PREDICTIVE RELATIONSHIPS FOR QUALITY ASSESSMENT OF DENSIFIED BIOMASS USING FUNDAMENTAL MECHANICAL PROPERTIES OF GROUND CORN STOVER, SWITCHGRASS, AND WILLOW

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by

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Abstract

Pelletization is one of the promising technologies utilized for densification of biomass, which results in better handling, storage, and reduced transportation cost of biomass. This process also increases the energy density and bulk density of biomass; thus addressing the bulkiness issue. Many studies have been conducted to determine optimum conditions for pelletizing. However, no relationship between the mechanical properties of ground biomass on the pellet quality is published in the literature. Therefore, the goal of this research is to develop and verify a predictive relationship between mechanical properties of the ground biomass and the quality metrics of pellets. Development of such a relationship will reduce the operational difficulties of densification process and will provide the framework for addressing better the issues such as quality control during pelletization.

To accomplish the goal, three selected biomass found in abundance in the Northeast U.S., namely, corn stover, switchgrass, and willow, were ground with two different screen sizes; 3.175 mm, and 6.35 mm and were conditioned at two different levels of moisture content; 17.5%, and 20% (w.b.). Hydrostatic triaxial compression (HTC) tests were performed using the Cubical Triaxial Tester (CTT) to determine the various mechanical properties, namely bulk modulus, compression index, and spring back index. They were determined using the HTC test at unloading pressures of 20, 45, 70, and 95 kPa. Pellets were formed by ground biomass using a lab-scale pellet mill. The physical properties such as diametral tensile strength, axial compressive strength, pellet density, and durability were determined.

Based on the determined \(D_{50}\) values for all three biomass materials, the size-reduced biomass with 6.35 mm screen size is higher by 30-40% than reduced with 3.175 mm screen size. For ground corn stover, the value of bulk modulus initially decreases slightly at 45 kPa and then increases till 95 kPa, whereas the value of bulk modulus of smaller screen size increases with increasing pressure. The bulk modulus values of switchgrass and willow increases with increase in the isotropic pressure for all four conditions. Compression index increases with pressure for all three materials. Spring-
back index values decrease with the increase in the isotropic pressure, however, the values slightly increase at high pressure, in some cases.

Pellet durability for all three materials is more than 80%. The pelletization process compresses the loose material nearly 12-14 folds for corn stover, 7-9 folds for switchgrass and 5-7 folds for willow, respectively. The achieved relative pellet densities are 96%-98% for corn stover, 67%-88% for switchgrass, and 91%-98% for willow, respectively. Diametral tensile strength is 4-5.8 MPa for corn stover, 0.9-3.8 MPa for switchgrass and 0.8-5.7 MPa for willow pellets, respectively. Axial compressive strength is 8-11.5 MPa for corn stover, 0.8-5.7 MPa for switchgrass and 1.5-8.5 MPa for willow pellets, respectively.

Correlation coefficients are calculated to evaluate the relationships and their level of significance after the determination of the physical and mechanical properties of ground biomass and pellets. The regression equations between pellet quality and mechanical property having $R^2$ value higher than 90% are selected for prediction. For switchgrass, spring-back index and compression index are found to be most suitable for predicting diametral tensile strength, and axial compressive strength, whereas, for willow, compression index is found to be most suitable for predicting diametral tensile strength, and axial compressive strength, respectively. For the validation of the developed relationships, switchgrass and willow ground with 6.35 mm screen size is conditioned at 18.75% w.b. to form pellets. The percent difference between the measured and predicted values for switchgrass and willow wood for axial compression strength and diametral compression strength are from 5.79% to 11.54%; well within the mean percent spread of 71% for the measured values. Based on the validation, it can be concluded that the strength quality metrics of switchgrass and willow pellets can be predicted using size reduced materials’ compression index and spring-back index, and compression index, respectively.
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Chapter 1. Introduction and Justification

Under the present scenario of depleting conventional energy sources, solid fuel from bio-based renewable materials is a potential source of alternative energy. In the United States, potentially about 111 million dry tons of primary crop residues are sold annually from the farm at feedstock prices of $60 per dry ton and more than three-fourths of this residue is corn stover (U.S.D.O.E., 2011). Switchgrass is a perennial, warm-season grass, which is one of the prevailing species of the central North American tallgrass prairie. Wood residues such as chips, shavings are very commonly used for bioenergy generation. Therefore, corn stover and other bio-feedstocks such as switchgrass and wood wastes are prime candidates of bio-renewable materials for value added products such as pellets. Pellets are a densified form of biomass thus making it easy and efficient to handle, transport, and utilize.

The bulkiness, i.e. low bulk density, of biomass is one of the major barriers in its effective utilization as a biofuel. Loose cut biomass has bulk density of less than 150 kg/m$^3$ depending on the particle size (Gilbert et al., 2009), for example, 60-120 kg/m$^3$ for switchgrass. The bales of corn stover of 23-40% wet basis moisture content generally have a low bulk density in the range of 95-150 kg/m$^3$ (Shinners et al., 2003). For the efficient use of bio-feedstock as an energy source, it is very important to form a densified product of biomass, which will not only provide product of higher energy density but will also result in a higher bulk density of solid biofuel than in the original form. Study conducted by Morey et al. (2009) cites many research articles that have been conducted in the field of logistics involving bales and ground biomass (Brechbill and Tyner, 2008; Cundiff and Grisso, 2008; Petrolia, 2008; Sokhansanj and Turhollow, 2004; Sokhansanj et al., 2009; Hess et al., 2007). Those studies have recommended increasing the bulk density of biomass to reduce the transportation cost and improve the handling of biomass.

An economic and feasible way to utilize biomass as an energy source is to densify it near to the source of raw material (Clarke, 2011). Efficient use of bio-feedstock will enable farmers and producers to better utilize the crop residues and will create more employment in the agricultural sector. Industries working in the field of densification of
biomass can benefit from the improved technology, which will help monitor, control, and deliver high quality densified biomass at affordable price.

For efficient production of densified biomass with improved quality, it is important to study the relationship between mechanical properties of ground bio-feedstocks such as compressive properties and quality of densified product. From the aspect of handling and transportation, pellet density, durability and strength of pellets are important quality attributes. Quality metrics of densified biomass such as diametral tensile strength, axial compressive strength, can be determined by using a universal testing machine. Measurements of various properties for both the biomass feedstock as brought to a biomass densification processing facility and the quality of the densified product will allow further understanding of promising methods and approaches for better densification systems and products.

Pelletization is one of the promising technologies for the densification of biomass (Tumuluru et al., 2011; and Stelte et al., 2012). However, pelletization generally lacks quantitative control over raw material, which is in granulated form. The cubical triaxial tester (CTT) can be used in the determination of fundamental mechanical properties of ground biomass feedstock. The CTT has been successfully used in characterization of fundamental mechanical properties of various granular materials (Mittal and Puri, 1999a and b; Mittal et al. 2001; Yi et al., 2001, 2002; and Pandeya and Puri, 2010). Kamath and Puri (1997) and Li and Puri (1997) developed the flexible boundary cubical triaxial tester for low (0 to 100 kPa) and medium (0 to 14 MPa) pressures, respectively. Among other stress paths, conventional triaxial compression (CTC) and hydrostatic triaxial compression (HTC) tests are the most common stress paths for characterization can be performed using the CTT. The HTC test is basically an iso-stress compression test, wherein all principal stresses are the same and increase at the same rate; whereas, the CTC test is a shear test wherein stresses in one direction increase at a specific rate while stresses in the other two directions remain unchanged.

Researches are needed to bridge the knowledge gap in the quality of pelletized biomass and mechanical properties of the ground biomass feedstock and to gain deeper insights to the mechanics of pelleting; such studies will provide possible improvements in densification methods. Additionally, studies will lead to standardized quality control of
bio-feedstock and produced pellet. Therefore, this study attempts to develop predictive relationships between mechanical properties of ground biomass and resulting characteristics of pellet quality.
Chapter 2. Review of Literature

2.1 Introduction
This literature review presents an overview of the densification process and the past research work performed in the area of mechanical properties of ground biomass and pellet quality. The review addresses the need of densification, various factors affecting the process and mechanical properties and physical properties of ground bio-feedstock and densified biomass. In this chapter, some of the key research findings are discussed and analyzed in order to identify the knowledge gap in this area for formulating a hypothesis leading to better understanding how and why of pelletization.

2.2 Biomass as an Energy Source
Biomass is an energy resource derived from organic matter, which includes wood, agricultural waste, and other living-cell materials that can be burned to produce heat energy. It also includes algae, sewage, and other organic substances that may be used to make energy through chemical processes (U.S.D.O.E., 2005).

In the U.S., biomass supplies approximately 3% of total energy consumption in the form of electricity, process heat, and transportation fuels (U.S.D.O.E., 2005). There is tremendous interest in using bio-feedstocks such as corn stover and switchgrass for producing biofuels, and in using bio-products to reduce dependence on conventional fuel source. In addition to numerous advantages, use of biomass materials in place of fossil fuels would result in low emissions of greenhouse and acid gases (U.S.D.O.E., 2005).

2.3 Densification of Biomass Feedstocks
The material flow in a typical biomass pelleting operation consists of three major unit operations; namely, drying, size reduction (grinding), and densification (Mani et al., 2006). Densification (briquetting, pelleting, or cubing) of a particulate matter is achieved by forcing the particles together by applying mechanical force to create inter-particle bonding, which makes well-defined shapes and sizes such as briquettes, pellets, and cubes (Kaliyan and Morey, 2009a).
As inferred by Fasina and Sokhansanj (1996), the flowability properties of dense pellets are similar to those of cereal grains; thus, conventional grain handling systems often can be used for their handling. This provides an advantage to form pellets over other densified product such as cubes, and briquettes. Pellets are cylindrical in shape. The diameter of pellets ranges from 6 to 8 mm and length ranges from 12 to 15 mm during the process of pelletization. Reported properties for safe and efficient storage and transportation of pellets are that they should have low moisture content (about 8% on wet basis) and a high bulk density, i.e., more than 600 kg/m$^3$ (Mani et al., 2006).

### 2.3.1 Need of Densification

Biomass materials involve significant costs related to handling, transportation, and storage because of low bulk density. One of the solutions to overcome these difficulties faced during transportation and storage of biomass is to densify biomass into briquettes, pellets, or cubes. Densification can increase the bulk density of biomass materials from an initial bulk density of 40 to 200 kg/m$^3$ to a final bulk density of 600 to 800 kg/m$^3$ (Holley, 1983; Colley et al., 2006).

Biomass has lower energy content than conventional sources of energy such as coal, oil, and natural gas, which means that more volume of fuel is required to get the same amount of energy (Clarke, 2011). Densification also increases the energy density of biomass by compressing the biomass to achieve comparable calorific value per unit of volume to conventional energy sources.

### 2.3.2 Factors Affecting Densification Processes

There are several factors such as moisture content, temperature, feedstock constituents, and physical properties of feedstocks, which play an important role in achieving the desired product quality. Therefore, these should be monitored and controlled for successful densification of biomass.

#### 2.3.2.1 Chemical Composition of Biomass

The natural binders found in biomass materials are lignin, crude protein, starch, crude fat, and water soluble carbohydrates (Table 2.1). Among these chemical components, lignin
has a low melting point of about $140^\circ$C. When biomass is heated, lignin becomes soft and exhibits thermosetting properties on melting (van Dam et al., 2004). Biomass from woody material contains higher percentages of lignin and resins as compared to agricultural crops residues (Mani et al., 2006).

Table 2.1 Compositions of corn stover and switchgrass grinds

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Cellulosec</td>
<td>49.4</td>
<td>31.3</td>
<td>30.6-38.1</td>
<td>43.8</td>
<td>44.3</td>
<td>27.8-37.1</td>
</tr>
<tr>
<td>Hemicellulosed</td>
<td>26.2</td>
<td>21.1</td>
<td>19.1-25.3</td>
<td>28.8</td>
<td>30.0</td>
<td>22.4-28.6</td>
</tr>
<tr>
<td>Lignine</td>
<td>8.8</td>
<td>3.1</td>
<td>17.1-21.3</td>
<td>9.2</td>
<td>7.4</td>
<td>13.2-22.5</td>
</tr>
<tr>
<td>Crude Protein</td>
<td>3.6</td>
<td>8.7</td>
<td>NA¹</td>
<td>3.9</td>
<td>1.6</td>
<td>NA</td>
</tr>
<tr>
<td>Starch</td>
<td>0.4</td>
<td>NA</td>
<td>NA</td>
<td>1.0</td>
<td>NA</td>
<td>NA</td>
</tr>
<tr>
<td>Crude Fat</td>
<td>0.7</td>
<td>1.3</td>
<td>NA</td>
<td>0.9</td>
<td>1.9</td>
<td>NA</td>
</tr>
<tr>
<td>Water Soluble Carbohydrates</td>
<td>7.9</td>
<td>NA</td>
<td>NA</td>
<td>2.2</td>
<td>NA</td>
<td>NA</td>
</tr>
<tr>
<td>Moisture Content</td>
<td>5.4</td>
<td>NA</td>
<td>9.8-13.5</td>
<td>5.7</td>
<td>NA</td>
<td>NA</td>
</tr>
<tr>
<td>Ash</td>
<td>11.2</td>
<td>7.5</td>
<td>5.0</td>
<td>5.5</td>
<td>2.5-7.6</td>
<td></td>
</tr>
</tbody>
</table>

a (% of dry matter)

b (range, % of mass)

c Cellulose = acid detergent fiber (ADF) – lignin

d Hemicellulose = neutral detergent fiber (NDF) – acid detergent fiber (ADF)

e Lignin values measured for the biomass materials used in Kaliyan and Morey (2009a) study and in Mani et al. (2006) were acid insoluble lignin contents, whereas the lignin contents obtained from DOE (2007) were total lignin in the biomass materials

f NA = data not available

g Not available for willow wood

During densification, protein and starch also play an important role as a binding agent between particles. Sokhansanj et al. (2005) identified that a feed material, which
contains higher proportions of starch and protein, will produce more durable and higher quality pellets than biomass containing only cellulosic material. Kaliyan et al. (2009) suggested that high-quality briquettes and pellets could be produced from corn stover and switchgrass without adding chemical binders by activating the natural binding components, such as water-soluble carbohydrates, lignin, protein, starch, and fat, present in the corn stover and switchgrass through moisture and temperature.

2.3.2.2 Moisture Content

Moisture content plays an important role in pelleting process for the development of intermolecular forces. Low moisture results in improved density and durability of the densified biomass (Shaw and Tabil, 2007). For biomass densification processes, the optimum moisture content is in the range of 12% to 20% (wet basis, w.b.) at room temperature (Kaliyan and Morey, 2009b). Generally, at moisture content more than 20%, densification may not be possible; likewise, at low moisture content the pellets may not form at all or they are fragile and/or of low quality.

Colley et al. (2006) reported that ground switchgrass at a moisture content of about 20% (w.b.) produced high-quality pellets. This study concluded that moisture content significantly affects the physical properties of switchgrass pellets.

In the study conducted by Wilson (2010) with woody biomass, the optimal moisture content for pelleting was 12% (w.b.). Different species of wood, namely maple, red oak, walnut, lodgepole pine, cedar/dodge fir, both in pure form and mix were used in this study. For densification of woody biomass, Obernberger and Thek (2004) postulated that the optimal moisture content is between 6% to 12% and 8% to 12%, respectively.

Study conducted by Mani et al. (2004) analyzed that corn stover grinds at low moisture content (7%) had higher compressibility than grind at high moisture content (15%) for both hammer mill screen sizes; 1.6 mm and 3.2 mm. At higher moisture content than 15%, corn stover grind exhibited high resistance to compression.

In the aforementioned literature, there is a very large range from 6% to 20% for reported optimal moisture content for densification. There is variation in the reported moisture content even for the same material, which indicates that the effect of moisture content on densification process needs further investigation.
2.3.2.3 Temperature

Durability and strength of densified material are significantly influenced by temperature. During compression at high temperatures, the protein and starch plasticizes and act as binders, which assists in increasing the strength of the pelletized product (Briggs et al., 1999). Hill and Pulkinen (1988) found that the pellet durability of alfalfa increased by about 30–35% when the pelleting temperature was increased from 60 to 104ºC. Mani et al. (2003) and Sokhansanj et al. (2005) also observed that higher temperatures resulted in reduced resistance of the material against equipment components thereby resulting in better quality pellets.

Kaliyan and Morey (2010) showed that if the temperature rise of the biomass grinds due to frictional heating in the briquetting and pelleting machines is in the range of glass transition (i.e., softening) temperature of some of the constituents of biomass materials (i.e., ≥75°C), then strong and durable briquettes and pellets could be produced without steam conditioning.

2.3.2.4 Particle Size

Particle sizes and size distribution along with moisture content are two of the most significant factors affecting the pellet quality. Finer particle sizes generally correspond with greater pellet strength and durability as larger particles serve as fissure points (MacBain, 1966). Several researchers observed that the optimal pellet quality is achieved with a mixture of particle sizes due to increased inter-particle bonding (mechanical interlocking) and the elimination of inter-particle spaces (attractive and adhesive and cohesive forces) (Payne, 1978; Kaliyan and Morey, 2006; Shaw, 2008). Fine particles usually absorb more moisture than large particles and, therefore, undergo a higher degree of conditioning.

Although fine particles produce more durable pellets, fine grinding is undesirable because of increased time and cost of production. Kaliyan and Morey (2009b) postulated that a mixture of different particle sizes would give an optimum pellet quality because the mixture of particles will produce better inter-particle bonding with nearly no inter-particle spaces. Among barley straw, wheat straw, corn stover, and switchgrass, ground
corn stover had the highest bulk density and particle density due to the smallest geometric mean particle diameter when it was ground with hammer mill screen sizes of 1.6 and 3.2 mm (Mani et al., 2006).

2.4 Quality Assessment of Densified Product

Kaliyan and Morey (2006) defined pellet quality in terms of the strength and durability of the pellets. Strength refers to both the impact and compressive resistances. Durability is a measure of the friability of the pellets. The bulk density and durability are properties of a bulk sample of densified biomass, whereas the crushing strength or hardness is considered a property of individual briquettes or pellets (Kaliyan et al., 2009).

According to Wilson (2010), there are several established methods originally developed by the feed industry for the evaluation of these qualities that can be applied to the use of pellets produce for energy systems. These methods were designed to simulate the forces induced on the pellets in the storage and handling process. Test results are used as indicators of the level of breakage in storage and handling as pellets are transported from production to utilization. Of the quality metrics available for pellet testing, durability is the most representative of the forces experienced in transportation, storage, and handling, and is the only physical quality metric considered by industry.

Colley et al. (2006) manufactured switchgrass pellets (die diameter = 4.76 mm) with bulk density of 687 kg/m³, durability of 96%, and pellet hardness of 27 N at a pellet temperature of 85°C and pellet moisture content of 11% (w.b.). Likewise, in Kaliyan et al. (2009) study, switchgrass pellets (die diameter = 9.5 mm) with bulk density of 570 kg/m³, durability of 86%, and pellet hardness of 216 N at a pellet temperature of 81°C and pellet moisture content of 11% (w.b.) were produced.

2.4.1 Pellet Density

Mani et al. (2006) reported that individual pellet density ranges from 1,000 kg/m³ to 1,200 kg/m³ and the bulk density of pellets ranges from 550 kg/m³ to 700 kg/m³ depending on size of pellets. According to Mani et al. (2003), individual pellet density and durability are influenced by physical and chemical properties of the feedstock, temperature and applied pressure during the pelleting process. Study conducted by Shaw
and Tabil (2007) indicated that pellet density was improved by increasing the die temperature, decreasing the screen size (particle size), and decreasing the feedstock moisture content.

### 2.4.2 Durability of Pellets

Durability is a widely used term by pellet manufacturers to quantify pellet quality and is also used to monitor parameters during pelleting process (Winowiski, 1998). According to the ASABE standard S269.4 (ASABE, 2007), the durability of the pellets is determined by tumbling a 100 g sample of pellets at 50 rpm for 10 min in a dust-tight enclosure. Durability is considered high when the computed value is above 80%, medium when between 70% and 80%, and low when below 70% (Tabil and Sokhansanj, 1996; Adapa et al., 2003).

In a study conducted by Colley et al. (2006), durability of the switchgrass pellets increased initially with moisture content and reached a maximum of 96.65% at 8.62% moisture content. Further increase in moisture content reduced durability to 78.44%. According to the durability rating developed by Adapa et al. (2003), switchgrass pellets can be classified to have high durability (89.06% to 95.91%) at moisture contents between 6.32% and 14.84% and medium durability at moisture content values greater than 14.84%. Carroll (2012) reported that willow wood, miscanthus, barley straw, and wheat straw pellets were of high durability (> 95%). Study performed by Wilson (2010) indicated that a woody pellet’s durability depends on the mechanical properties of feedstock rather than its compositional difference.

### 2.4.3 Strength of Pellets

Strength refers to both the compressive and impact resistance of pellets. Compressive resistance (or crushing resistance or hardness) is the maximum crushing load a pellet/briquette can withstand before cracking or breaking (Kaliyan and Morey, 2009a).

Colley et al. (2006) determined the hardness of the switchgrass pellets by using a texture analyzer (model TA-HD, Stable Micro Systems, Surrey, U.K.). A single pellet was placed on the platform of the texture analyzer in its natural position (the radial
dimension was in the same direction as that of the compressive force). A flat plate (50.8 mm diameter) plunger was pressed onto the pellet at a speed of 10 mm/s. Pellet hardness decreased (30.21 to 21.6 N) with increase in moisture content (8.62% to 17%).

2.5 Mechanical Properties of Ground Biomass

The fundamental mechanical properties of a ground bio-feedstock are its genuine signature which can be utilized to develop the relationship with the quality parameters of pellets. A set of selected mechanical properties that were determined are summarized in Table 2.2. These and other parameters were correlated to the loading rate, confining pressure, and unloading-reloading stress level (Mittal and Puri 1999a, 1999b, and 2003).

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Significance</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bulk Modulus</td>
<td>Measure of the material’s resistance to volumetric deformation at a given isotropic pressure</td>
</tr>
<tr>
<td>Compression Index</td>
<td>Quantifies compressibility of powder at a given isotropic pressure</td>
</tr>
<tr>
<td>Spring-back Index</td>
<td>Quantifies powder’s ability to recover/relax after release of stress at a given isotropic pressure</td>
</tr>
<tr>
<td>Failure Stress (Strength)</td>
<td>Ultimate value at which a given powder fails during shear loading. Leads to the development of critical state line (CSL) or fixed yield surface</td>
</tr>
<tr>
<td>Shear Modulus</td>
<td>Measure of material’s resistance to change in shape at a given pressure difference in principal directions</td>
</tr>
</tbody>
</table>

Determination of the fundamental mechanical properties of ground bio-feed materials can be done with the cubical triaxial tester (CTT). Data from the CTT can be used to analyze and evaluate the mechanical responses for the determination of the fundamental mechanical properties associated with elastic, elastoplastic, and rate-dependent responses of selected ground bio-feedstocks.

2.5.1 Cubical Triaxial Tester

The cubical triaxial tester (CTT) is the most important device for this study, which is capable of measuring the true 3D stress-strain behavior of granular materials over a wide range of compression and extension conditions without the confounding
effect of die-wall friction. Kamath and Puri (1997) and Li and Puri (1997) developed the flexible boundary cubical triaxial tester for low (< 100 kPa) and medium (< 14 MPa) pressures, respectively. CTT has been successfully used in characterization of fundamental mechanical properties of various granular materials (Mittal and Puri 1999a and b; Mittal et al. 2001; Yi et al, 2001, 2002; and Pandeya and Puri, 2010).

The low pressure CTT, developed by Kamath and Puri (1997), was originally designed for pressure ranging from 0 to 700 kPa with the precision of 7.5 kPa. It was modified to control and measure pressure more accurately and thus has lower pressure range from 0 to 100 kPa with the 0.1 kPa precision. A photograph of the CTT is shown in Figure 2.1.

Figure 2.1: Photograph of the assembled cubical triaxial tester (CTT)

Hydrostatic triaxial compression (HTC) and conventional triaxial compression (CTC) tests can be conducted using the CTT to determine the various constitutive parameters. The HTC test is basically an isotropic compression test, whereas, the CTC test is a shear test. The HTC and CTC tests are described below.

2.5.1.1 Hydrostatic Triaxial Compression (HTC) Test

In HTC tests, the test sample is subjected to an isotropic loading and the stress state is the same in all three principal directions such that \( \sigma_1 = \sigma_2 = \sigma_3 \), where, \( \sigma_1 \), \( \sigma_2 \), and \( \sigma_3 \) are the major, intermediate, and minor principal stresses, respectively (Figure 2.2). The pressures are then increased uniformly according to the pre-determined stress paths.
2.5.1.2 Conventional Triaxial Compression (CTC) Test

In CTC tests, equal pressure is maintained initially on the six faces of the cubical specimen until a pre-determined pressure value (known as the confining pressure, $\sigma_0$) is reached such that $\sigma_0 = \sigma_1 = \sigma_2 = \sigma_3$ (Figure 2.3). After the confining pressure is reached, four sides of the CTC (i.e., right and left, and front and back faces) are maintained at the confining pressure value, whereas, the pressure is increased on the top and bottom faces until failure.

The review of literature presented in the previous sections gives the overview of the work done by various researchers in the area of biomass pelletization. From the review of literature it can be stated that a few researchers studied mechanics of densification of bio-feedstock and worked to identify the factors affecting the process. However, there have been only a limited number of studies on mechanical properties of biomass and its effect on densification.

Mani et al. (2006) investigated the effect of mechanical properties of grass on compressed pellets properties including density and asymptotic modulus. Colley (2006) studied the effect of moisture content, temperature and size of die on the switchgrass pelletization. Shaw (2008) studied pressure and temperature of die and particle size and moisture contents of feedstock to characterize the resulting pellet’s density using poplar and wheat straw. Adapa et al. (2009) compared energies required to compact different species at different pressures. However, these studies do not address characteristics of densified biomass in terms of the desirable pellet quality.

From the review of literature, it can be concluded that there are no identified or quantified relationships between the effects of mechanical properties of ground biomass and the parameters related to pellet quality. There is, thus, a need to identify the characteristics of densified biomass and develop the relationships of these characteristics with quantitative mechanical properties of ground biomass and lay the foundation for rational basis of how and why of mechanics of pellet formation.
Chapter 3. Goal, Hypothesis and Objectives

3.1 Goal

From the review of literature, it can be stated that there have been several studies characterizing mechanical properties of biomass and densification process of biomass. However, the relationships among these properties have not been studied to date. Development of such relationships will lead to processing steps with less operational difficulties of densification and will provide a framework to better address the issues such as quality control. In addition, the fundamental ground bio-feedstock properties will provide a foundational step toward the rational basis for describing the mechanics of pellet formation. Accordingly, the goal of this research is to develop relationships between mechanical properties of ground biomass and resulting characteristics of pellet quality for selected biomass materials.

3.2 Hypothesis

The statistical null and alternate hypotheses are:

Ho: A direct and positive correlation between fundamental mechanical properties of ground biomass and pellet qualities does not exist.

Ha: A direct and positive correlation ($R^2 > 90\%$) between fundamental mechanical properties of ground biomass and pellet qualities does exist.

The hypothesis will be tested at significance level of $\alpha = 0.05$.

3.3 Objectives

Following specific objectives were formulated to achieve the goal and test the hypothesis:

1. to measure mechanical and physical properties of the bio-feedstocks in the ground form,
2. to measure mechanical properties and quality metrics of pellets made from the selected ground bio-feedstocks,
3. to develop predictive relationship(s) among the mechanical properties of the selected ground biomass and the properties of resulting pellets using statistical analysis, and
4. to validate the predictive relationship(s) using conditions other than those used to develop the relationships.
Chapter 4. Methodology

This chapter presents the methods of research to accomplish the proposed objectives. Materials used for this research are included in this chapter. This chapter also includes the preconditioning of material. Various tests conducted to determine the physical and mechanical properties of ground biomass are described. The densification process and pellet quality parameters are also presented. Finally, methods to develop predictive relationships between mechanical properties of ground biomass and pellet quality metrics are explained.

4.1 Overview and Flowchart of Methodology

The goal of the proposed research was to relate the fundamental mechanical properties of bio-feedstock to the mechanical properties of the pellet quality parameters. Figure 4.1 provides an overall flow chart of the methodology for this study. The methodology is described in five phases, which is given below.

**Phase I:** Phase I included the collection of three types of bio-feedstock representative of northeast region of the U.S.

**Phase II:** During phase II, grinding of raw material was performed by a size reduction machinery (Munson SCC-10, City, State), which is based on the shear cutting mechanism. The moisture content of ground biomass was measured by the oven drying method according to the ASABE S358.2 standard (ASABE, 2007).

**Phase III:** Measurement of physical and mechanical properties of ground biomass was completed with a Pycnometer using Helium medium (for measurement of particle density), cubical triaxial tester (compressibility properties such as bulk modulus, compression index, and rebound properties such as spring-back index). The particle size distribution of ground biomass was determined by a sieve shaker according to the ASABE S319.3 standard (ASABE, 2006b).
Figure 4.1 Flow chart of methodology to determine the correlation among mechanical properties of ground biomass and pellet qualities and to develop predictive relationships.
Phase IV: Pelletization task was performed with a lab-scale pellet mill by pre-conditioning of the ground biomass to certain moisture content (wet basis). This phase also dealt with the measurement of pellet quality such as durability, strength, and density. For the measurement of the pellet strength, a universal testing machine was utilized and durability was tested.

Phase V: In phase V, the focus was on the development of relationships among the properties obtained in phases III and IV, i.e. physical and mechanical properties of ground bio-feedstock and pellet quality.

4.2 Materials and Lab Description

The research was conducted using three types of biomass. The collections of three type of bio-feedstock were based on availability and are representative of northeast region of the U.S. All of the instruments used for this study are currently available in the Department of Agricultural and Biological Engineering.

4.2.1. Corn Stover

In the United States, more than three-fourth of the agricultural crop residue resource is corn stover (U.S.D.O.E., 2011). Corn stover consists of the residues of maize plants in a field after harvesting that includes stalk, leaves, and husk. For this study, corn stover was collected from the Pennsylvania State University Farm, which was harvested in October 2011.

4.2.2 Switchgrass

Switchgrass is a perennial grass native to the tallgrass prairies. Currently it attracts much attention as a model herbaceous energy crop for the U.S. (Sanderson et al., 2006). Attributes of switchgrass desirable for bioenergy cropping includes its demonstrated long-term (> 10 year) high productivity across many environments, suitability for marginal land, relatively low water and nutrient requirements, and positive environmental benefits (Sanderson et al. 2006). For this study, switchgrass was collected from a farm located at Julian, PA, which was harvested in November 2011.
4.2.3 Willow

Hardwood such as willow, oak, maple, beech, cherry and other species is common to the northeast U.S. Willow is a light weight and uniform grained wood, which grows rapidly and reaches maturity in 50 to 70 years (Cassens, 2007). Willow, a short rotation crop, has ability to resprout after multiple cuts, and has tolerance of dense planting (Abrahamson et al., 2012). According to Cassens (2007), its bending and breaking strengths are very low, but the shear strength and side hardness are comparable to the weaker intermediate weight woods. Willow wood chips were obtained in March 2013 from Cornell University, NYS Agricultural Experiment station, Geneva.

4.3 Experimental Design

For this research, three biomass types were collected to conduct experiments. Two important parameters, size reduction and moisture content, were considered for preconditioning of the materials, which are the basis of the design of experiment. Two screen sizes; 3.175 mm, and 6.35 mm, were used for size reductions task and thus two different sets of particle size distributions of material were obtained from ground material.

Ground biomass was conditioned by spraying water to achieve required moisture content on a wet basis. The water was mixed using manual Mini-Inversina (Bioengineering AG, Switzerland) capable of giving 360° motion in the mixer. Several trial runs were performed to determine the mixing duration and based on these runs, the mixing was performed at 80 rpm for two minutes. The conditioned material was kept for 24 hours for moisture equilibration. Based on review of literature and preliminary runs performed, two moisture content levels considered for conditioning of materials were 17.5% (w.b.) and 20.0% (w.b). For each sample set included in Table 4.1, pellets were formed and HTC test were conducted with the CTT. Number of replicates for each treatment is included in Table 4.2, which is based on past researches conducted and several preliminary runs.
Table 4.1 Design of experiment of selected biomass of different sizes and at different moisture content for pelletization, for conducting durability test, to determine strength, and for conducting HTC test on CTT

<table>
<thead>
<tr>
<th>#</th>
<th>Variables</th>
<th>Number of levels</th>
<th>Levels</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Material</td>
<td>3</td>
<td>1. Corn stover</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>2. Switchgrass</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>3. Willow</td>
</tr>
<tr>
<td>2.</td>
<td>Screen Size</td>
<td>2</td>
<td>a) 3.175 mm</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>b) 6.350 mm</td>
</tr>
<tr>
<td>3.</td>
<td>Moisture content</td>
<td>2</td>
<td>i) 17.5% w.b.</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>ii) 20.0% w.b.</td>
</tr>
</tbody>
</table>

Table 4.2 Experimental plan for replicates for each treatment

<table>
<thead>
<tr>
<th>Property/ test</th>
<th>Apparatus</th>
<th>Replicates</th>
</tr>
</thead>
<tbody>
<tr>
<td>Particle Density</td>
<td>Pycnometer</td>
<td>5*</td>
</tr>
<tr>
<td>Bulk Density</td>
<td>Container of known volume</td>
<td>5*</td>
</tr>
<tr>
<td>Particle Size Distribution</td>
<td>Ro-tap sieve shaker</td>
<td>5*</td>
</tr>
<tr>
<td>HTC test</td>
<td>CTT</td>
<td>5*</td>
</tr>
<tr>
<td>Durability</td>
<td>Durability tester</td>
<td>5*</td>
</tr>
<tr>
<td>Axial Compressive Strength</td>
<td>Universal Testing Machine</td>
<td>10**</td>
</tr>
<tr>
<td>Diametral Tensile Strength</td>
<td>Universal Testing Machine</td>
<td>10**</td>
</tr>
<tr>
<td>Pellet Density</td>
<td>-</td>
<td>20**</td>
</tr>
</tbody>
</table>

* Based on review of literature and preliminary runs performed

**Based on the review of literature, initially five runs were performed. Based on preliminary runs performed and statistical analysis to ensure that the data set is within the 95% confidence interval, 10 replicates for strength and 20 replicates for pellet density were performed

4.4 Size Reduction

Size reduction machine (Munson SCC-10, Utica, New York) which is based on shear cutting mechanism, was used for reducing the size of the as-received test material. Based on review of literature, two different screen sizes, 3.175 mm and 6.35 mm, were utilized for size reduction. The main purpose of this machinery is to coarsely grind the material into particles. The biomass is fed from the top of the machinery and collected from the chute at the bottom of the machinery.
Inlet

Outlet

Figure 4.2 Size Reduction Machine (Munson SCC-10)

4.5 Moisture Content

The moisture content of ground biomass, i.e. after grinding the biomass were measured by the oven drying method according to ASABE S358.2 standard (ASABE, 2006a). A sample of 25 g was oven dried for 24 hours at 103°C ± 2°C. The moisture content values were reported in percent wet basis. Four replications were used to determine moisture content of ground biomass to ensure that the data set is within the 95% confidence interval. The moisture content was calculated using following equation:

\[
\text{Moisture content} = \frac{\text{(weight before drying} - \text{weight after drying})}{\text{weight before drying}} \times 100 \text{ (% w.b.)} \quad \ldots (4.1)
\]
4.6 Physical Properties of Ground Biomass

Physical properties of ground biomass include the particle density, bulk density, particle size distribution. Methodologies for testing of these properties are described in detail in following sub-sections.

4.6.1 Particle Size Distribution

The particle size distributions of ground biomass were determined using a Ro-tap sieve shaker according to ASABE S319.3 standard (ASABE, 2006b). A sample of ground biomass of 100 g was used to determine the particle size distribution using Ro-tap sieve shaker. U.S. Standard Sieve numbers 5, 7, 10, 14, 18, 25, 35, 45, 60, 80, 120, 170, 230 (Sieve size: 4.0, 2.81, 2.0, 1.4, 1.0, 0.71, 0.5, 0.353, 0.25, 0.176, 0.125, 0.088, 0.062 mm, respectively) were used for this analysis.

The set of sieves were placed on Ro-tap sieve shaker and the sieving was performed for duration of 15 minutes. After sieving, the mass retained on each sieve was measured and analyzed to determine particle size distribution according to ASABE standards (2006b). For all three selected biomass reduced with two different screen sizes; 3.175 mm and 6.35 mm, five replications for each set were used for conducting this test.

4.6.2 Particle and Bulk Density

Measurement of particle density of ground biomass was performed with the multipycnometer (Quantachrome Instruments, MVP-2, Serial # 11496060701, Boynton Beach, Florida) using Helium as a pressure medium. Based on volume-pressure relationship (Boyle’s law), volume of sample chamber is calculated based on known volume of reference cell and obtained pressures in respective chambers. These values were then used to calculate the particle density of the material. Five replications were used for each treatment to measure the bulk density and particle density of ground biomass. Following equation was used to determine the value of volume of sample cylinder:

\[
V_p = V_c - V_r \left[ \frac{p_1}{p_2} - 1 \right] \quad \ldots (4.2)
\]

where \(V_p\) is volume of the ground material, \(V_c\) is volume of sample cell (manufacture supplied information; 149.284 cm\(^3\)), \(V_r\) is volume of reference cell (manufacture supplied information; 149.284 cm\(^3\)).
information; 90.56 cm³), P₁ is pressure reading after pressurizing the reference cell and P₂ denotes the pressure reading after including the sample cell in the loop.

Bulk density was determined by filling the container of known volume (135 cm³) with the material by spoon-by-spoon and weighed to within ± 0.01g to obtain the weight of the material. Mass per unit volume provided the value of bulk density.

4.7 Mechanical Properties of Ground Biomass

HTC test were performed using the low pressure CTT to determine the mechanical properties of ground biomass. Low pressure CTT was designed and developed by Kamath and Puri (1997). Change in the dimensions can be measured using linear motion potentiometers (LMPs). Real-time data of the pressure and the displacement experienced by the ground biomass sample were collected. The HTC test data were stored with a data acquisition software developed in-home with LabVIEW (National Instruments Corporation, version 8.2, Austin, Texas) and analyzed further to determine the parameters such as bulk modulus, compression index, and spring back index. Five replications of each treatment included in Table 4.1 were used to perform HTC tests, which followed stress path mentioned in Table 4.3.

**Table 4.3 Stress path for HTC Test**

<table>
<thead>
<tr>
<th>Stress Path (kPa)</th>
<th>Increment Rate</th>
</tr>
</thead>
<tbody>
<tr>
<td>0 – 20 – 0 – 45 – 0 – 70 – 0 – 95 – 0 – 95</td>
<td>0.5 kPa/5 sec</td>
</tr>
</tbody>
</table>

HTC tests were conducted at 0.5 kPa/5sec to minimize any time dependent effects with stress path of 0 – 20 – 0 – 45 – 0 – 70 – 0 – 95 – 0 – 95, i.e., first the pressure up to 20 kPa was applied. After reaching 20 kPa the pressure was reduced to 0kPa. In the second step the pressure was brought up to 45 kPa and again released to 0 kPa and so on. Five replications were performed for each test.

4.7.1 Determination of Bulk Modulus (K)

The bulk modulus was determined by using the HTC test results. To determine the bulk modulus at a given mean pressure a linear regression was performed on the entire unloading and reloading curve (Figure 4.3). Slope of the linear regression line
Fig 4.3: Example plot of isotropic pressure vs. volumetric strain for determining bulk modulus (K) (Pandeya and Puri, 2010).

4.7.2 Determination of Compression Index

Compression index (λ) was calculated from the HTC test data an example of which is plotted in Figure 4.4. The graph of ln (pressure) vs. void ratio was plotted. The slope of the virgin consolidation line gives the compression index (Desai and Siriwardane, 1984 and Pandeya and Puri, 2010).

4.7.3 Determination of Spring-back Index

Spring-back index (κ) was calculated from the HTC test data from the same graph as for the compaction index (Desai and Siriwardane, 1984 and Pandeya and Puri, 2010). The slope of the unloading reloading line gives the spring back index (see Figure 4.4).
Figure 4.4: Example plot of ln(pressure) vs. void ratio (Pandeya and Puri, 2010)

4.8 Pelletization

Pelletization of conditioned material was performed with the help of pellet mill (PelletPros, Model VI84TTFB4026AA, Dubuque, Iowa) as shown in Figure 4.5. All combinations for pelletization are included in Table 4.1. For these combinations, four batches of pellets were formed using 500 g of ground biomass. Those batches were used to conduct various quality tests for the pellets.

Figure 4.5 Lab-scale pellet mill
4.9 Pellet Quality

Strength of pellets, durability, and pellet density are the parameters to measure pellet quality. Strengths of pellets were measured by conducting tests on a universal testing machine (Instron model 3345, Norwood, MA). Durability test was performed using a friability tester.

4.9.1 Strength

The various quality parameters such as axial compressive strength and diametral tensile strength were determined by using a universal testing machine (Instron model 3345, Norwood, MA) (Figure 4.6) at a compressive speed of 0.5 mm/min to maintain quasi-static state. Ten replications each on randomly selected pellets were used to measure the diametral tensile and axial compressive strengths.

Figure 4.6 Universal testing machine (Instron model 3345, Norwood, MA)
4.9.1.1 Diametral Tensile Strength

Figure 4.7 shows the schematic of sample testing to determine diametral tensile strength in universal testing machine. The diametral tensile strength (ASTM, 2008), \( \sigma \) were calculated according to ASTM standard D3967 as

\[
\sigma = \frac{2P}{\pi LD}
\]

… (4.3)

where \( D \) and \( L \) are diameter and length respectively of the pellet, and \( P \) is force applied.

![Schematic of pellet positioning for diametral tensile strength testing and actual positioning on universal testing machine](image)

4.9.1.2 Axial Compressive Strength

Figure 4.8 shows the schematic of sample testing to determine axial compressive strength in universal testing machine, which were calculated as

\[
\sigma = \frac{4P}{\pi D^2}
\]

… (4.4)

where \( D \) is diameter of the pellet and \( P \) is force applied.

![Schematic of pellet positioning for axial compressive strength testing and actual positioning on universal testing machine](image)
4.9.2 Durability

This test was conducted using a friabilator, which was fabricated in the Engineering Machine Shop of Department of Agricultural and Biological Engineering (Pandey, 2009). For this test, known weight of pellets was placed in the friabilator. For determination of durability of pellets, a 50 g sample of pellets was tumbled at 55 rpm for 10 minutes in a dust-tight enclosure. A sieve No. 5 with an aperture of 4.0 mm was used to measure the weight retained during tumbling process. Durability is expressed as the percent ratio of the mass of pellets retained on the sieve after tumbling to the mass of pellets before tumbling. Five replications of treatments included in Table 4.1 were used to conduct durability test.

4.9.3 Pellet Density

Pellet density was calculated by measuring the pellet weight and calculating the volume (ASABE, 2007). For volume calculation, the pellet shape was assumed to be cylindrical; its volume was calculated using the measurement of its length and diameter. Initial measurements confirmed that the diameter along the length varied by less than 5%, i.e., a single measurement of diameter is sufficient. Twenty replications of randomly selected pellets from each set were used to measure the individual pellet density.

4.10 Data Analysis

Minitab software (Version 16.1.0.0, State College, PA) was used to perform ANOVA and regression analysis on obtained data, to determine if the relationships are significant or not and for the development of predictive relationships. The hypothesis was tested at 95% confidence level using Minitab software.

4.11 Development and Validation of Relationships and Testing of Hypotheses

The physical properties of the pellets were determined by the methods described in section 4.9. Pellet quality properties included diametral strength, axial strength, and durability. Mechanical properties included bulk modulus, compression index, and spring-back index. Linear regressions were performed between mechanical properties of ground biomass and pellet quality using Minitab (Version 16.1.0.0, State College, PA) for
selected materials. Based on the results, the correlation ($R^2 > 90\%$) between the mechanical properties of the ground biomass and pellet qualities were established.
Chapter 5. Results and Discussion

The physical and mechanical properties of all three ground biomass preconditioned test materials and quality properties of their pellets are presented in this chapter. In addition, the comparisons of three different test materials on the basis of different properties are discussed. Furthermore, the relationships among the mechanical and physical properties of each ground material with the quality of pellets are developed and validated in the subsequent sections.

5.1 Corn Stover

Corn stover from the field was ground with the help of size reduction machinery. Two screen sizes, i.e., 3.175 mm and 6.35 mm, were selected on the basis of previous studies conducted in the field of pelletization of biomass. The moisture content of the ground corn stover was 4.2% w.b. The ground corn stover was conditioned at two different moisture contents, i.e., 17.5% and 20% (w.b.), based on literature review and preliminary runs as described in section 4 (Table 4.1).

5.1.1 Physical Properties of Ground Corn Stover

The values of D$_{50}$, particle density, and bulk density of ground corn stover reduced with screen sizes 3.175 mm and 6.35 mm are presented in Table 5.1. D$_{50}$ diameter was calculated using the values of particle size distribution, which was performed using the Ro-top sieve shaker. D$_{50}$ is the value of particle diameter at 50% in the cumulative mass distribution. It represents the median value of the particle size distribution.

<table>
<thead>
<tr>
<th>Screen size</th>
<th>D$_{50}$* (mm) (N = 5)</th>
<th>Particle density** (kg/m$^3$) (N = 5)</th>
<th>Bulk density* (kg/m$^3$) (N = 5)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3.175 mm</td>
<td>0.70 ± 0.04</td>
<td>1368.93 ± 22.2</td>
<td>102.8 ± 0.98</td>
</tr>
<tr>
<td>6.35 mm</td>
<td>1.00 ± 0.04</td>
<td>1355.14 ± 50.6</td>
<td>92.0 ± 2.15</td>
</tr>
</tbody>
</table>

* Significantly different between two screen sizes (p<0.05),
** Not significantly different (p>0.05), where N is number of samples
Particle density and bulk density of conditioned ground corn stover for two conditions is listed in Table 5.2. Figure 5.1 shows the particle size distribution of ground corn stover for two screen sizes. The $D_{50}$ of corn stover reduced with 6.35 mm screen size (1.0 mm) was higher by 30.0% than reduced with 3.175 mm screen size (0.7 mm).

The screen size has a significant effect on: (1) the particle size distribution ($p<0.05$), (2) $D_{50}$, and (3) bulk density ($p<0.05$), but not on the particle density ($p>0.05$). Since the same material is size reduced with different screen sizes, it does not change the particle density significantly. However, the size reduction affects bulk density, which is dependent on the occupied volume by the ground material, size spread, and inter-particle spaces. Since the smaller screen size has larger spread of finer particles than larger screen (Figure 5.1), it aids in packing the material better by filling the voids between larger particles by finer particles. This results in increased bulk density of the material.

### Table 5.2 Physical properties of conditioned ground corn stover

<table>
<thead>
<tr>
<th>Condition</th>
<th>Bulk density* (kg/m$^3$) (N = 5)</th>
<th>Particle Density** (kg/m$^3$) (N = 5)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Screen size (mm)</td>
<td>Moisture content (% w.b.)</td>
<td></td>
</tr>
<tr>
<td>3.175</td>
<td>20</td>
<td>93.2 ± 0.9</td>
</tr>
<tr>
<td>6.35</td>
<td>17.5</td>
<td>88.0 ± 0.9</td>
</tr>
</tbody>
</table>

* Significantly different between two screen sizes ($p<0.05$),  
** Not significantly different ($p>0.05$)

Figure 5.1 Particle size distribution of ground corn stover for two screen sizes with error bars (Mean ± Standard deviation) (N=5)
5.1.2 HTC Test Results for Ground Corn Stover

HTC test results for ground corn stover are presented in the following subsections. The initial bulk density of ground material was calculated from the mass of material that was present in the sample chamber. Particle density of conditioned corn stover was measured with the help of a pycnometer.

5.1.2.1 Bulk Modulus

A typical data collected for ground corn stover illustrating the ground biomass’s response to volumetric deformation is given in Figure 5.2. The ground biomass was subjected to stress path mentioned in Table 4.3 at compression rate of 0.5 kPa/5 sec. The values of bulk modulus were calculated as explained in section 4.7.1. For determination of bulk modulus, the plot was made with volumetric strain on x-axis and pressure on y-axis. The ground biomass was subjected to stress path mentioned in Table 4.3 at compression rate of 0.5 kPa/5 sec. The sample was filled at 0 kPa and the test was started at the compression rate of 0.5 kPa/5 sec. After reaching 5 kPa in the beginning of the test, the base pressure was changed to 5kPa for rest of the test. The loops formed in Figure 5.2 are the ground material’s response to unloading and reloading of pressure. For example, for second loop at 45 kPa, after reaching pressure at 45 kPa at A, the pressure reduced (i.e. unloading path) from A to B, i.e., to base pressure and then reloaded from B to C till it reaches 70 kPa. The stress path in the same manner was followed to generate additional loading-unloading loops as shown in Figure 5.2.

Figure 5.3 shows the bulk modulus of ground corn stover (screen size 6.35 mm) conditioned at 17.5% w.b. moisture content and (screen size 3.175 mm) conditioned at 20% w.b. moisture content at specific unloading pressures. The obtained values of bulk modulus at two different conditions were significantly different at p<0.05 at 20 kPa, 45 kPa, 70 kPa, and 95 kPa. For smaller screen size, there is an increasing trend in bulk modulus, whereas, in case of the larger screen size, the value of bulk modulus initially decreased slightly and then followed the same trend as the smaller screen size. The reason for the slight initial decrease bulk modulus value can be attributed to the greater rearrangements of the particles at initial stages.
Figure 5.2 A typical isotropic stress (pressure) vs. volumetric strain response with multiple loading-unloading loops for ground corn stover (screen size 6.35 mm) conditioned at 17.5% w.b. moisture content.

Figure 5.3 Bulk modulus of ground corn stover (screen size 6.35 mm) conditioned at 17.5% w.b. moisture content and (screen size 3.175 mm) conditioned at 20% w.b. moisture content.
5.1.2.2 Compression Index

Compression index is an elastoplastic parameter to measure the particulate material’s compressibility at a particular isotropic pressure (Mittal, 2003). It is an important parameter that can be used to formulate the modified Cam-clay model (Desai and Siriwardane, 1984). Figure 5.4 shows the void ratio vs ln(pressure) for ground corn stover (screen size 6.35 mm) conditioned at 17.5% w.b. moisture content and (screen size 3.175 mm) conditioned at 20% w.b. moisture content at specific unloading pressures. The values of compression index were calculated based on the method summarized in section 4.7.2.

With the increase in pressure and due to particles rearrangements, voids ratio decreases resulting in increased compression index. This trend can be seen in Figure 5.5 and similar trends were observed by different researchers using different materials such as pharmaceutical powder formulations and formulations widely used in ceramic industries (Mittal and Puri, 1999; Li 1999 and Mittal, 2003). The values of compression index of two different conditions i.e., 3.175 mm screen size conditioned at 20% moisture content and 6.35 mm screen size at 17.5 % moisture content w.b., are significantly different (p<0.05) for 20 kPa, 45 kPa, and 70 kPa.

Figure 5.4 A typical void ratio vs ln (Pressure) plot with multiple loading-unloading loops for ground corn stover (screen size 6.35 mm) conditioned at 17.5% w.b. moisture content
Figure 5.5 Compression index of ground corn stover (screen size 6.35 mm) conditioned at 17.5% w.b. moisture content and (screen size 3.175 mm) conditioned at 20% w.b. moisture content

Figure 5.6 Spring-back index of ground corn stover (screen size 6.35 mm) conditioned at 17.5% w.b. moisture content and (screen size 3.175 mm) conditioned at 20% w.b. moisture content
5.1.2.3 Spring-back Index

Spring-back index is an elastoplastic parameter, which represents the ability of bulk particulate materials to elastically recover (i.e. swell) after the applied pressure has been released. The values of spring-back index were calculated based on the method discussed in the section 4.7.3. From Figure 5.6, it can be observed that the spring-back index decreases up to a certain pressure value, but thereafter, it shows an increase at higher pressure. The spring-back index values obtained for two conditions were not significantly different (p>0.05) at 20 kPa, 45 kPa, 70 kPa, and 95 kPa.

5.1.3 Properties of Corn Stover Pellets

Conditioned material of 3.175 mm screen size at 20% and 6.35 mm screen size at 17.5% formed better pellets than other combinations tested, i.e. 3.175 mm screen size at 17.5% (w.b.) and 6.35 mm at 20% (w.b.). The length of pellets formed with 6.35 mm screen size reduced particles at 17.5% w.b. was 15.94 ± 3.55 mm and for 3.175 mm at 20% w.b. was 15.87 ± 3.05 mm. The diameter for 6.35 mm at 17.5% was 5.97 ± 0.04 mm and for 3.175 mm at 17.5% was 5.92 ± 0.05 mm. The diameter of pellets, though slightly different for the two different screen sizes, are statistically significantly different (p<0.05) however, the lengths of pellets does not show any significant difference for two screen sizes (p>0.05). In the process of pelletization, the pellets were extruded through the horizontal die and the formed pellets came out of the chute of the pelletizer (Figure 4.5). Since the flow of pellets from the pelletizer is only dependent on the gravitational force, there was no control for governing the length of the pellets.

Table 5.3 lists the strengths, pellet density, and durability values of corn stover pellets for two different conditions. They are discussed in subsequent sub-sections.

Table 5.3 Quality metrics of corn stover pellets

<table>
<thead>
<tr>
<th>Condition of Pellets</th>
<th>Diametral Tensile Strength** (MPa) (N = 10)</th>
<th>Axial Compressive Strength** (MPa) (N = 10)</th>
<th>Pellet Density** (kg/m³) (N = 20)</th>
<th>Durability** (%) (N = 5)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Screen size (mm)</td>
<td>Moisture content (% w.b.)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>3.175</td>
<td>20</td>
<td>5.2 ± 0.6</td>
<td>10.1 ± 1.4</td>
<td>1317.4 ± 47.8</td>
</tr>
<tr>
<td>6.35</td>
<td>17.5</td>
<td>4.9 ± 1.0</td>
<td>9.5 ± 1.5</td>
<td>1325.7± 39.3</td>
</tr>
</tbody>
</table>

** Significantly not different (p>0.05)
5.1.3.1 Pellet Density

The mean density of corn stover pellets (Table 5.3) was 1,317.4 kg/m³ for screen size 3.175 mm and 1,325.7 kg/m³ for screen size 6.35 mm. The larger screen size had pellet density that was higher by a minimal amount of 0.6%, which were not significantly different (p>0.05). Comparing the pellet with bulk densities for the two screen sizes given in Table 5.1, the pelletization process compressed the loose material nearly 12.8 (screen size 3.175 mm) and 14.4 (screen size 6.35 mm) folds. In fact, the relative densities attained by pellets were 96.2% and 97.8% for screen sizes 3.175 mm and 6.35 mm, respectively. This shows that the pelletizer was very effective in forming pellets considering the density metric.

5.1.3.2 Diametral Tensile Strength

Diametral tensile strength of pellets can be observed from the Table 5.3, which were 5.2 ± 0.6 MPa (screen size 3.175 mm) and 4.9 ± 1.0 MPa (screen size 6.35 mm). Pellets formed with the smaller screen size, 3.175 mm, at 20% (w.b.) moisture content had 5.4% higher diametral tensile strength than the 6.35 mm at 17.5% (w.b.), however, they were not significantly different (p>0.05). In general, smaller screen size results in finer ground material, which results in better coordination number and, therefore, higher strength (Feda, 1982).

5.1.3.3 Axial Compressive Strength

Axial compressive strength of pellets can be observed from the Table 5.3, which were 10.1 ± 1.4 MPa (screen size 3.175 mm) and 9.5 ± 1.5 MPa (screen size 6.35 mm). These values were not significant different at p< 0.05. Pellets formed with the smaller screen size, 3.175 mm, at 20% (w.b.) moisture content had 5.6% higher axial compressive strength than the 6.35 mm at 17.5% (w.b.) for reasons discussed in the preceding sections.

5.1.3.4 Durability

Table 5.3 includes the results for durability of corn stover pellets for two different conditions, i.e., 3.175 mm screen size at 20% w.b. moisture content and 6.35 mm screen
size at 17.5% w.b. moisture content. These values were not significant different at \( p < 0.05 \) for different pellet conditions. Since the range of variation of pellet durability for corn stover is very narrow from 96 to 98%, developing predictive relationships was not very meaningful. Therefore, no predictive relationships between durability and mechanical properties from the HTC tests were developed.

5.1.4 Relationship among Physical and Mechanical Properties of Ground Corn Stover and Pellet Qualities

The mechanical properties of ground corn stover such as bulk modulus and compression index, were significantly different for two conditions \( (p<0.05) \) at 45 kPa, 70 kPa, and 95 kPa pressure, however, the pellet quality properties such as diametral tensile strength, axial compressive strength, and pellet density does not show any significant difference \( (p>0.05) \). \( D_{50} \) and bulk density of ground corn stover were significantly different \( (p<0.05) \).

5.2 Switchgrass

Switchgrass from the field was ground with the help of a size reduction machinery. Two screen sizes, i.e., 3.175 mm and 6.35 mm, were selected on the basis of previous studies conducted in the field of pelletization of biomass. The moisture content of the ground switchgrass was 3.8% wet basis (w.b.). The ground switchgrass was conditioned at two different moisture contents, i.e., 17.5% and 20% (w.b.), based on literature review and preliminary runs. Four combinations were tested for this material.

5.2.1 Physical Properties of Ground Switchgrass

The values of \( D_{50} \), particle density, and bulk density of ground switchgrass reduced with screen size 3.175 mm and 6.35 mm are presented in Table 5.4. A Ro-tap sieve shaker was used to determine the particle size distribution. The \( D_{50} \) of particle size distribution was determined, which is the value of particle diameter at 50% in the cumulative mass distribution and represents the median value of the particle size distribution.
Table 5.4 Physical properties of preconditioned ground switchgrass

<table>
<thead>
<tr>
<th>Screen size</th>
<th>( D_{50} ) (mm)*</th>
<th>Particle density (kg/m(^3))*</th>
<th>Bulk density (kg/m(^3))*</th>
</tr>
</thead>
<tbody>
<tr>
<td>3.175 mm</td>
<td>0.61 ± 0.01</td>
<td>1298.6 ± 31.9</td>
<td>141.2 ± 1.3</td>
</tr>
<tr>
<td>6.35 mm</td>
<td>0.97 ± 0.07</td>
<td>1283.3 ± 19.7</td>
<td>114.9 ± 5.7</td>
</tr>
</tbody>
</table>

* Significantly different between two screen sizes (p<0.05),
** Not significantly different (p>0.05)

Particle density and bulk density of ground switchgrass reduced with 3.175 mm screen is higher than as compared to 6.35 mm screen size. The screen size has a significant effect on: (1) the particle size distribution (p<0.05), (2) \( D_{50} \), and (3) bulk density (p<0.05), but not on the particle density (p>0.05).

Figure 5.7 shows the particle size distribution of ground switchgrass from two screen sizes. \( D_{50} \) of switchgrass reduced with 6.35 mm screen size (0.97 mm) was higher by 37.1% than reduced with 3.175 mm screen (0.61 mm). Since the smaller screen size has larger spread of finer particles than the larger screen, it aids in packing the material better by filling the voids between larger particles by finer particles and results in increased bulk density of the material.

![Figure 5.7 Particle size distribution of ground switchgrass (screen size: 3.175 mm and 6.35 mm) (N = 5)](image-url)
Particle density and bulk density of conditioned ground switchgrass reduced with 3.175 mm screen size and 6.35 mm screen size are listed in Table 5.5. The screen size significantly affects bulk density (p<0.05), whereas, it does not affect the particle density significantly (p>0.05). Moisture content does not significantly effects (p>0.05) particle density and bulk density for the respective screen sizes.

Table 5.5 Physical properties of conditioned ground switchgrass

<table>
<thead>
<tr>
<th>Condition</th>
<th>Screen size (mm)</th>
<th>Moisture content (% w.b.)</th>
<th>Particle Density** (kg/m$^3$) (N = 5)</th>
<th>Bulk Density* (kg/m$^3$) (N = 5)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>3.175</td>
<td>17.5</td>
<td>1310.4 ± 25.5</td>
<td>134.4 ± 4.4</td>
</tr>
<tr>
<td></td>
<td>6.35</td>
<td>17.5</td>
<td>1297.8 ± 36.6</td>
<td>123.8 ± 3.9</td>
</tr>
<tr>
<td></td>
<td>3.175</td>
<td>20</td>
<td>1316.7 ± 29.8</td>
<td>132.5 ± 3.2</td>
</tr>
<tr>
<td></td>
<td>6.35</td>
<td>20</td>
<td>1305.6 ± 43.3</td>
<td>121.5 ± 2.8</td>
</tr>
</tbody>
</table>

* Significantly different (p<0.05),
** Not significantly different (p>0.05)

5.2.2 HTC Test Results for Ground Switchgrass

HTC test results for ground switchgrass are presented in the following sub-sections. The initial bulk density of ground material was calculated from the mass of material in the sample chamber.

5.2.2.1 Bulk Modulus

A typical data collected for ground switchgrass illustrating the ground biomass response to volumetric deformation is given in Figure 5.8. The trend of pressure vs. volume strain is similar to that of corn stover, however, the magnitudes are different. The values of bulk modulus were calculated as explained in the section 4.7.

Figure 5.9 shows the bulk modulus ground switchgrass of four different conditions at specific unloading pressures. For all four different conditions, there is an increasing trend in the bulk modulus. Similar trends have been reported by other researchers using different materials (Mittal and Puri, 1999; Li 1999; Mittal, 2003, and Pandeya, 2009) and for corn stover in section 5.1.2.1. As bulk modulus is a measure of resistance to compressibility, higher bulk modulus would be a result of less recovery of
Figure 5.8 A typical isotropic stress (pressure) vs. volumetric strain response with multiple loading-unloading loops for ground switchgrass (screen size 3.175 mm) conditioned at 17.5% w.b. moisture content.

Figure 5.9 Bulk modulus of ground switchgrass for four different conditions at four unloading pressures (N=5)
material upon releasing the pressure. On increasing the pressure, the material had less recovery, which results in the increase of bulk modulus.

The bulk modulus at different unloading pressures, i.e., 20 kPa, 45 kPa, 70 kPa, and 95 kPa, are significantly different (p<0.05) for 3.175 screen size, whereas, for screen size 6.35 mm, the bulk modulus at 20 kPa and 45 kPa unloading pressures are significantly different (p<0.05). The bulk modulus at 20% w.b. are not significantly different (p>0.05) for two screen sizes at different unloading pressures, i.e., 20 kPa, 45 kPa, 70 kPa, and 95 kPa, whereas at 17.5% w.b., the values of bulk modulus at 45 kPa and 70 kPa unloading pressures are significantly different (p<0.05).

5.2.2.2 Compression Index

Compression index is an elastoplastic parameter to measure the particulate material’s compressibility at a particular isotropic pressure (Mittal, 2003). It is an important parameter that can be used to formulate the modified cam-clay model. Figure 5.10 shows the void ratio vs. ln(pressure) for ground switchgrass (screen size 3.175 mm) conditioned at 17.5% w.b. moisture content, which is used to calculate the compression index and spring-back index (section 5.2.2.3).

![Figure 5.10 A typical void ratio vs ln (Pressure) plot with multiple loading-unloading loops for ground switchgrass (screen size 3.175 mm) conditioned at 17.5% w.b. moisture content](image)
With the increase in pressure and due to particles’ rearrangements, voids ratio decreases resulting in increased compression index. This trend can be seen in Figure 5.11 and similar trends were observed by different researchers for other materials (Mittal and Puri, 1999b; Li 1999 and Mittal, 2003). All the values of compression index at different unloading pressures, i.e., 20 kPa, 45 kPa, 70 kPa, and 95 kPa, are significantly different (p<0.05) among replicates of same conditions of screen size and moisture content. At 20 kPa, screen size has significant effect on compression index (p<0.05).

![Figure 5.11 Compression index of ground switchgrass at four different conditions at four unloading pressures (N = 5)](image)

**5.2.2.3 Spring-back Index**

Spring-back index is an elastoplastic parameter, which represents the ability of bulk particulate material to swell (i.e., recover) after the applied pressure has been released. From Figure 5.12, it is observed that the spring-back index decreases up to a certain pressure value but thereafter, it shows an increase at higher pressure. With the increase in pressure, the recovery of material decreases as it gets stiffer (more plastic) and hence the spring back index decreases.

All the values of compression index at different unloading pressures, i.e., 20 kPa, 45 kPa, 70 kPa, and 95 kPa, are significantly different (p<0.05) among replicates of same
conditions of screen size and moisture content. For screen size 3.175 mm, moisture content has significant effect (p<0.05) on spring-back index, whereas, no significant effect (p>0.05) was observed in the case of larger screen size.

![Figure 5.12 Spring-back index of ground switchgrass at four different conditions](image)

### 5.2.3 Properties of Switchgrass Pellets

Table 5.6 includes the length and diameter of the switchgrass pellets formed at four different conditions. Both diameter and length of pellets are not significantly different (p>0.05) among different conditions.

<table>
<thead>
<tr>
<th>Condition of Pellets</th>
<th>Length** (mm) (N = 20)</th>
<th>Diameter** (mm) (N = 20)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Screen size (mm)</td>
<td>Moisture content (% w.b.)</td>
<td></td>
</tr>
<tr>
<td>3.175</td>
<td>17.5</td>
<td>8.51 ± 1.7</td>
</tr>
<tr>
<td>6.35</td>
<td>17.5</td>
<td>9.08 ± 1.63</td>
</tr>
<tr>
<td>3.175</td>
<td>20</td>
<td>8.78 ± 1.56</td>
</tr>
<tr>
<td>6.35</td>
<td>20</td>
<td>9.37 ± 2.38</td>
</tr>
</tbody>
</table>

** Not significantly different (p>0.05)
Diametral tensile strength, axial compressive strength, pellet density and durability of switchgrass pellets formed at four different conditions are listed in Table 5.7.

Table 5.7 Quality metrics of switchgrass pellets

<table>
<thead>
<tr>
<th>Condition of Pellets</th>
<th>Diametral Tensile Strength ** (MPa) (N = 10)</th>
<th>Axial Compressive Strength ** (MPa) (N = 10)</th>
<th>Pellet Density** (kg/m$^3$) (N = 20)</th>
<th>Durability** (N = 5)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Screen size (mm)</td>
<td>Moisture content (% w.b.)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>3.175</td>
<td>17.5</td>
<td>2.6 ± 1.2</td>
<td>6.0 ± 2.4</td>
<td>1152.8 ± 85.7</td>
</tr>
<tr>
<td>6.35</td>
<td>17.5</td>
<td>1.8 ± 0.6</td>
<td>4.1 ± 1.5</td>
<td>1021.2 ± 89.0</td>
</tr>
<tr>
<td>3.175</td>
<td>20</td>
<td>2.1 ± 0.4</td>
<td>4.5 ± 1.4</td>
<td>1069.0 ± 80.2</td>
</tr>
<tr>
<td>6.35</td>
<td>20</td>
<td>1.8 ± 0.9</td>
<td>2.9 ± 1.7</td>
<td>886.2 ± 167.0</td>
</tr>
</tbody>
</table>

**Not significantly different (p>0.05)

5.2.3.1 Pellet Density

The density of switchgrass pellets can be observed from Table 5.7. Pellets formed using screen size of 6.35 mm at 17.5% w.b. has the highest pellet density as compared to the other conditions. According to Shaw and Tabil (2007), pellet density increases with the decrease in screen size, which can also be observed from the Table 5.7. Mani et al. (2006) also mentioned the influence of physical properties of material on the pellet density. On comparing the pellet densities for these conditions with the bulk densities of ground switchgrass given in Table 5.5, the pelletization process compressed the loose material nearly 8.6 (screen size 3.175 mm at 17.5% w.b.), 8.2 (screen size 3.175 mm at 20% w.b.), 8.1 (screen size 6.35 mm at 17.5% w.b.), and 7.3 (screen size 6.35 mm at 20% w.b.) folds. The relative densities attained by pellets were 88.0% (screen size 3.175 mm at 17.5% w.b.), 78.7% (screen size 3.175 mm at 20% w.b.), 81.2% (screen size 6.35 mm at 17.5% w.b.), and 67.9% (screen size 6.35 mm at 20% w.b.) of the particle density.

5.2.3.2 Diametral Tensile Strength

Diametral tensile strength of pellets can be observed from Table 5.7. Pellets formed with the smaller screen size, 3.175 mm, at 17.5% (w.b.) moisture content has highest diametral tensile strength (2.6 ± 1.2 MPa) among all four conditions as shown in Figure 5.13. The values of diametral strength are not significantly different (p>0.05).
5.2.3.3 Axial Compressive Strength

Axial compressive strength of pellets can be observed from the Table 5.7. Pellets formed with the smaller screen size, 3.175 mm, at 17.5% (w.b.) moisture content has highest axial compressive strength (6.0 ± 2.4 MPa) among all four conditions as seen in Figure 5.14. The values of axial compressive strength are not significantly different (p>0.05) among different conditions.
5.2.3.4 Durability

Table 5.7 includes the results for durability of switchgrass pellets for four different conditions. In Figure 5.15 it can be observed that for all four conditions, durability is more than 80%, hence it can be said that pellets formed at these conditions were highly durable. The values obtained are not significantly different (p>0.05) among the different conditions. However, the range of variation of pellet durability is very narrow from 88% to 91%. Therefore, no predictive relationships between durability and mechanical properties from the HTC tests were developed.

![Figure 5.15 Durability of ground switchgrass at different conditions](image)

5.2.4 Relationship among Physical and Mechanical Properties of Ground Switchgrass and Pellet Qualities

Table 5.8 includes the correlation coefficients of strengths of pellets and mechanical properties of ground switchgrass. It can be observed from the table that values for mechanical properties above 20 kPa are highly correlated, i.e. $R^2>80\%$, with the strength of pellets. The highest correlation coefficient for each property is bolded and highlighted in the Table 5.8. Respective values will be used for developing the predictive relationships among mechanical properties of ground material and pellets in section 5.4.
Table 5.8 Correlation coefficients for strengths of pellets and mechanical properties of ground switchgrass

<table>
<thead>
<tr>
<th></th>
<th>Axial Compressive Strength</th>
<th>Diametral Tensile Strength</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bulk Modulus - 20 kPa</td>
<td>0.606</td>
<td>0.435</td>
</tr>
<tr>
<td>Bulk Modulus - 45 kPa</td>
<td>0.898</td>
<td>0.879</td>
</tr>
<tr>
<td>Bulk Modulus - 70 kPa</td>
<td>0.911</td>
<td>0.928</td>
</tr>
<tr>
<td>Bulk modulus - 95 kPa</td>
<td>0.782</td>
<td>0.808</td>
</tr>
<tr>
<td>Compression Index - 20 kPa</td>
<td>0.860</td>
<td>0.806</td>
</tr>
<tr>
<td>Compression Index - 45 kPa</td>
<td><strong>0.949</strong></td>
<td>0.938</td>
</tr>
<tr>
<td>Compression Index - 70 kPa</td>
<td>0.924</td>
<td><strong>0.997</strong></td>
</tr>
<tr>
<td>Compression Index - 95 kPa</td>
<td>0.882</td>
<td>0.950</td>
</tr>
<tr>
<td>Spring-back Index - 20 kPa</td>
<td>0.938</td>
<td>0.941</td>
</tr>
<tr>
<td>Spring-back Index - 45 kPa</td>
<td>0.938</td>
<td>0.980</td>
</tr>
<tr>
<td>Spring-back Index - 70 kPa</td>
<td><strong>0.971</strong></td>
<td><strong>0.988</strong></td>
</tr>
</tbody>
</table>

Highlighted and bolded values – highest correlation coefficient for respective properties among four different unloading pressures

5.3 Willow

The willow from the field was ground with size reduction machinery. Two screen sizes, i.e., 3.175 mm and 6.35 mm, were selected on the basis of previous studies conducted in the field of pelletization of biomass. The moisture content of the ground willow was 5.5% wet basis (w.b.). The ground willow was conditioned at two different moisture contents, i.e., 17.5% and 20% (w.b.), based on literature review and preliminary runs as explained in Table 4.1.

5.3.1 Physical Properties of Ground Willow

The values of D_{50}, particle density, and bulk density of ground willow reduced with screen size 3.175 mm and 6.35 mm are presented in Table 5.9. A ro-tap sieve shaker was used to determine the particle size distribution. The D_{50} of particle size distribution was determined, which is the value of particle diameter at 50% in the cumulative mass distribution and represents the median value of the particle size distribution. Particle density and bulk density of ground willow reduced with 3.175 mm screen is higher than as compared to 6.35 mm screen size (Table 5.9).
Table 5.9 Physical properties of preconditioned ground willow

<table>
<thead>
<tr>
<th>S.N.</th>
<th>Screen size (mm)</th>
<th>$D_{50}$ (N = 5)</th>
<th>Particle density (kg/m$^3$) (N = 5)</th>
<th>Bulk density (kg/m$^3$) (N = 5)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>3.175 mm</td>
<td>0.94 ± 0.04</td>
<td>1335.0 ± 7.8</td>
<td>209.5 ± 0.5</td>
</tr>
<tr>
<td>2.</td>
<td>6.35 mm</td>
<td>1.26 ± 0.09</td>
<td>1268.7 ± 7.9</td>
<td>173.7 ± 2.8</td>
</tr>
</tbody>
</table>

* Significantly different (p<0.05)

The screen size has significant effect (p<0.05) on the particle size distribution, bulk density, and particle density. High porosity of the biomass material might affect the particle density. Particle density and bulk density of conditioned ground willow is presented in Table 5.10.

Table 5.10 Physical properties of conditioned ground willow

<table>
<thead>
<tr>
<th>Condition</th>
<th>Screen size (mm)</th>
<th>Moisture content (% w.b.)</th>
<th>Particle Density** (kg/m$^3$) (N = 5)</th>
<th>Bulk Density* (kg/m$^3$) (N = 5)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>3.175</td>
<td>17.5</td>
<td>1248.0 ± 19.7</td>
<td>187.8 ± 0.7</td>
</tr>
<tr>
<td></td>
<td>6.35</td>
<td>17.5</td>
<td>1212.7 ± 20.9</td>
<td>163.2 ± 1.9</td>
</tr>
<tr>
<td></td>
<td>3.175</td>
<td>20</td>
<td>1200.7 ± 15.1</td>
<td>191.2 ± 0.4</td>
</tr>
<tr>
<td></td>
<td>6.35</td>
<td>20</td>
<td>1180.6 ± 33.3</td>
<td>170.5 ± 2.1</td>
</tr>
</tbody>
</table>

*Significantly different (p<0.05)

**Not significantly different (p>0.05)

Figure 5.16 shows the particle size distribution of ground willow of two screen sizes. $D_{50}$ of willow reduced with 6.35 mm screen size (1.26 mm) was higher by 33.7% than reduced with 3.175 mm screen (0.94 mm). Since the smaller screen size has larger spread of finer particles than the larger screen, it aids in packing the material better by filling the voids between larger particles by finer particles and results in increased bulk density of the material.
Figure 5.16 Particle size distribution of ground willow (screen size: 3.175 mm and 6.35 mm) (N = 5)

5.3.2 HTC Test Results for Ground Willow

HTC test results for ground willow are presented in the following sub-sections. The initial bulk density of ground material was calculated from the mass of material in the sample chamber.

5.3.2.1 Bulk Modulus

A typical data collected for ground willow illustrating the ground biomass’s response to volumetric deformation is given in Figure 5.17. For determination of bulk modulus, plot was made with volumetric strain on x-axis and pressure on y-axis and values were calculated as explained in Chapter 4.
Figure 5.17 A typical isotropic stress (Pressure) vs. volumetric strain response with multiple loading-unloading loops for ground willow (screen size 3.175 mm) conditioned at 17.5% w.b. moisture content

Figure 5.18 shows the bulk modulus of ground willow for four different conditions at specific unloading pressures (20, 45, 70, and 95 kPa). For all four different conditions, there is an increasing trend in the bulk modulus. Similar trends have been reported by other researchers using different materials (Mittal and Puri, 1999b; Li 1999; Mittal, 2003, and Pandeya, 2009) and for corn stover and switchgrass in sections 5.1.2.1 and 5.2.2.1, respectively. As bulk modulus is a measure of the resistance to compressibility, higher bulk modulus would be a result of less recovery of material upon releasing the pressure. In Figure 5.18, it can be observed that on increasing the pressure, the material had less recovery, which resulted in the increase of bulk modulus. The moisture content had significant effect (p<0.05) on bulk modulus for screen size 3.175 mm at all four different unloading pressures, i.e., 20 kPa, 45 kPa, 70 kPa, and 95 kPa, whereas, it did not have a significant effect (p>0.05) for screen size 6.35 mm.
Figure 5.18 Bulk modulus of ground willow for four different conditions at four unloading pressures (N=5)

5.3.2.2 Compression Index

Compression index is an elastoplastic parameter to measure the particulate material’s compressibility at a particular isotropic pressure (Mittal, 2003). It is an important parameter that can be used to formulate the modified Cam-clay model (Desai and Siriwardane, 1984). Figure 5.19 shows the void ratio vs. ln(pressure) for ground willow (screen size 3.175 mm) conditioned at 17.5% w.b. moisture content, which is used to calculate the compression index and spring-back index (section 5.3.2.3).

With the increase in pressure and due to particles rearrangements, voids ratio decreases resulting in increased compression index. This trend can be seen in Figure 5.20 and similar trends were observed by different researchers with other materials (Mittal and Puri, 1999b; Li 1999 and Mittal, 2003). Similar trend was observed for ground switchgrass in section 5.2.2.2. The compression index at different unloading pressures, i.e., 20 kPa, 45 kPa, 70 kPa, and 95 kPa, is not significantly different (p<0.05) among four different conditions of ground willow.
Figure 5.19 A typical void ratio vs ln (Pressure) plot with multiple loading-unloading loops for ground willow (screen size 3.175 mm) conditioned at 17.5% w.b. moisture content.

Figure 5.20 Compression index of ground willow at four different conditions at four unloading pressures (N = 5)
5.3.2.3 Spring-back Index

From Figure 5.21, it is observed that the spring-back index decreases with the increase in pressure. With the increase in pressure, the recovery of material decreases as it gets stiffer (more plastic) and hence the spring back index decreases. The moisture content has significant effect (p<0.05) on spring-back index for 3.175 mm screen size at different unloading pressures, i.e., 20 kPa, 45 kPa, 70 kPa, and 95 kPa, whereas, it did not significantly affect (p>0.05) spring-back index for the larger screen size, 6.35 mm.

![Graph showing spring-back index of ground willow at four different conditions](image)

**Figure 5.21 Spring-back index of ground willow at four different conditions**

5.3.3 Properties of Willow Pellets

Table 5.11 includes the length and diameter of the willow pellets formed at four different conditions. Both diameter and length of pellets do not show any significant difference (p>0.05) among different conditions.

<table>
<thead>
<tr>
<th>Condition of Pellets</th>
<th>Length (mm) (N = 20)</th>
<th>Diameter (mm) (N = 20)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Screen size (mm)</td>
<td>Moisture content (% w.b.)</td>
<td></td>
</tr>
<tr>
<td>3.175</td>
<td>17.5</td>
<td>12.66 ± 3.22</td>
</tr>
<tr>
<td>6.35</td>
<td>17.5</td>
<td>15.03 ± 2.09</td>
</tr>
<tr>
<td>3.175</td>
<td>20</td>
<td>13.89 ± 1.11</td>
</tr>
<tr>
<td>6.35</td>
<td>20</td>
<td>14.80 ± 2.20</td>
</tr>
</tbody>
</table>
Diametral tensile strength, axial compressive strength, pellet density and durability of willow pellets formed at four different conditions are listed in Table 5.12.

**Table 5.12 Quality metrics of willow pellets**

<table>
<thead>
<tr>
<th>Condition of Pellets</th>
<th>Diametral Tensile Strength* (MPa) (N = 10)</th>
<th>Axial Compressive Strength* (MPa) (N = 10)</th>
<th>Pellet Density* (kg/m³) (N = 20)</th>
<th>Durability* (%) (N = 5)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Screen size (mm)</td>
<td>Moisture content (% w.b.)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>3.175</td>
<td>17.5</td>
<td>2.9 ± 0.5</td>
<td>5.2 ± 2.0</td>
<td>1201.7 ± 72.7</td>
</tr>
<tr>
<td>6.35</td>
<td>17.5</td>
<td>4.6 ± 0.9</td>
<td>7.1 ± 1.4</td>
<td>1188.3 ± 90.3</td>
</tr>
<tr>
<td>3.175</td>
<td>20</td>
<td>1.5 ± 0.7</td>
<td>2.1 ± 0.4</td>
<td>1115.0 ± 92.2</td>
</tr>
<tr>
<td>6.35</td>
<td>20</td>
<td>1.9 ± 1.0</td>
<td>3.9 ± 1.2</td>
<td>1086.2 ± 87.0</td>
</tr>
</tbody>
</table>

*Not significantly different (p>0.05)

### 5.3.3.1 Pellet Density

The pellet density of willow pellets listed in Table 5.12 are not significantly different for different conditions (p>0.05). Pellets formed by 3.175 mm screen size reduced at 17.5% w.b. have highest pellet density (1201.7 ± 72.7 kg/m³) as compared to the other conditions. On comparing the pellet densities for these conditions with the bulk densities of ground willow given in Table 5.10, the pelleting process compressed the loose material nearly 6.4 (screen size 3.175 mm at 17.5% w.b.), 7.3 (screen size 3.175 mm at 20% w.b.), 5.8 (screen size 6.35 mm at 17.5% w.b.), and 6.3 (screen size 6.35 mm at 20% w.b.) folds. The relative densities attained by pellets were 96.2% (screen size 3.175 mm at 17.5% w.b.), 98.0% (screen size 3.175 mm at 20% w.b.), 92.8% (screen size 6.35 mm at 17.5% w.b.), and 91.9% (screen size 6.35 mm at 20% w.b.) of the particle density.

### 5.3.3.2 Diametral Tensile Strength

Diametral tensile strength of pellets can be observed from the Table 5.12. Pellets formed with the larger screen size, 6.35 mm, at 17.5% (w.b.) moisture content has highest diametral tensile strength (4.6 ± 0.9 MPa) among all four conditions as shown in Figure 5.19. The moisture content effects diametral strength significantly (p<0.05) for the same screen sizes.
5.3.3.3 Axial Compressive Strength

Axial compressive strength of pellets can be observed from the Table 5.12. Pellets formed with the larger screen size, 6.35 mm, at 17.5\% (w.b.) moisture content has highest axial compressive strength ($7.1 \pm 1.4$ MPa) among all four conditions as seen in Figure 5.20. The moisture content effects axial compressive strength significantly ($p<0.05$) for same the screen size.
5.3.3.4 Durability

Table 5.12 includes the results for durability of willow pellets for four different conditions. The values of durability are not significantly different (p>0.05). In Figure 5.24, it can be observed that for all four conditions, durability is more than 80%, hence it can be said that pellets formed at these conditions were highly durable. However, the range of variation of pellet durability is narrow from 84% to 90%. Therefore, no predictive relationships between durability and mechanical properties from the HTC tests will be developed.

![Figure 5.24 Durability of ground willow at four different conditions](image)

5.3.4 Relationship among Physical and Mechanical Properties of Ground Willow and Pellet Qualities

The correlation coefficients of strengths of pellets and mechanical properties of ground willow are included in the Table 5.13. It can be observed from the table that the values for compression index at 20 kPa are highly correlated (R²>80%) with the axial compressive and diametral tensile strength of willow pellets. The highest correlation coefficient for each property is highlighted and bolded in the Table 5.13. The property value with the highest R-square is compression index. Respective values were used for developing the predictive relationships among mechanical properties of ground material and pellets in section 5.4.
Table 5.13 Correlation coefficients for mechanical properties of ground willow and pellets*

<table>
<thead>
<tr>
<th></th>
<th>Axial Compressive Strength</th>
<th>Diametral Tensile Strength</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bulk Modulus - 20 kPa</td>
<td>-0.295</td>
<td>-0.282</td>
</tr>
<tr>
<td>Bulk Modulus - 45 kPa</td>
<td>-0.060</td>
<td>-0.048</td>
</tr>
<tr>
<td>Bulk Modulus - 70 kPa</td>
<td>-0.134</td>
<td>-0.141</td>
</tr>
<tr>
<td>Bulk modulus - 95 kPa</td>
<td>0.045</td>
<td>0.005</td>
</tr>
<tr>
<td>Compression Index - 20 kPa</td>
<td><strong>0.963</strong></td>
<td><strong>0.998</strong></td>
</tr>
<tr>
<td>Compression Index - 45 kPa</td>
<td>0.721</td>
<td>0.590</td>
</tr>
<tr>
<td>Compression Index - 70 kPa</td>
<td>0.144</td>
<td>0.110</td>
</tr>
<tr>
<td>Compression Index - 95 kPa</td>
<td>0.151</td>
<td>0.127</td>
</tr>
<tr>
<td>Spring-back Index - 20 kPa</td>
<td>-0.372</td>
<td>-0.317</td>
</tr>
<tr>
<td>Spring-back Index - 45 kPa</td>
<td>-0.031</td>
<td>-0.006</td>
</tr>
<tr>
<td>Spring-back Index - 70 kPa</td>
<td>-0.004</td>
<td>-0.004</td>
</tr>
<tr>
<td>Spring-back Index - 95 kPa</td>
<td>0.084</td>
<td>0.076</td>
</tr>
</tbody>
</table>

Highlighted and bolded values – highest correlation coefficient for respective properties among four different unloading pressures

5.4 Comparison among Various Properties of Three Materials

This section compares the three test materials on the basis of different physical and mechanical properties discussed in sections 5.1, 5.2, and 5.3. Following sub-sections will compare results for physical and mechanical properties of ground biomass, and pellet qualities of three biomass.

5.4.1 Physical Properties of Ground Biomass

The bulk density, particle density, and particle size distribution of ground corn stover, switchgrass, and willow are discussed in details in sections 5.1.1, 5.2.1, and 5.3.1, respectively. For all three biomass, the screen size has a significant effect (p<0.05) on the particle size distribution, $D_{50}$, and bulk density, however, it does not significantly affect the particle density (p>0.05).

In Figure 5.25, it can be observed that the ground switchgrass has the smallest $D_{50}$, whereas, willow has the highest $D_{50}$, when size reduced with the same screen size. This same trend can be observed for both screen sizes. The initial moisture content of these biomass were 4.2% w.b. for corn stover, 3.8% w.b. for switchgrass, and 5.5% w.b. for willow, which can be an influencing factor for different particle size distribution of...
three biomass for the same screen size, in addition to their different chemical composition.

Figure 5.25 Particle size distribution of ground corn stover, switchgrass, and willow (screen size: 3.175 mm and 6.35 mm) (N = 5)

\[ D_{50} \] of corn stover reduced with 6.35 mm screen size (1.0 mm) was higher by 30.0% than reduced with 3.175 mm screen size (0.7 mm). \[ D_{50} \] of switchgrass reduced with 6.35 mm screen size (0.97 mm) was higher by 37.1% than reduced with 3.175 mm screen (0.61 mm), whereas, \[ D_{50} \] of willow reduced with 6.35 mm screen size (1.26 mm) was higher by 33.7% than reduced with 3.175 mm screen (0.94 mm). Hence, it can be said that \[ D_{50} \] values for all three biomass, size reduced biomass with 6.35 mm screen size was higher by 30-40% than that reduced with 3.175 mm screen size.

5.4.2 Mechanical Properties of Ground Biomass

The mechanical properties of ground corn stover, switchgrass, and willow are discussed in the sections 5.1.2, 5.2.2, and 5.3.2, respectively. The trends of pressure vs. volume strain and of void ratio vs. ln(pressure) are similar for all three materials, i.e.,
corn stover, switchgrass, and willow, however, the magnitudes are different. In addition, the trend of bulk modulus with increase in unloading pressure is also similar for all three materials (Figures 5.3, 5.9, and 5.18). Willow size reduced with 3.175 mm screen size at 17.5% w.b. has highest bulk modulus at 20, 45, 70 and 95 kPa unloading pressures among different conditions of all three biomass. Higher bulk modulus indicates that there was less recovery of material upon releasing the pressure.

The values of compression index increases with increase in unloading pressure for the same material and similar trend can be observed for all three materials (Figure 5.5, 5.11, and 5.20). As the pressure increases, it facilitates particle rearrangement. The voids ratio decreases resulting in increased compression index. In case of spring-back index, it decreases up to a certain pressure value but thereafter, it shows an increase at higher pressure. With the increase in pressure, the recovery of material decreases as it gets stiffer and hence the spring back index decreases. A similar trend was observed for all three materials, except for ground willow size reduced with 3.175 mm screen size.

**5.4.3 Pellet Quality**

The properties of corn stover, switchgrass, and willow pellets are discussed in sections 5.1.3, 5.2.3, and 5.3.3, respectively. From figure 5.26, it can be observed that the diametral tensile strength is 4-5.8 MPa for corn stover, 0.9-3.8 MPa for switchgrass and 0.8-5.7 MPa for willow pellets, respectively. Axial compressive strength is 8-11.5 MPa for corn stover, 0.8-5.7 MPa for switchgrass and 1.5-8.5 MPa for willow pellets, respectively. Corn stover pellets formed with 3.175 mm screen size at 20% w.b. has the highest diametral tensile and axial compressive strengths among different conditions of three biomass.

As discussed in the aforementioned sections, pellet durability for all three materials is more than 80%. The pelletization process compresses the loose material nearly 12-14 folds for corn stover, 7-9 folds for switchgrass and 5-7 folds for willow, respectively. The achieved relative pellet densities are 96%-98% for corn stover, 67%-88% for switchgrass, and 91%-98% for willow, respectively.
Figure 5.26 Diametral tensile and axial compressive strengths of corn stover, switchgrass, and willow pellets at different conditions

*CS denotes Corn stover, SW - switchgrass, and WI – willow
*3.175 mm and 6.350 mm are the screen sizes and 17.5% and 20% are moisture content (w.b.) conditions

5.5 Development of Predictive Relationships among Physical and Mechanical Properties of Ground Biomass and Pellet Qualities

This section focus on developing predictive relationships based on regression analysis using Minitab (Version 16.1.0.0, State College, PA). As mentioned previously, the purpose of these correlations is to develop relationship among the mechanical properties of ground material and quality properties of resulting pellets.

Since ground corn stover formed durable pellets at two conditions, i.e. 3.175 mm screen size at 20% w.b. and 6.35 mm screen size at 17.5% w.b., enough data was not produced to develop predictive relationship for this material. Switchgrass and willow produced pellets at all four conditions, which has been used in developing predictive relationships.

As noted in respective section, the range of variation of pellet durability for all three materials is very narrow; hence, developing predictive relationships is not very
meaningful. Therefore, no predictive relationships between durability and mechanical properties from the HTC tests were developed.

5.5.1 Switchgrass

Based on correlation coefficients of mechanical properties of ground switchgrass and pellets quality in Table 5.8, regression analysis was performed using Minitab. The parameters with the highest R-square coefficients were bulk modulus, compressive index, and spring-back index. From Figure 5.27, the bulk modulus of ground switchgrass screen size 3.175 mm conditioned at 17.5% w.b. and diametral tensile strength of pellets at same condition is highest among all four conditions. The bulk modulus of ground switchgrass screen size 6.35 mm at 20% w.b. and diametral tensile strength is least among all four conditions. These trends suggest that stronger pellets are formed at condition which has high bulk modulus. Higher value of bulk density suggests low tendency to recover, i.e. exhibits more plastic behavior. The particle size distribution also plays an important role in the packing of material. The voids created between larger particles filled with finer particles, which results in stronger pellets.

![Figure 5.27 Diametral tensile strength of pellets vs. bulk modulus of ground switchgrass at 95 kPa for four different conditions](image-url)
Based on regression analysis, the regression equation to predict diametral tensile strength (DTS) on basis of bulk modulus (BM) at 95 kPa is given below,

\[ \text{DTS}_{\text{switchgrass}} = 9.000 \times \text{BM}_{\text{switchgrass}} + 0.627, \quad R^2 = 88.32\% \quad \ldots (5.1) \]

Similar trend can be observed for axial compressive strength (ACS) of pellets and bulk modulus (BM) of ground switchgrass at 70 kPa in Figure 5.28. Figure 5.28 includes the fit line and regression equation for these two properties is given below,

\[ \text{ACS}_{\text{switchgrass}} = 4.600 \times \text{BM}_{\text{switchgrass}} - 0.716, \quad R^2 = 83.83\% \quad \ldots (5.2) \]

![Figure 5.28 Axial compressive strength of pellets vs. bulk modulus of ground switchgrass at 70 kPa for four different conditions](image)

In Figure 5.29, compression index of ground switchgrass and diametral tensile strength of pellets is highest with screen size 3.175 mm at 17.5% w.b. condition. As compression index is a measure of the materials’s compressibility, it leads to stronger pellets formation at higher index. It can also be observed that for the same screen size, conditions at lower moisture content forms stronger pellets and the trend can also be observed that smaller screen size forms the better pellets. Figure 5.29 also presents the fit
line and regression equation for these properties, which is highly correlated at calculated $R^2$ value of 99.49%. The regression equation for diametral tensile strength (DTS) and compressive index (CI) at 95 kPa is given below,

$$DTS_{\text{switchgrass}} = 5.724 \times CI_{\text{switchgrass}} + 3.790, \quad R^2 = 99.49\% \quad \ldots(5.3)$$

![Figure 5.29 Diametral tensile strength of pellets vs. compression index of ground switchgrass at 95 kPa for four different conditions](image)

A similar trend can be observed in Figure 5.30 for axial compressive strength and compression index. The regression equation for axial compressive strength and compressive index at 70 kPa is given below,

$$ACS_{\text{switchgrass}} = 14.085 \times CI_{\text{switchgrass}} + 9.711, \quad R^2 = 90.1\% \quad \ldots(5.4)$$

In Figure 5.31, diametral tensile strength and spring-back index of pellets is highest with screen size 3.175 mm at 17.5% w.b. condition. The regression equation for diametral tensile strength and spring-back index (SI) at 95 kPa is given below,

$$DTS_{\text{switchgrass}} = 7.566 \times SI_{\text{switchgrass}} + 3.447, \quad R^2 = 97.58 \quad \ldots(5.5)$$
Figure 5.30 Axial compressive strength of pellets vs. compression index of ground switchgrass at 70 kPa for four different conditions

Figure 5.31 Diametral tensile strength of pellets vs. spring-back index of ground switchgrass at 95 kPa for four different conditions
The similar trend can be observed in Figure 5.32 for spring-back index of ground switchgrass and axial compressive strength. The regression equation for axial compressive strength and spring-back index at 70 kPa is given below,

\[ \text{ACS}_{\text{switchgrass}} = 26.133 \times \text{SI}_{\text{switchgrass}} + 9.166, \quad R^2 = 94.21 \quad \cdots (5.6) \]

Figure 5.32 Axial compressive strength of pellets vs. spring-back index of ground switchgrass at 95kPa for four different conditions

Based on the regression analysis (Table 5.8), it was observed that bulk modulus, compression index, and spring-back index are suitable parameters for predicting diametral tensile strength and axial compressive strength of switchgrass pellets. Table 5.14 summarizes the regression equations obtained.
Table 5.14 Regression equation for predicting diametral tensile strength and axial compressive strength for switchgrass pellets*

<table>
<thead>
<tr>
<th>Regression equation</th>
<th>R²</th>
</tr>
</thead>
<tbody>
<tr>
<td>$DTS_{\text{switchgrass}} = 9.0 \times BM_{95 \text{ kPa, switchgrass}} + 0.627$</td>
<td>88.32</td>
</tr>
<tr>
<td>$ACS_{\text{switchgrass}} = 4.6 \times BM_{70 \text{ kPa, switchgrass}} - 0.716$</td>
<td>83.83</td>
</tr>
<tr>
<td>$DTS_{\text{switchgrass}} = 5.724 \times CI_{95 \text{ kPa, switchgrass}} + 3.790$</td>
<td>99.49</td>
</tr>
<tr>
<td>$ACS_{\text{switchgrass}} = 14.085 \times CI_{70 \text{ kPa, switchgrass}} + 9.711$</td>
<td>90.10</td>
</tr>
<tr>
<td>$DTS_{\text{switchgrass}} = 7.566 \times SI_{95 \text{ kPa, switchgrass}} + 3.447$</td>
<td>97.58</td>
</tr>
<tr>
<td>$ACS_{\text{switchgrass}} = 26.133 \times SI_{95 \text{ kPa, switchgrass}} + 9.166$</td>
<td>94.21</td>
</tr>
</tbody>
</table>

*Diametral tensile strength, Axial compressive strength, Bulk modulus are in MPa

5.5.2 Willow

Based on correlation coefficients of mechanical properties of ground willow and pellet quality parameters in Table 5.13, regression analysis was performed using Minitab. In Figure 5.33, compression index (CI) of ground willow and diametral tensile strength (DTS) of pellets is highest with screen size 3.175 mm at 17.5% w.b. condition. As compression index is a measure of the material’s compressibility, it leads to stronger pellets formation at higher CI values. It can also be observed that for the same screen size, conditions at lower moisture content forms stronger pellets and the trend can also be observed that smaller screen size forms the better pellets. The regression equation for diametral tensile strength and compressive index at 20 kPa is given below,

$$DTS_{\text{willow}} = 51.280 \times CI_{\text{willow}} + 26.056, R^2 = 99.67$$  \quad \ldots (5.7)

A similar trend can be observed in Figure 5.34 for axial compressive and compression index. The regression equation for diametral tensile strength and compressive index at 20 kPa is given below,

$$ACS_{\text{willow}} = 75.502 \times CI_{\text{willow}} + 38.927, R^2 = 92.71$$  \quad \ldots (5.8)
Figure 5.33 Diametral tensile strength of pellets vs. compression index of ground willow at 20 kPa for four different conditions

![Graph showing diametral tensile strength vs. compression index.]

- Formula: $y = 51.280x + 26.056$
- $R^2 = 0.9967$

Figure 5.34 Axial compressive strength of pellets vs. compression index of ground willow at 20 kPa for four different conditions

![Graph showing axial compressive strength vs. compression index.]

- Formula: $y = 75.502x + 38.927$
- $R^2 = 0.9271$
Compression index is an indication of compressibility of the material. Based on the regression analysis (Table 5.13), it was observed that compression index at 20 kPa is the most suitable parameter for predicting diametral tensile strength and axial compressive strength of willow pellets. Table 5.15 summarizes the regression equations obtained by analysis.

Table 5.15 Regression equation for predicting diametral tensile strength and axial compressive strength for willow pellets (Compression index at 20 kPa)

<table>
<thead>
<tr>
<th>Regression equation</th>
<th>$R^2$ (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\text{DTS}<em>{\text{willow}} = 51.28*\text{CI}</em>{20 \text{kPa, willow}} + 26.056$</td>
<td>99.67</td>
</tr>
<tr>
<td>$\text{ACS}<em>{\text{willow}} = 75.502*\text{CI}</em>{20 \text{kPa, willow}} + 38.927$</td>
<td>92.71</td>
</tr>
</tbody>
</table>

5.5 Validation of the Predictive Relationships

For the validation of the developed relationships, switchgrass and willow ground with 6.35 mm was conditioned at 18.75% w.b. to form pellets. Based on the results of this study, it was observed that the screen size did not significantly effects (p>0.05) the mechanical properties of ground biomass and quality metrics of pellets, whereas, moisture content affects them significantly (p<0.05). Hence, the larger screen size was selected for the validation to save the energy input for the pelletization. The selected moisture content value is the average of two moisture content values used in this study. Pellets were subjected to diametral tensile strength and axial compressive strength tests on the universal testing machine.

5.5.1 Switchgrass

Switchgrass pellets formed with 6.35 mm screen size at 18.75% w.b. moisture content has 1.9 ± 0.5 MPa diametral tensile strength of the pellets and 3.9 ± 1.1 MPa axial compressive strength. The value of spring-back index for screen size 6.35 mm at 18.75% w.b. can be predicted by Figure 5.3.
Figure 5.35 Spring-back index vs. moisture content for ground switchgrass (6.35 mm screen size reduced)

From Figure 5.33, the spring-back value for ground switchgrass at 18.75% w.b. found out to be -0.2186. To validate the axial compressive strength vs spring-back index equation listed in Table 5.14, the calculated value is used in this equation, which gives the value of 3.45 MPa for axial compressive strength. The percentage difference between predicted (3.45 MPa) and measured value (3.9 ± 1.1 MPa) is 11.54%, which is well within the measured percentage difference spread in the axial compressive strength of 78.57%. Therefore, the predictive relationship for axial compressive strength values for switchgrass can be used with confidence.

Similarly, the predicted value of diametral tensile strength from the equation is 1.79 MPa, whereas the actual value is 1.9 ± 0.5 MPa. The percentage difference between predicted and actual value is 5.79%, which is well within the measured percentage difference spread in the diametral tensile strength of 71.43%. Therefore, the predictive relationship for diametral tensile strength values for switchgrass can be used with confidence.
5.5.2 Willow

Willow pellets formed with 6.35 mm screen size at 18.75% w.b. moisture content has 2.8 ± 0.7 MPa diametral tensile strength of the pellets and 5.9 ± 1.5 MPa axial compressive strength. The value of axial compressive index for screen size 6.35 mm at 18.75% w.b. can be predicted by Figure 5.36.

![Figure 5.36 Compression index vs. moisture content for ground willow (6.35 mm screen size reduced)](image)

From Figure 5.34, the compression index for ground willow at 18.75% w.b. found out to be -0.446. To validate the axial compressive strength vs compression index equation listed in Table 5.15, the calculated value is used in this equation, which gives the value of 5.25 MPa for axial compressive strength. The percentage difference between predicted (5.25 MPa) and measured value (5.9 ± 1.5 MPa) is 11.02%, which is well within the measured percentage difference spread in the axial compressive strength of 68.18%. Therefore, the predictive relationship for axial compressive strength value for willow can be used with confidence.

Similarly, the predicted value of diametral tensile strength from the equation is 3.18 MPa, whereas, the measured value is 2.8 ± 0.7 MPa. The percentage difference between predicted and measured value is 13.57%, which is well within the measured
percentage difference spread in the axial compressive strength of 66.67%. Therefore, the predictive relationship for axial compressive strength values for willow can be used with confidence.
Chapter 6. Summary, Conclusions, and Future Recommendations

In the present study, linear relationships between the mechanical properties of ground biomass and quality of pellets were developed. The physical properties and mechanical properties of ground biomass (corn stover, switchgrass, and willow) were determined. Hydrostatic triaxial compression (HTC) tests were conducted using a CTT for three biomass ground with two different screen sizes and conditioned at two moisture contents. Parameters such as bulk modulus, compression index, and spring-back index were determined using data obtained from HTC tests. Diametral strength, axial compressive strength, and durability tests were conducted to evaluate the pellets’ quality. Relationships between the mechanical properties of ground biomass and pellet quality parameters were developed. In the following sections, the key conclusions are summarized.

6.1 Physical and Mechanical Properties of Ground Biomass

The size distribution, D_{50}, particle density, and bulk density of ground biomass were determined. The bulk modulus, compression index, and spring-back index were determined using the HTC test at unloading pressures of 20, 45, 70, and 95 kPa. The key conclusions are noted below.

- Based on the determined D_{50} values for all three biomass, size reduced biomass with 6.35 mm screen size was higher by 30-40% than that reduced with 3.175 mm screen size
- The same material size reduced with different screen sizes does not change the particle density significantly (p>0.05); however, the size reduction significantly effects (p<0.05) the bulk density. Bulk density of size reduced biomass with smaller screen size is higher than that of larger screen size.

6.1.1 Corn Stover

- Bulk modulus of larger screen was higher than smaller screen for same unloading pressure.
In case of the larger screen size, the value of bulk modulus initially decreased slightly at 45 kPa and then increased till 95 kPa, whereas the value of bulk modulus of smaller screen size increased with increasing pressure.

Compression index value increased with pressure in all cases, i.e., material became more compressible with increase in pressure.

Spring-back index values initially decreased till 70 kPa and then slightly increased at 95 kPa.

6.1.2 Switchgrass

- Bulk modulus increased with increase in the isotropic pressure in all four conditions.
- Compression index increased with increase in the isotropic pressure in all four conditions.
- Spring-back index values initially decreased till 70 kPa and then slightly increased at 95 kPa.

6.1.3 Willow

- Bulk modulus increased with increase in the isotropic pressure in all four conditions.
- Compression index increased with increase in the isotropic pressure in all four conditions.
- For smaller screen size, the spring-back index decreased with the increase in the unloading pressure. However, for larger screen size, the spring-back index values initially decreased till 70 kPa and then slightly increased at 95 kPa.

6.2 Quality Metrics of Resulting Pellets

The pellet density, durability, axial compressive strength, and diametral tensile strength were determined. The key conclusions are noted below.

- The pellet densities were not significantly different (p>0.05) for different screen sizes.
• The screen size does not have significant effect (p>0.05) on the axial compressive strength and diametral tensile strength, however, moisture content has significant effect (p<0.05) on the axial compressive strength and diametral tensile strength.

• Pellets formed with the smaller screen size reduced biomass has higher axial compressive and diametral tensile strength, however, the values are not significantly different (p>0.05) than the larger screen size reduced biomass.

• Pellet durability for all three materials was more than 80% and the range of durability values for corn stover, switchgrass, and willow were, 94%- 98%, 87%- 94%, and 83%-91%, respectively.

• The pelleting process compresses the loose material nearly 12-14 folds for corn stover, 7-9 folds for switchgrass and 5-7 folds for willow. The achieved relative pellet densities were 96%-98% for corn stover, 67%-88% for switchgrass, and 91%-98% for willow.

6.3 Development of Relationships among the Physical and Mechanical Properties of Ground Material and Pellet Qualities

Compression index and spring-back index (at 70 kPa and 95 kPa unloading pressure) were found most suitable ($R^2 > 90\%$) for predicting diametral tensile strength and axial compressive strength of switchgrass pellets. Compression index at 20 kPa unloading pressure was found most suitable ($R^2 > 90\%$) for predicting diametral tensile strength and axial compressive strength for willow pellets. Based on the observations, predictive relationships for selected pellet qualities of switchgrass and willow pellets were developed.

6.4 Validation of Relationships among the Physical and Mechanical Properties of Ground Material and Pellet Qualities

For the validation of the developed relationships listed in Table 5.15 and 5.15, switchgrass and willow ground with 6.35 mm screen size was conditioned at 18.75% w.b. to form pellets. The percentage differences of predicted and measured strength, which were 6%-12% for switchgrass and 12%-14% for willow, are comparable to spread in the
measured strength. Hence, these developed relationships can be used to predict the quality of pellets.

6.5 Recommendations for Future Work

The present research investigated the relationship between the physical and mechanical properties of ground biomass and pellet quality parameters. Following recommendations are proposed based on this study:

1. Mechanical properties of unconditioned biomass should be determined to study the effect on mechanical properties of conditioned biomass and on pellet qualities.
2. The conventional triaxial compression (CTC) test should be conducted to determine the shear modulus and failure stress.
3. Since this study emphasized on bulk material properties, further investigation of individual particle characterization should be performed to understand the science behind the densification and interaction of particles during the densification process.
4. In industries, some binders such as corn starch, vegetable oils are used to form the pellets; therefore, effect of binders should also be studied for effective pelletization.
5. Since biomass densification (pelletization) is very similar to the powder compaction field, which has achieved much more in predicting the compaction behavior of irregular particle distributions, the powder compaction field could be looked upon for future research opportunities in the field of particle technology and compaction.
6. For a more fundamental understanding of why the predictive relationships work, a rational principle-based theoretical framework should be investigated and developed.
7. The results were analyzed using statistical techniques, however, explanation using physics for the measured magnitudes and trends needs to be included; this fundamental link needs to be developed for deeper insights to the pelletization/compaction process.
References


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Qualifications:
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• “Development of Dehydrated Spinach Product” under the guidance of Dr. S. K. Garg for partial fulfillment of Bachelor of Technology at G.B.P.U.A.T.

Presentations: