material, which is depicted by a sudden increase in strain at the lower bottom graph.
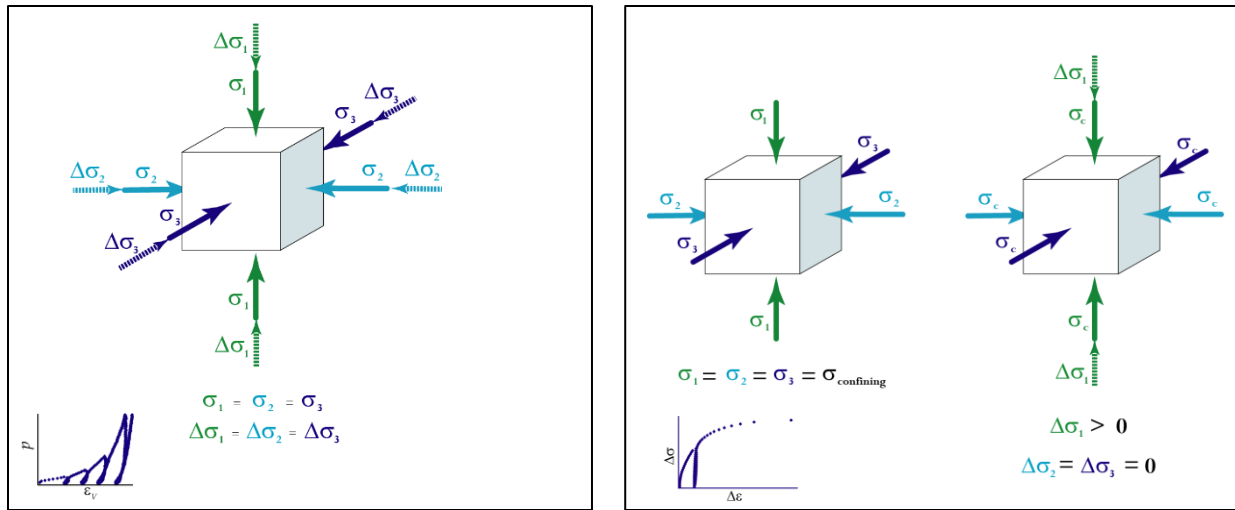
Figure 4. Two modes of triaxial tests. Left) A hydrostatic triaxial compression (HTC) test. In this test mode test specimen are subjected to a same level of pressure on all surfaces introducing hydrostatic pressure to a sample. An HTC test determines the sample's mechanical behavior under compression. Right) A conventional triaxial compression (CTC) test. In this test mode test specimen is first subjected to a predetermined consolidation hydrostatic stress that simulates a stress condition that is of interest. A CTC test determines sample's mechanical behavior under shear. Most importantly, the failure stress can be determined with CTC tests.

The PSU CTT is sufficiently large to test 3 mm and smaller particulate materials. While this scale is consistent with a limited set of biomass feedstock materials, a much larger version of the CTT is necessary for evaluation of a typical bioenergy feedstocks. Forest Concepts (FC) has collaborated with PSU to develop a new CTT specifically for addressing flowability measurements and modeling of biomass feedstocks materials.

CTT Design Requirements

To use such continuum scale constitutive models, mechanical parameters of a chosen constitutive model should be determined with reasonable repeatability. Considering the intrinsic variability of biomass feedstock, a reliable test protocol for biomass characterization is essential. This test protocol should systematically account for the moisture content fluctuation, changes in the friction, and stress state due to different moisture content levels. Biomass feedstocks may also be handled over a wide span of temperatures, such as feeding into an integrated biorefinery. Changes in the friction and stress state due to temperature should be considered.

Sizing Experiment

Arthur's state-of-the-art review of cubical triaxial testers (Arthur 1988) observed that unwanted sample-boundary interactions must be eliminated or controlled. He does not offer guidance about the relationship between sample chamber size and particle size beyond noting that "the ideal of homogeneity cannot be achieved at the scale of individual particles." This observation reinforces the need for our experimental effort to use bulk density as an analogous, but easily actionable, method to assess the relationship between particle and chamber size.
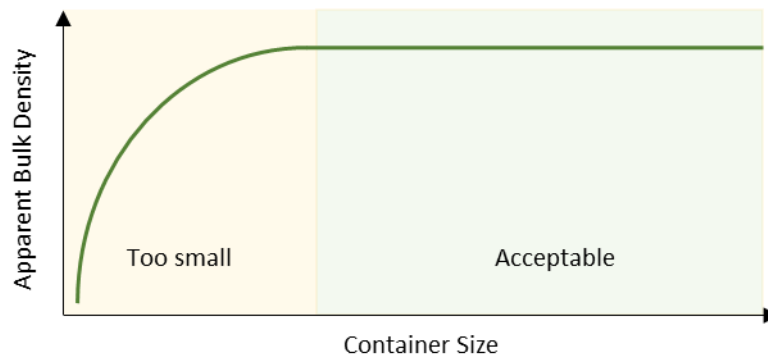
Figure 5. Depiction of the relationship between container size and particulate matter apparent bulk density

Arthur also suggested that there may be a desirable upper limit on the size of a CTT sample chamber as a function of particle size. He observed that stress-arching may occur within large volumes of uniform particles which would affect CTT results. This observation may support designing smaller inserts for our CTT to be used with small particles. Unfortunately,

we found no specific studies of the relationship between particle size and the performance of either triaxial testers or bulk density testing.

If a new CTT is necessary to test a broad range of biomass feedstock materials, it is of paramount importance that it is properly scaled. The particle size of the biomass materials is the principle driver in setting the minimum chamber size. The results from literature reviews and calculations were used to inform the design of an experiment to assess the effect of container size on measured apparent bulk density of a set of biomass materials and feedstocks (Dooley, Slosson et al. 2018). The experimental results suggest that the test container should be at least 200 mm in cross-section and does not need to be larger than 300 mm. However, particle size is far from the only important factor involved in optimizing the CTT chamber size. The following table is used for organizing the considered factors used to refine the chamber size between 200 mm and 300 mm.

Table 2. Selection of factors considered when determining the scale of a biomass CTT

Factor	Direction of Sizing Pressure	Strength of influence
Device Manufacturing Cost	Smaller is better	Weak: Size influences the quantity of structural material and therefore cost of raw materials. However, manufacturing cost is dominated by machining time, especially machining setups. Therefore, savings over this size range is small.
Chamber Handling – Maneuvering	Smaller is better	Medium: The mass of material in the chamber increases cubically with the linear size. Even the largest size chamber is still a manageable mass when filled with wet biomass
Chamber Handling – Filling	No influence	Highly flowable materials will fill faster with a smaller chamber, however, less flowable materials will require more time to "top-off" as the chamber size decreases.
Chamber Handling – Heating	Smaller is better	Weak: Heat transfer into the material is diffusion limited and a function of linear distance to center. There is (approximately) a 1:1 ratio of increased time to increased linear distance to center
Sensor Placement	No influence	Within the size range considered there is no limitations to sensor placement options
Sensor Resolution	No influence	Within the size range considered the selected sensors have sufficient resolution and range of measurement
Control Resolution	No influence	Within the size range considered the pressure and temperature controllers have sufficient resolution
Effect of membrane holder structure	Bigger is better	Weak: There is a minimum structure size due to minimum fastener sizing. The ratio of the "affected zone" (where the sample holder structure takes up space inside of the sample chamber) to sample chamber size decreases as the sample chamber size increases. However, the exact impact is not calculable and, within the size range considered, can be managed through calibration.
Test runtime	No influence	There is no influence on the actual runtime of a test. Only the sample handling time before and after the test described under Chamber Handling

Membrane Shape Experiment

After the size of the sample chamber is settled, there is a question of how to measure the position of the flexible membranes as stress is applied. The Penn State CTT uses a sample holding membrane whose dimension is approximately 5 cm cubed. That is about the volume of a tennis ball. This is sufficiently sized for the only smallest of biomass feedstock materials. Such as 2mm crumbled corn stover shown in Figure 6, but clearly not sufficient for handling most biomass materials.

Note the characteristic deformed concave shape of the compressed sample. The sample was subjected to a hydrostatic stress test, meaning uniform pressure was applied to all 6 sides. The CTT at Penn state utilizes a linear motion potentiometer (LMP) on each face to measure the position of the membrane while pressure is applied. This technique, which assumes a flat face compression, has been effective and suitable for this scale. However, it was not clear if a central point measurement would be suitable for a larger scale.

Figure 6. Shape of corn stover sample after HTC compression in PSU CTT

The first question regarding flatness is simply *does it matter?* After all, the biomass properties are ultimately derived from the volumetric compression ratio, not the linear compression ratio. To answer that question, we explored the implications on the range of volumetric compression ratios one might reasonably calculate based on a single point measurement in the middle of each side membrane. The two deflection shapes were 1) a flat plane of shrinking edge length as the 6 sides of the cube impacted each other, and 2) each membrane resembled a 4-sided pyramid pushing into the cube of biomass with the central point as the apex of the pyramid and the 4 corners at the base of the pyramid coincident with the 4 corners of the uncompressed membrane. The relationship between linear compression ratio and volumetric compression ratio for the two shapes are as follows:

1) Flat deflection (Figure 7, Middle)

Assuming all six sides of the cube compress evenly, the edge length of a side can be calculated from the deflection of two abutting sides. For example, consider the cube of Figure 7 below. If membranes are all under compression then the effective horizontal edge length of face 2 is reduced by the amount of deflection of faces 1 and 6. Similarly, the effective vertical edge length of face 2 is reduced by the amount of deflection of faces 3 and 4.
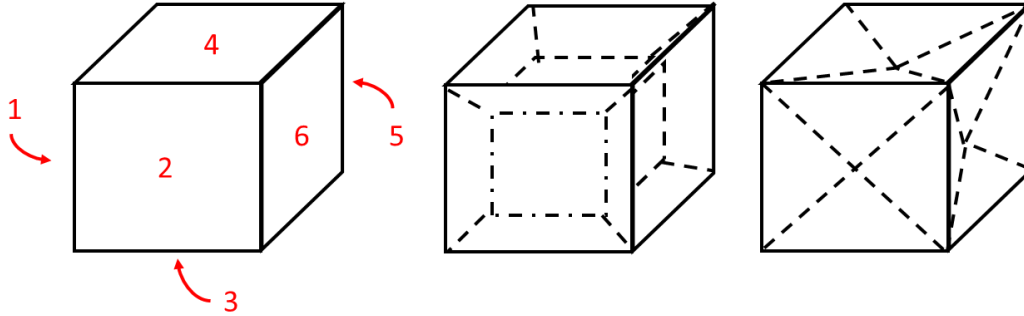

Figure 7: Left) Cube with numbered faces, Middle) Flat deflection, Right) Pyramid deflection

Equation 1 below converts the linear compression ratio to the volumetric compression ratio under the flat membrane assumption.

$$\forall_{flat} = (1 - \mathcal{L})^3$$

Equation 1. Linear to Volumetric: Flat Membrane

Where

$\forall$ = Volumetric compression ratio, and

$\mathcal{L}$ = Linear compression ratio

2) Pyramid deflection (Figure 7, Right)

Similar to the flat deflection method, this analysis assumes uniform deflection on all six sides, but the cube becomes less and less cubic as compression increases. Ultimately the pyramid point of all six faces will meet in the middle of the cube at the same time all pyramid edges come together in their respective corners. The conversion from linear compression ratio to volumetric compression ratio is as follows:

$$V_p = \frac{l^2 h}{3}$$

Equation 2. Volume of a Pyramid

Where

l = Length of the cube side

h = Height of the pyramid

$$V_0 = l^3$$

Equation 3. Volume of a cube

$$h = \frac{\mathcal{L}}{2} \times l$$

Equation 4. Height of pyramid relation to linear compression ratio

$$V_c = V_0 - 6V_p$$
$$= l^3 - \frac{6l^2}{3} h$$
$$= l^3 - \frac{6l^3}{3} \frac{\mathcal{L}}{2}$$
$$= l^3 (1 - \mathcal{L})$$

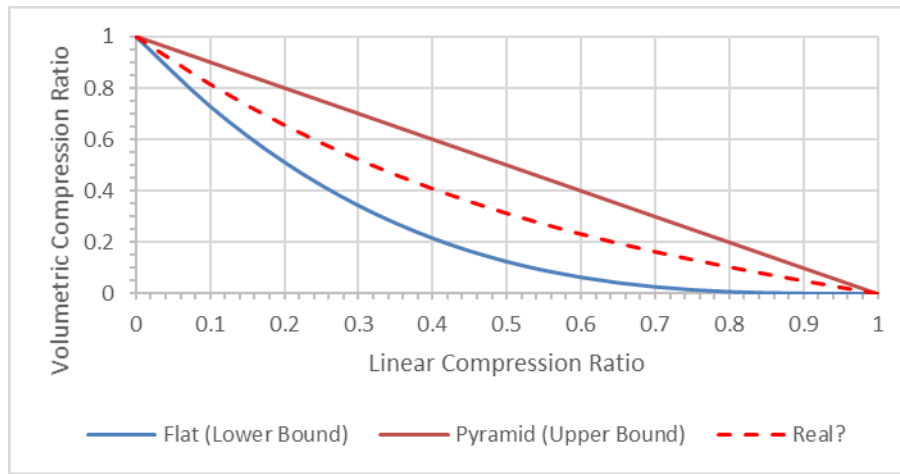**Equation 5. Volume of a cube as function of linear compression ratio**

$$\forall_{pyramid} = \frac{V_c}{V_0}$$
$$= \frac{l^3 (1 - \mathcal{L})}{l^3}$$
$$= (1 - \mathcal{L})$$

Equation 6. Linear to Volumetric: Pyramid Membrane

A plot of the linear compression ratio to volumetric compression ratio results of the two assumed membrane shapes is shown in Figure 8 below.

Figure 8. Comparison of Volumetric Compression ratios by calculation method

The two assumed shapes for the deflected membrane were chosen as the boundaries of possible deflection shapes. In other words, if only one membrane position sensor were installed on each face, then the true volumetric compression ratio would be known to be at least equal to the pyramid method but no more than the flat method. The real volumetric compression ratio is likely somewhere in the middle as indicated by the "real?" dashed line in the figure above.

We can now answer the *does it really matter* question posed at the beginning of this section. Clearly, the answer is *yes*. We expect linear compression ratios as high as 0.65, based on preliminary data in from the PSU CTT, and the two methods diverge rapidly even at low linear compression levels. Therefore, a biomass scale CTT should be capable of detecting the shape of the membrane faces, not just a central point.

Additional Considerations

Upon surveying related literature, the landscape of current devices, consultation with PSU, and review of preliminary experimentation a set of functional and technical requirements for a biomass scale CTT were established.

Functional

- 250 mm Cubic sample holder as established by experiment

- Long term design for up to 2MPa stress application to cover for models related to plug screw feeders, but short-term application of up to 350 kPa relevant to atmospheric hoppers and augurs

- Biomass moisture levels 0.5% (wb) to saturated

- Minimum temperature span of Ambient to 150°C

Technical

- Deformation Resolution of 0.1% linear strain

- Membrane surface mapping with minimum of 9 points per face

- Control pressure resolution +/- 0.6 kPa

- Sensing pressure resolution +/- 0.3 kPa

- Sampling frequency ≥ 1 Hz

Mechanical Design

In pursuit of achieving the function and technical requirements in a safe and user-friendly manner, the device was divided into three major mechanical components: the sample holder, the CTT body, and the frame. Each will be looked at in turn.

Sample Holder

In the PSU CTT a very thin, frameless membrane served as the sample holder. That holder was filled in a rigid container and then removed from the filling container and very carefully transferred to the CTT body. While transferring, the sample did not have any rigid support. This works well enough for materials with a small amount of cohesion, such as wet biomass or powders with static attraction, but other materials could substantially deform from the cubic shape before completing the transfer. Once in position, the biomass and thin frameless holder would slide down into a cavity formed by the primary pressure application membranes. Note, there are two layers of membranes in this configuration. At very low pressures, there is a possibility of membrane separation causing an error in the linear displacement measurement. While functional, there

was always some small concern about non-uniformly pre-compressing the material along the z-axis (up/down) when the sample bumped into the bottom primary membrane. Additionally, a primary membrane rupture, such as may happen in shear (CTC) tests, required a tear-down of the body of the CTT to replace the ruptured membrane. Often, a ruptured membrane also resulted and bent the LMP's which would then need replacing as well.

While the strategy employed in the PSU CTT works well, it is not suitable to the biomass feedstock scale CTT. None of the biomass materials considered reliably sustain their shape as an unsupported 250 mm cube. Additionally, utilizing a single layer membrane is desirable for reducing potential for measurement errors at very low pressures where a double membrane setup may exhibit an undetectable gap between membranes. Upon considering several possibilities, a rigid sample holder frame shown (Figure 9) with individual flat sheet membranes was designed. The design also includes provision for a rigid external bottom plate, fixed or removable solid side plates, and a removable lid.

Figure 9. Biomass scale CTT sample holder with fixed rigid side plates installed

There is a "neutral plane" on each face where the membranes are flat and relaxed. When the sample chamber is filled, it is best if the membranes remain in the neutral plane position for the most cubic of initial biomass shapes. Some test scenarios or stress paths may cause the membranes to expand beyond the neutral plane. Removable side plates are used in conjunction with those stress paths, but generally, the fixed rigid side plates are preferred. Fixed plates, as shown Figure 9, also reduce the number of steps taken by the operator for each test run, thereby reducing the test turn-around time. Also, rigid side plates ensure the biomass material remains secure in its cubical shape during the entire loading and pre-test process. Only with the application of prescribed stress does the sample begin to deform from its initial cubical shape.

While the side plates are rigid, they are not solid as can be clearly seen in Figure 9. The unusual pattern of slots in the plates allow measurement of the membrane surfaces. Another paper will describe the membrane position sensing and analysis details, but an overview is included in the CTT body discussion below.

Another consideration that greatly increased the complexity of designing a CTT is the need for the sample to remain at atmospheric air pressure while stress (provided by compressed air) is applied to all six sides of the cube and without disturbing potential shear planes that may develop within the biomass sample. To achieve this, the sample holder has drain ports in each of the 8 corners. The ports in the corners align with ports in the CTT body to provide continuous exchange of air within the biomass material to atmosphere outside of the CTT body.

Figure 10. Example of laser traces and location of fixed frame of sample holder. Axis marks indicate distance (meters) from the center point of the sample holder

The sample holder has a removable lid for filling and removing biomass. It also is placed into another rigid body where ports must align and seal. Thus, the frame cannot be symmetrical. Nonetheless, once assembled and in-place, the membrane surfaces adjacent to the biomass are fully symmetrical. This ensures that asymmetrical test measurements are directly related to asymmetrical aspects of the biomass material.
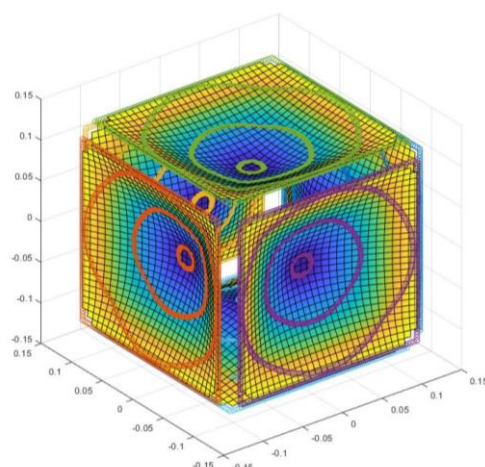
CTT body

The purpose of the CTT body is to house the sample chamber and serve as a pressure vessel tied to each of the three principle axes. The body also holds the sensors necessary for sensing the motion of the sample holder membranes. Operating at elevated temperatures precludes the possibility of using multiple LMP devices to measure membrane position. All but a few electronic devices fare poorly in a 150°C pressurized environment.

To meet the 9-point minimum position measurement requirement, a rotating 3-laser assembly was selected (Keyence IL-300 Laser displacement sensors with IL-1000 amplifiers). Mounted on a stepper motor for precise speed and position control, the 3 lasers each sweep across the membrane face area at exactly 3 Hz. The radius of sweep is set such that the central laser traces a small radius analogous to the LMP on the PSU CTT. The outermost laser traces a circle just inside the frame or the 250 mm sample holder and the central laser traces a circle at about 120 mm. On a "clean" surface (side plates removed, or the lid) the 3 leasers report back 360 points each per sweep every 1/3 of a second. That is more than enough to

generate a surface map of each membrane. More importantly, even when a substantial portion of the measurement points are blocked by rigid plates or other obscurities, an accurate map of the membrane can still be created. The slots in the rigid plates shown in Figure 9 are specifically positioned to align with the arcs of laser position sensors.

The laser array is mounted outside the pressure vessel to preserve the electronic components from being baked. Additionally, even in the worst-case membrane rupture, the sensors are protected so the operator never needs to worry about replacing sensors. Moving the sensors outside of the pressure vessel also has a downside. Somehow, the sensors must "see" through the pressure vessel walls. Upon consulting the book *Fundamentals of Inorganic Glasses* (Varshneya 2013), the author, and glass manufacturers, a chemically treated multi-layer soda-glass chamber endcap was designed. The specific glass treatment, layer thickness, number of layers, and interlayer material are all important for safe operation at the temperatures and pressures of interest in the biomass scale CTT. Carefully specified SolidWorks® simulations were performed to verify the integrity of the glass at full pressure and temperature with at least a safety factor of five. Each of the pressure chamber assemblies was also proof tested to 2x the operating pressure in an isolated environment prior to installation.

Figure 11. CTT body with lid raised and partially inserted sample holder. Sample holder shown with removable side plates.

The cubic body (shown in Figure 11) is an assembly of four identical side plates, a base plate, and a removable top plate. Affixed to the body plates are the pressure chambers. Within the walls of the pressure chambers are electric insertion heaters and resistance temperature detectors (RTDs). Outside of the pressure chambers are the laser arrays and array covers. All six of the pressure chamber assemblies are identical to minimize component variations. Also, each assembly is indexed in such a way that it is not possible to misalign the laser array zero-angle position.
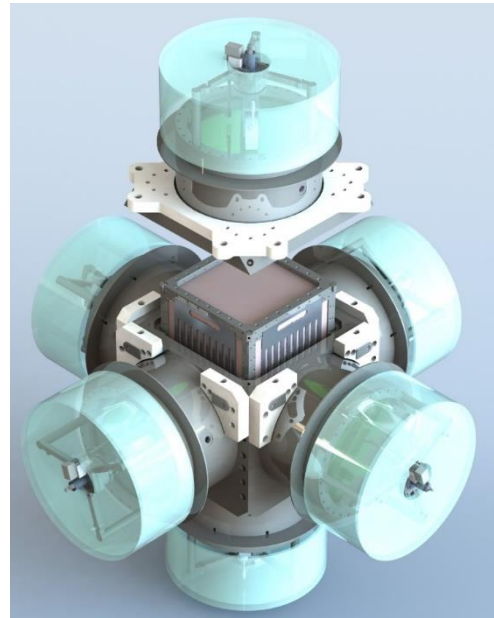
The CTT body is designed to handle up to 2 MPa (290 psi). To make the lid easily removable, it was desirable to use a minimum number of lid hold-down bolts. At the same time, it was also desirable to minimize the torque of the hold down bolts for managing operator exertion. Other hold down methods such as toggle clamps or pin locks were considered, but for safety and simplicity, bolts were ultimately selected. Anybody that maintains machines knows the bolt threads invariably stretch over time which can damage the receiving threads. For easy maintenance and preservation of the body plates, mounts were made to hold standard nuts into which the hold-down bolts threaded. After a certain number of cycles, both bolts and nuts are replaced.

To provide maximum utility of the CTT body, a set of transition blocks take up the space between the CTT body and the sample holder. In the event that a different size of sample holder is desired (as Arthur suggests may be the case for smaller biomass materials), or should the sample holder frame be re-designed to meet some future criteria, the transition blocks may also be re-designed to match the new sample holder to the existing CTT body. The transition blocks also provide sealing between the sample holder frame and the pressure chambers. At the time of this writing, the current configuration had fully isolated Z (up/down) chambers, but the X-Y (horizontal axis) chambers are open to each other. Transition blocks also manage the passageway between the sample holder drain ports and the CTT body drain ports.

Frame

The biomass scale CTT fully loaded sample holder may weigh upwards of 25 kg (55lbs) when testing wet material. Operator safety is always a top priority. Therefore, a lifting aide was built into the frame. The lifting aide also reduces the possibility of the operator coming into contact with the hot CTT body during heated testing. It also helps securely move the sample holder with finesse from a cart into the body and back out of the body at the end of the test. Similarly, the CTT body lid (top pressure chamber shown lifted in Figure 11) is very heavy. It too has a lifting aide to prevent operator strain. Both lifting aids are suspended from a track to allow translation of the lid and sample holder away from the CTT body. Figure 12 shows the CTT body positioned on the frame.

Due to the size, weight, and power requirements a device such as this may end up in a space that is more akin to a shop or loading dock that a nice lab space. For convenience, the frame of the CTT provides a workstation where the operator can take notes and/or place required companion bench-scale equipment such as a scale and a pycnometer. The frame is also pallet jack friendly from all four sides.
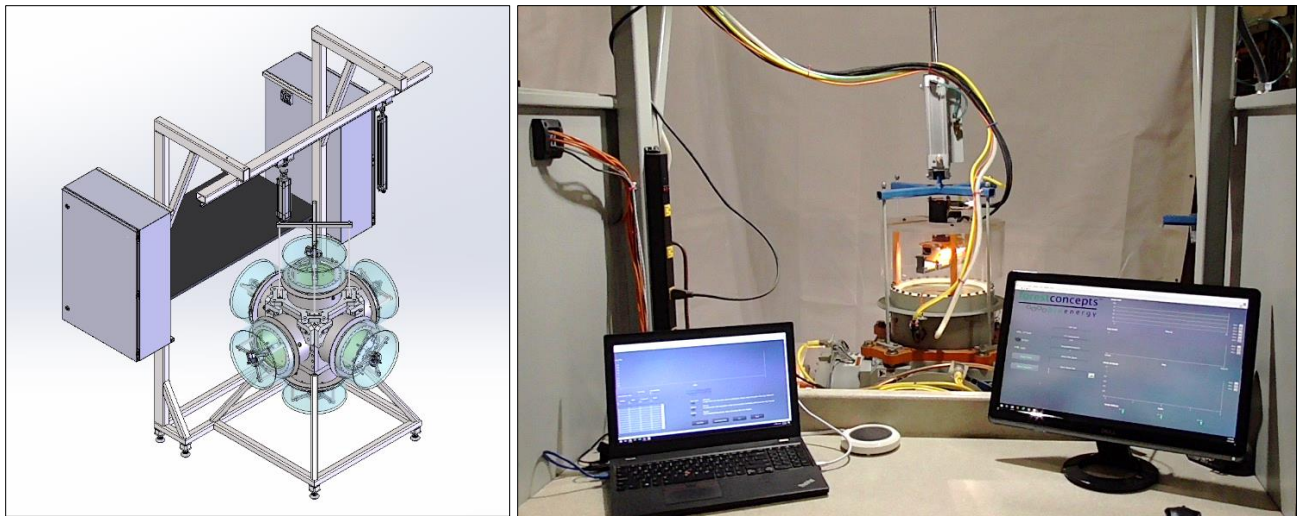
Figure 12. Left) CAD model of CTT body on frame with build-in operators' station and lifting aids. Right) view of CTT from operator's station.

The CTT frame also provides a place to hang the electrical cabinets, which house some sensor components, the DAQ, and power management components. Additionally, "low" pressure tests utilize shop air at 8 bar (116 psi) for applying pressure. However, the pressure controllers are sensitive to contamination and heat. Therefore, the framework also provides mounting for filtering incoming air and cooling of air relieved from the CTT so as to not bake the pressure controllers.

Validation of biomass hopper flow models

As reported in *Computational Modeling of Continuum Scale Constitutive Equations to Improve Biomass Feedstock Material Handling and Conveying Systems* (Yi, Puri et al. 2019), an experiment was performed to validate the technique of model development based on CTT data sets. The initial data set that was utilized in the validation experiment was generated by the smaller CTT at Penn state since the CTT at Forest Concepts had not yet been completed. Nonetheless, output generated from the biomass scale CTT is comparable to that in this validation study. Details are available in the study itself. In short, a hopper that can change wall angle was built. The initial flow patterns of air-dried 2 mm Crumbled corn stover and 1 mm Douglas fir Crumbles from chips were observed. Flow simulation outputs by various models and typical results of hopper flow experiments are shown in the Figure 13.
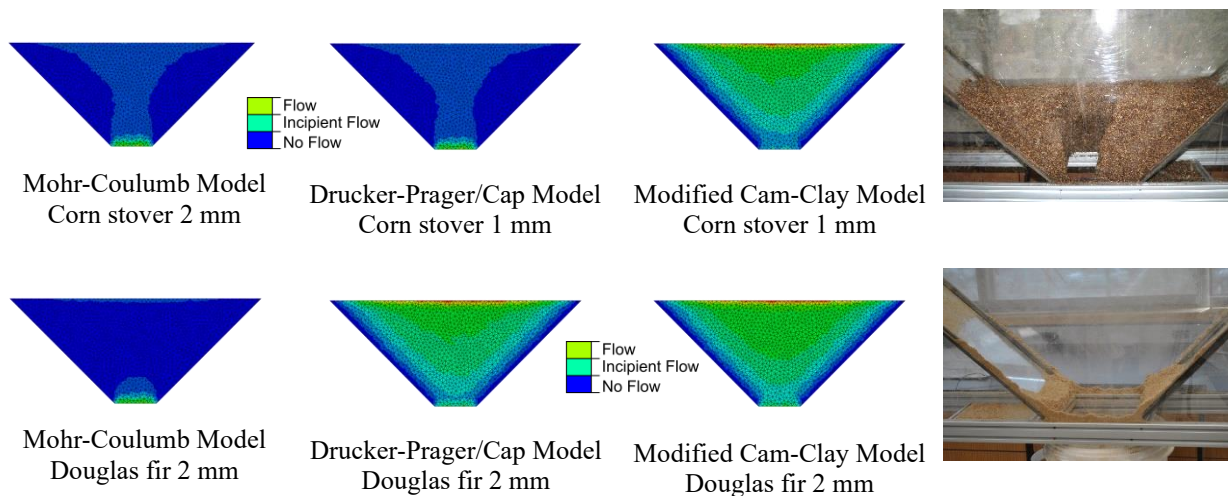

Figure 13. Hopper flow model simulation results of 2mm Crumbled corn stover and 1mm Douglas fir Crumbles from chips using Mohr-Coulomb, Druker-Prager/Cap, and modified Cam-clay models. Finite element simulation results are compared with experimental hopper flow of corn stover 1mm and Douglas (Yi, Puri et al. 2019)

Results and On-Going Work

Further details of the analysis and modeling aspects related to the CTT reported here can be found in other related papers such as *Computational Modeling of Continuum Scale Constitutive Equations to Improve Biomass Feedstock Material Handling and Conveying Systems* (Yi, Puri et al. 2018), (Yi, Puri et al. 2019). As this is an on-going project, other results

and documentation are yet to be reported.

Ultimately, we will have a reliable biomass flow model that will predict the flow condition and rate of chosen biomass at different levels of moisture content, temperature, and applied driving forces in the case of a hoper or screw feeder. This tool will produce an engineering design tool and framework that allows reliable determination of the flowability of biomass in specific feeding systems. Furthermore, this quantitative information will make it possible to design biomass conveying systems in Integrated Biorefineries. For example, we can utilize the findings from friction between bulk biomass and wall so that we can choose right materials and dimensions of hoppers. As a result, we will be able to operate biomass feeding with minimal downtime.

References

Arthur, J. R. F. (1988). Cubical devices: Versatility and Constraints. Advanced Triaxial Testing of Soil and Rock, ASTM STP 977. R. T. Donaghe, R. C. Chaney and M. L. Silver. Philadelphia, PA, ASTM International: 743-765.

ASTM (2007). ASTM D2850-03a (2007), Standard Test Method for Unconsolidated-Undrained Triaxial Compression Test on Cohesive Soils. West Conshohocken, PA, ASTM International.

ASTM (2011). ASTM D4767-11, Standard Test Method for Consolidated Undrained Triaxial Compression Test for Cohesive Soils, . West Conshohocken, PA, ASTM International, www.astm.org.

ASTM (2011). ASTM D7181-11, Standard Test Method for Consolidated Drained Triaxial Compression Test for Soils. West Conshohocken, PA, ASTM International.

ASTM (2013). ASTM E873-82(2013), Standard Test Method for Bulk Density of Densified Particulate Biomass Fuels. West Conshohocken, PA,, ASTM International.

ASTM (2015). ASTM D2850-15, Standard Test Method for Unconsolidated-Undrained Triaxial Compression Test on Cohesive Soils. West Conshohocken, PA, ASTM International.

ASTM (2016). ASTM D6128-16, Standard Test Method for Shear Testing of Bulk Solids Using the Jenike Shear Tester, ASTM International, West Conshohocken, PA, 2016, www.astm.org.

ASTM (2017). ASTM D6682-08, Standard Test Method for Measuring Shear Stresses of Powders Using Peschl Rotational Split Level Shear Tester (Withdrawn 2017). West Conshohocken, PA, ASTM International.

Bell, J. M. (1965). Stress-strain characteristics of cohesionless granular materials subjected to statically applied homogeneous loads in an open system. PhD Dissertation. Pasadena, California, California Institute of Technology.

Dooley, J. H., J. Slosson, M. J. Wamsley, H. Yi, V. M. Puri and C. J. Lanning (2018). Effect of Container Size on Measured (Apparent) Bulk Density of Biomass Materials. Auburn, WA, Forest Concepts, LLC: 15.

Forest Products Laboratory (2010). Wood Handbook: Wood as an Engineering Material (Centennial Edition). Madison, WI, USDA Forest Service, Forest Products Laboratory.

Grima, A., B. P. Mills and P. W. Wypych (2010). Investigation of measuring wall friction on a large scale friction tester and the Jenike direct shear tester. 3rd International conference exhibition BulkSolids Europe 2010. G. Kielburger. Wollongong, Australia, University of Wollongong: 1-14.

Heyman, J. (1997). Coulomb's memoir on statics : an essay in the history of civil engineering. London, UK, University of Cambridge.

Hoyos, L. R., A. Laikram, D. Pérez-Ruiz and A. J. Puppala (2009). Modeling unsaturated soil behavior under multiaxial stress states. Amsterdam, IOS Press: 182-185.

Institution of Chemical Engineers (1989). Standard shear testing technique for particulate solids using the Jenike shear cell: A report of the EFCE Working Party on the Mechanics of Particulate Solids. Rugby, England, Institution of Chemical Engineers.

ISO (2016). ISO 17827-1:2016 Solid biofuels -- Determination of particle size distribution for uncompressed fuels -- Part 1: Oscillating screen method using sieves with apertures of 3,15 mm and above. Geneva, Switzerland, International Organization for Standardization.

Ittershagen, T., J. Schwedes and A. Kwade (2011). "A new powder tester to investigate the anisotropic consolidation behaviour." Powder Technology 211(1): 85-89.

Jenkins, B. M. (1989). Physical Properties of Biomass. Biomass Handbook. O. Kitani and C. Hall. New York, NY, Gordon and Breach Science Publishers: 860-891.

Jenkins, B. M. and H. R. Sumner (1986). "Harvesting and Handling Agricultural Residues for Energy." Transactions of the ASABE 29(3).

Kamath, S. and V. M. Puri (1997). "Measurement of powder flow constitutive model parameters using a cubical triaxial tester." Powder Technology 90(1):

59-70.

Kamath, S., V. M. Puri, H. B. Manbeck and R. Hogg (1993). "Flow Properties of Powders Using Four Testers — Measurement, Comparison and Assessment." Powder Technology **76**(3): 277-289.

Karamchandani, A., H. Yi and V. M. Puri (2014). Development and Validation of Predictive Relationships between Mechanical Properties of Ground Willow and Pellet Quality. ASABE Paper No. 141893325. 2014 Montreal, Quebec Canada July 13 – July 16, 2014. St. Joseph, Mich., ASABE: 1.

Karamchandani, A., H. Yi and V. M. Puri (2015). "Fundamental Mechanical Properties of Ground Switchgrass for Quality Assessment of Pellets." Powder Technology **283**(October): 48-56.

Kjellman, W. (1936). Report on an apparatus for consummate investigation of the mechanical properties. Proc. 1st Int. Conference on Soil Mechanics and Foundation Engineering. Cambridge, MA: 16-20.

Mittal, B. and V. M. Puri (2002). Determination of visco-elastoplastic properties of a powder using cubical triaxial tester. St. Joseph, MI, ASAE.

Mittal, B. and V. M. Puri (2005). "Rate-Dependent Elasto-Viscoplastic Constitutive Model for Industrial Powders. Part 1: Parameter Quantification." Particulate Science and Technology **23**(3): 249-264.

Mohsenin, N. N. (1965). "Physical Properties of Agricultural Products." Transactions of the ASABE **8**(1): 25-29.

Mohsenin, N. N. (1970). Physical Properties of Plant and Animal Materials. Volume 1. Structure, Physical Characteristics and Mechanical Properties. New York, Gordon and Breach Science Publishers.

Rankine, W. (1856). "On the stability of loose earth." Phyilosophical Transactions of the Royal Society of London **147**.

Schwedes, J. (2003). "Review on testers for measuring flow properties of bulk solids." Granular Matter **5**(1): 1-43.

Scott, O. J. and S. Keys (1992). The Variation of Boundary Friction for Granular Products. Proceedings of the 4th International Conference on Bulk Materials Storage, Handling and Transportation (ICBMH 92), Wollongong, Australia: 279-286.

Varshneya, A. (2013). Fundamentals of inorganic glasses. Sheffield, England, Society of Glass Technology.

Yi, H., V. M. Puri, C. J. Lanning and J. H. Dooley (2018). Computational modeling of continuum scale constitutive equations to improve biomass feedstock material handling and conveying systems. ASABE paper number 1800867. 2018 ASABE Annual International Meeting. St. Joseph, MI, ASABE: 4.

Yi, H., V. M. Puri, C. J. Lanning and J. H. Dooley (2019). Computational modeling of continuum scale constitutive equations to improve biomass feedstock material handling and conveying systems. 2019 ASABE Annual International Meeting. St. Joseph, MI, ASABE: 1.