EVALUATION OF DRY STEAM PRECONDITIONING ON SWITCHGRASS PELLET QUALITY METRICS

A Thesis in

Agricultural and Biological Engineering

by

Curtis Covelli

Submitted in Partial Fulfillment of the Requirements for the Degree of

Master of Science

December 2016
The thesis of Curtis Covelli was reviewed and approved* by the following:

Virendra M. Puri  
Distinguished Professor of Agricultural and Biological Engineering  
Thesis Adviser

Hojae Yi  
Research Associate of Agricultural and Biological Engineering

Daniel Ciolkosz  
Research Associate and Assistant Professor, of Agricultural and Biological Engineering

Dawn Luthe  
Professor of Plant Stress Biology

Paul Heinemann  
Professor of Agricultural and Biological Engineering  
Head of the Department of Agricultural and Biological Engineering

*Signatures are on file in the Graduate School
ABSTRACT

This research investigated the impact of thermal preconditioning with dry steam on the quality of pellets manufactured from ground switchgrass. The intended impact of preconditioning was to utilize the softening of naturally present binders, such as lignin, to improve pelletized biomass quality.

An autoclave was used in dry steam preconditioning of ground switchgrass and to investigate the effect of elevated time-temperature treatment on switchgrass pellet quality metrics. Switchgrass was moisture conditioned at 17.5% and 20.0% wet basis (w.b) and preconditioned in the autoclave with dry steam at temperatures of 100°C and 120°C for five minutes. The properties of ground material and the pellets (both control and dry steam preconditioned) were characterized. Determined ground material properties included particle density, bulk density, and particle size distribution. Determined pellet quality metrics included diametral strength, axial strength, friability, durability, and pellet density.

Particle density was observed to significantly decrease (p<0.05) when conditioned by the dry steam. This might have been caused by effects by moisture inducing the migration of natural binders to the outside of the ground particles and eventual evaporation. An increase in pellet diametral strength was observed for the 120°C dry steam preconditioning (p>0.05). Axial strength values of single pellets for control and dry steam preconditioned ground switchgrass were not significantly different (p>0.05). Pellet durability analysis in bulk pellets resulted in an average friability around 94% for control and all treatments. The more aggressive box durability tests indicated that dry steam conditioned pellets had a 7-10% lower durability around 78%. Pellet quality metrics of pellet density, strength, friability, and durability were found to be minimally impacted from dry steam preconditioning.
# Table of Contents

List of Figures ................................................................. vii
List of Tables ........................................................................ ix
1. Introduction........................................................................ 1
2. Literature Review ............................................................ 2
   2.1. Purpose of Densification ................................................... 2
   2.2. The Compaction Process ................................................ 2
   2.3. Types of Biomass Densification ......................................... 3
       2.3.1. Cubes and Unground Agglomerations ....................... 4
       2.3.2. Advantage of Pellets ............................................... 4
   2.4. Properties of Ground Biomass ......................................... 6
   2.5. Mechanical Properties of Ground Biomass ....................... 8
   2.6. Cubical Triaxial Tester (CTT) ........................................... 9
   2.7. Pelletization Process ..................................................... 10
   2.8. Properties of Densified Biomass ..................................... 11
       2.8.1. Pellet Density ....................................................... 11
       2.8.2. Pellet Strength ..................................................... 12
       2.8.3. Pellet Quality Standards ......................................... 13
       2.8.4. Pellet Durability .................................................... 13
   2.9. Factors Affecting Pelletization ....................................... 15
       2.9.1. Moisture Content ................................................... 15
       2.9.2. Die Temperature .................................................... 16
       2.9.3. Thermal Activation of Natural Binders ...................... 16
       2.9.4. Particle Size ......................................................... 16
   2.10. Densification Preprocessing and Preconditioning ............. 17
       2.10.1. Additives .......................................................... 17
       2.10.2. Steam Explosion .................................................... 18
       2.10.3. Torrefaction ......................................................... 18
       2.10.4. Thermal Preconditioning with Steam ....................... 19
   2.11. Evaluation of Thermal Preconditioning with Steam on Switchgrass Pellet Quality .......... 19
3. Goal, Hypothesis, and Objectives ...................................... 21
   3.1. Goal ........................................................................... 21
3.2. Hypotheses .................................................................................................................. 21
3.3. Objectives: .................................................................................................................... 22
4. Methodology ................................................................................................................... 23
  4.1. Overview and Flowchart of Methodology ................................................................. 23
  4.2. Materials and Equipment ......................................................................................... 24
  4.3. Experimental Design................................................................................................. 25
  4.4. Material Processing .................................................................................................... 26
    4.4.1. Size Reduction ........................................................................................................ 26
    4.4.2. Moisture Preconditioning .................................................................................... 27
    4.4.3. Preconditioning with Dry Steam ........................................................................... 28
  4.5. Physical Properties of Ground Switchgrass ............................................................. 30
  4.6. Pellet Formation ........................................................................................................ 31
5. Results and Discussion .................................................................................................... 37
  5.1. Physical properties of ground switchgrass .............................................................. 37
    5.1.1. Particle Density ....................................................................................................... 38
    5.1.2. Bulk density .......................................................................................................... 38
    5.1.3. Particle Size Distribution ...................................................................................... 38
  5.2. Pellet density ............................................................................................................. 39
  5.3. Pellet Strength ........................................................................................................... 41
    5.3.1. Axial compressive strength ................................................................................... 42
    5.3.2. Diametral strength ............................................................................................... 44
  5.4. Durability .................................................................................................................. 45
    5.4.1. Box Durability ....................................................................................................... 46
    5.4.2. Friabilator Durability ............................................................................................ 47
6. Summary, Conclusions, and Future Recommendations ................................................ 48
  6.1. Physical Properties of Ground Biomass .................................................................... 48
    6.1.1 Bulk Density ........................................................................................................... 48
    6.1.2 Particle Density ...................................................................................................... 48
    6.1.3 Particle Size Distribution ..................................................................................... 48
  6.2. Pellet Properties ....................................................................................................... 49
    6.2.1 Pellet Density ......................................................................................................... 49
    6.2.2 Pellet Strength ....................................................................................................... 49
    6.2.3 Pellet Durability .................................................................................................... 49
6.3. Recommendations for Future Work................................................................. 50
7. References........................................................................................................... 51
List of Figures

Figure 2.1 Force-displacement curve where A is compaction and B is decompression. Arrows indicate the desire from real (solid lines) to ideal (dotted line). [adapted from:(Stanley-Wood, 1983)] ............................................................... 3
Figure 2.2 Images of (a) pellets, (b) briquettes, and (c) cubes. (Clarke et al., 2011) .......... 4
Figure 2.3 Bulk densities of densification technologies and coal (Clarke et al. 2011) .......... 5
Figure 2.4 Chemical Structures of (a) Lignin, (b) Hemicellulose, and (c) Cellulose (Wikimedia.org) .................................................................................................................. 8
Figure 2.5 A typical isotropic stress (Pressure) vs. volumetric strain response with a cyclic hydrostatic stress path (Karamchandani et al., 2015) ......................................................... 10
Figure 2.6 Model of the densification process in a typical pellet mill (Nielsen, Gardner, Poulsen, & Felby, 2009) ............................................................................................................. 11
Figure 2.7 Axial compressive strength of switchgrass under varied conditions (Karamchandani et al., 2015) ............................................................................................................. 11
Figure 2.8 Switchgrass diametral compressive strength at varied conditions (Karamchandani et al., 2015) ............................................................................................................. 13
Figure 2.9 Image of tumbling box fabricated in the Agricultural and Biological Engineering Department according to ASABE standard S269.5 specification to be used to test pellet durability .......................................................................................................................... 15
Figure 4.1 Flowchart of methodology .............................................................................. 24
Figure 4.2 Switchgrass material, before (a) and after (b) size reduction using 6.35 mm screen size. ......................................................................................................................... 25
Figure 4.3 Size reduction machinery (Munson SCC-10) and particle collector (Cincinnati Fan, 50S/TI) ............................................................................................................................. 27
Figure 4.4 Hard Goods Cycle held for 5-minute exposure time of target 100°C steady state temperature .......................................................................................................................... 29
Figure 4.5 Layout of steam trays in autoclave .................................................................. 29
Figure 4.6 Farm-scale, batch pelletizer ............................................................................. 32
Figure 4.7 Image of friability tester .................................................................................. 32
Figure 4.8 Universal testing machine before a diametral compressive strength test .......... 34
Figure 4.9 Axial compressive strength schematic (a) and image of test (b) (Karamchandani et al., 2015) ................................................................. 34
Figure 4.10 Diametral tensile strength schematic (a) and image of test (b) (Karamchandani et al., 2015) ................................................................. 35
Figure 5.1 Mean particle size distributions for all treatment levels ................................................................. 39
Figure 5.2 Interval plot of pellet density with 17.5% and 20.0% w.b. moisture content, for untreated and steam temperature of 100 and 120°C treatment condition ................................................. 41
Figure 5.3 Confidence interval (95%) plot of pellet axial compressive strength with 17.5% and 20.0% (w.b.) moisture content, for untreated and steam temperature treatments of 100 and 120°C (N=10) ........................................................................................................................................................................... 43
Figure 5.4 95% confidence interval plot of pellet diametral tensile strength with 17.5% and 20.0% w.b. moisture content, for untreated and steam treatment at 100 and 120°C .................. 44
Figure 5.5 95% confidence interval (CI) plot of pellet friability with 17.5% and 20.0% w.b. moisture content, for untreated and steam temperature of 100 and 120°C treatment condition. . 47
List of Tables

Table 2.1 General summary and comparison of densification processes (Clarke et al., 2011; Stelte, Clemons, et al., 2012; Tumuluru & Wright, 2011)................................................................. 6
Table 2.2: Typical lignocellulosic composition of selected biomaterials (Kaliyan & Morey, 2009; Szczukowski, Tworkowski, Klasa, & Stolarski, 2002; Tumuluru & Wright, 2011)......................... 8
Table 2.3 Explanation of fundamental mechanical parameters (Li & Puri, 2003).......................... 9
Table 2.4 Summary of switchgrass pellet quality metrics (Karamchandani et al., 2015)............. 12
Table 4.1 Design of experiment........................................................................................................... 26
Table 4.2 Experimental plan for replications.................................................................................... 26
Table 5.1 Physical Properties of ground switchgrass ........................................................................ 37
Table 5.2 Pellet Density ....................................................................................................................... 40
Table 5.3 Pellet strength results ........................................................................................................ 42
Table 5.4 Durability of Switchgrass Pellets....................................................................................... 46
ACKNOWLEDGEMENTS

This thesis becomes a reality thanks to the help and support of a number of kind individuals. The following section takes time to extend my sincere thanks to all of them.

I would like to express my heartfelt thanks and gratitude to my advisor and co-advisor, Dr. Virendra M. Puri, and Dr. Hojae Yi. I would like to thank Dr. Puri for his guidance, knowledge and wisdom throughout my college career. As my undergraduate advisor as well, the past six years have been a pleasure to learn and grow under his teachings. Thanks also to Dr. Yi for many of our insightful discussions. His thoughtful advice in times of struggle was much appreciated. I am humbled that despite both of their busy schedules they always managed to make time to provide their knowledge and comments. Thank you both for the continued support and guidance.

Thank you to my committee members for their thoughtful revisions and suggestions. To Dr. Daniel Ciolcosz for his thorough review and expertise; and Dr. Dawn Luthe for her encouragement and perspective from outside the department.

A sincere thank you to the Pennsylvania Agricultural Experiment Station and Northeast Sun Grant Initiative for providing funding and financial support for this research. Thanks to Mr. Randall G. Bock and Dr. Roderick S. Thomas for their technical support. Thanks as well as Kay Dimarco and Mark Signs for usage of size reduction equipment. Thanks also to Dr. Paul Heineman, Head of the Department of Agricultural and Biological Engineering, for the resources and environment. The department has come to feel like a home away from home.

Special thank you to fellow graduate student Apoorva Karamchandani, whose past research allowed me to build further for research of my own. Thanks as well for your mentorship during and beyond my transition as a graduate student.

Last a sincere thank you to my mom and dad, Carmen and Curt Covelli, as well as my sister Gina. I am forever grateful for your love, support and guidance.
1. Introduction

Biomass is the third largest energy resource behind coal and oil (Tumuluru & Wright, 2011). In addition to biofuel production, biomass is a commonly used and growing source of heat and power (Vinterbäck 2004; Stelte et al. 2012b). A major challenge using biomass originates from its low bulk density, which makes biomass inefficient and costly to handle, store, and transport. For example, transportation cost is the second highest expense after capital cost when considering a direct biomass fired power plant (Biswas, Yang, & Blasiak, 2011). Pelletizing is a promising option to address these logistical barriers.

Forming biomass into pellets increases bulk density of pellets *en masse* to about 700 kg/m$^3$ from 40-150 kg/m$^3$ for grasses and 150-200 kg/m$^3$ for woodchips (Stelte et al. 2012b). This manifold increase in bulk density makes the biomass much easier and more cost efficient to transport and store. The biomass pellet also has an advantage over other densification technologies such as briquetting because it easily integrates into existing feed pellet systems (Stelte et al. 2012b). The pellet industry is in a period of significant growth. It is estimated that the consumption by Europe alone will reach 50 million tons in 2020 (Biswas et al., 2011). The United States saw a 14% increase in production from 19 million tons in 2012 to 20.6 million tons in 2013. (Faostat, 2012)

Pretreatment and preconditioning provide promise for improving the pelleting process and products. A number of technologies such as torrefaction and steam explosion are being explored but they are energy intensive and provide mixed results (Biswas et al., 2011; Ciolkosz & Wallace, 2011). Gilbert et al. (2009) found that preheating switchgrass to a temperature of 75°C formed better pellets than those formed at room temperature, even at higher pressures. It was suggested that preheating softens the lignin, which acted as a binding agent (Gilbert, Ryu, Sharifi, & Swithenbank, 2009). Low temperature (<125°C) thermal preconditioning may be a less energy intensive and comparable pre-processing method for the production of pellets. In order to test and verify this idea, a quantitative evaluation of such thermal preconditioning effects are needed. Therefore, this study will focus on evaluating the effect of thermal preconditioning with dry steam on the mechanical properties of the formed pellets using ground switchgrass.
2. Literature Review

Herein, detailed review is presented of the literature relevant to the densification process and of previous work on mechanical properties of ground and densified biomass. The need for biomass densification is discussed, as well as the benefits of pelletizing material. The factors affecting the pelletizing process as well as the mechanical and physical properties of densified biomass and ground biofeedstock are identified. The metrics for quality assessment of densified material are reviewed as well.

The various methods of preconditioning, preprocessing, and pretreatment are also explored. Existing pretreatment methods and the effects of using steam are discussed. With the focus on steam conditioning, key findings are discussed to identify knowledge gaps and to better understand its effect on pelleting process.

2.1. Purpose of Densification

A major drawback to the utilization of biomass for heat and power production is its low bulk density. The density of most grasses is between 40 to 150 kg/m\(^3\) while the density of most woodchips is about 150 to 200 kg/m\(^3\) (Stelte et al. 2012b). For logistics such as transportation, volume is a much greater limiting factor than total weight. Since the bio-feedstock is generally transported from agricultural lands to industrial areas, the distance of transportation can be substantial. Low bulk density also is a logistical problem for material storage, its higher volume taking up more space and its often higher moisture content hindering long term storability. Densification increases the bulk density from the aforementioned 40-200 kg/m\(^3\) to 400-800 kg/m\(^3\), thereby increasing energy density as well (Clarke, Eng, & Preto, 2011).

2.2. The Compaction Process

Compaction is the process of reduction of the space between particles (voids) and increase in density. The limit of compaction is the density of the particle or the “true density”.

Compaction correlates to the change in magnitude of the applied pressure. This pressure is modeled as a combination of hydrostatic and deviatoric stress. Hydrostatic stress is uniform in all directions around a material. It can be visualized as the stress on a sphere when submerged in water. The result is a change in volume, termed dilation, but no change in shape. Deviatoric
stress (or stress deviator), which represents pure shear, is the opposite; it is the result of differing principal stress components which sum to zero (i.e., pure shear) and causes a change in shape. This shape change is the result of shear stresses.

A body’s response to stress is displacement or strain in the form of deformation. Both elastic and plastic deformations may occur. Elastic deformation occurs due to the stretching (or compressing) of atomic bonds and changes in internal energy. The reversible “bounce back” from elastic deformation hinders bonding and results in a less compact product. Plastic deformation is the major mechanism to increase contact area and bonding in particulate systems (Yates, 2013). Plastic strain occurs mostly due to the sliding of planes of atoms. The process is neither homogeneous nor reversible. Figure 2.1 shows perfect plastic behavior by the dotted line. The solid line is the real material behavior, i.e. elastoplastic response, and the arrows indicate the direction toward the ideal cases, namely perfectly elastic or plastic responses.

Figure 2.1 Force-displacement curve where A is compaction and B is decompression. Arrows indicate the desire from real (solid lines) to ideal (dotted line). [adapted from:(Yates, 2013)]

2.3. Types of Biomass Densification

Biomass densification involves the use of a mechanical force to compact biological materials into similarly sized solid items. Generally accepted size and nomenclature of densified products is somewhat unclear and can be confusing. Names such as pellets, briquettes, cubes, pucks, and wafers are not universally defined. This is partly because these technologies have been adapted from other industries; for example, pellet technology is primarily used by the animal feed industry (Thomas, van Zuilichem, & van der Poel, 1997). It is also important to note that biomass briquettes are distinct from coal briquettes, which are formed from a roller press and have a different shape (Clarke et al., 2011). ASABE standard S269.5 groups densified materials
by whether the ingredients are ground or unground before agglomeration (ASABE, 2012). In both cases, densification of biomass is achieved of feed materials.

2.3.1. Cubes and Unground Agglomerations

The ASABE defines a cube as agglomeration of unground ingredients and is not necessarily cubic in shape. By this definition cubes can also be referred to as briquettes, wafers, or pucks (ASABE, 2012). Regardless of name, these densified biomass forms are all significantly larger in size than pellets. Compacts most commonly called briquettes are somewhat large cylindrical compacts. They have diameters greater than 25 mm and are formed by extruding feedstock through a heated die with a piston or screw.

Compacts most commonly referred to as cubes are box shaped and are produced by a device known as a cuber. The cuber consists of an auger and a roller press that passes over square die openings. Cubes have a size range of 13–38 mm in cross section, and a length range of 25–102 mm. Images of each respective product are shown in Figure 2.2 and a more detailed comparison of densification technologies is provided in Table 2.1

![Images of pellets, briquettes, and cubes.](Clarke et al., 2011)

2.3.2. Advantage of Pellets

Pellets are defined as generally cylindrical agglomerations formed from ground material (ASABE, 2012). Densification by pelletizing can increase the bulk density of pellet *en masse* to approximately 700 kg/m³ (Sokhansanj & Turhollow, 2004), which is within the reported range of 400 and 800 kg/m³ mentioned in preceding section 2.1. Large-scale pellet mills often utilize a vertically oriented ring die and an eccentrically oriented roller. Smaller scale pellet systems utilize vertical rollers on a horizontal die.
Pelletization is popular because the process integrates easily into existing animal feed pellet production and handling systems. Especially, wood pellet production has shown strong increases both globally and in the United States. In the U.S., the production was approximately 7.5 million tons in 2013, a 14% increase from 2012. (Faostat, 2012). Strong growth has occurred within both the European and North American wood pellet markets (Faostat, 2012). Figure 2.3 shows the bulk density of biomass pellets compared to other densified materials, as well as compared to coal. The bulk density of pellets is the third highest (Figure 2.3) and is similar to the bulk density of coal (lignite) (Clarke et al., 2011).

![Figure 2.3 Bulk densities of densification technologies and coal (Clarke et al. 2011)](image-url)
Table 2.1 General summary and comparison of densification processes (Clarke et al., 2011; Stelte, Clemons, et al., 2012; Tumuluru & Wright, 2011)

<table>
<thead>
<tr>
<th>Item Produced</th>
<th>Pellet Mill</th>
<th>Piston Press</th>
<th>Cuber</th>
<th>Screw Press</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Pellets</td>
<td>Briquettes</td>
<td>Cubes</td>
<td>Briquettes</td>
</tr>
<tr>
<td>Optimum moisture</td>
<td>10-15</td>
<td>10-15</td>
<td>15-25</td>
<td>4-8</td>
</tr>
<tr>
<td>content of the raw material (w.b.%)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Particle Size</td>
<td>&lt;3</td>
<td>6-12</td>
<td>12-16</td>
<td>2-6</td>
</tr>
<tr>
<td>requirements (mm)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Addition of binder</td>
<td>Not required</td>
<td>Not required</td>
<td>Required</td>
<td>Not required</td>
</tr>
<tr>
<td>Dimensions (mm)</td>
<td>6-25 (diameter); 3-50 (length)</td>
<td>32 (diameter) x 25 (thick)</td>
<td>33x33 cross section and 25.4 to 101 (length)</td>
<td>Length: 1940 740 (w); 1310 (h) (smaller dies produce smaller extruded logs)</td>
</tr>
<tr>
<td>Wear of contact parts</td>
<td>High</td>
<td>Low</td>
<td>Low</td>
<td>High</td>
</tr>
<tr>
<td>Output from machine</td>
<td>Continuous</td>
<td>In strokes</td>
<td>Continuous</td>
<td>Continuous</td>
</tr>
<tr>
<td>Specific energy consumption (kWh/ton)</td>
<td>16.4-74.5</td>
<td>37.4-77</td>
<td>28-75</td>
<td>36.8-150</td>
</tr>
<tr>
<td>Through puts (ton/hr)</td>
<td>5</td>
<td>2.5</td>
<td>5</td>
<td>0.5-1</td>
</tr>
<tr>
<td>Unit density (g/cm³)</td>
<td>1.1-1.2</td>
<td>&lt;0.1</td>
<td>0.8</td>
<td>1-.4</td>
</tr>
<tr>
<td>Bulk density (g/cm³)</td>
<td>0.65-0.75</td>
<td>0.4-0.5</td>
<td>0.45-0.53</td>
<td>0.5-0.6</td>
</tr>
<tr>
<td>Maintenance</td>
<td>Low</td>
<td>High</td>
<td>Low</td>
<td>Low</td>
</tr>
<tr>
<td>Homogeneity of</td>
<td>Homogeneous</td>
<td>Not homogeneous</td>
<td>Not homogeneous</td>
<td>Homogeneous</td>
</tr>
<tr>
<td>densified biomass</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

2.4. Properties of Ground Biomass

Unlike the production of biomass briquettes or cubes, particle size reduction is a necessary step before the pelletization process. The comparatively smaller sized pellets require smaller particle size while briquettes and cubes can be formed from baled material (Karamchandani et al. 2015; Stelte et al. 2012b). Important as well are the properties of the material to be pelletized. Both the mechanical and compositional properties of the incoming material affect the outgoing product.
quality. Factors, such as choice of material, material properties, and mechanical properties, are discussed. For example, choice of material can greatly affect the quality of resulting pellets. While it may seem obvious that wood pellets are different than straw pellets, significant differences in pellet quality as well as pelleting behavior exist across similar species (Theerarattananoon et al., 2011; Tumuluru & Wright, 2011; Wilson, 2010). For this reason, it is necessary to characterize a material’s composition, basic properties, and mechanical properties if one is to engineer the biomass pellet process and achieve desired quality characteristics in a repeatable manner.

Raw biomass is composed of a number of micro and macro molecular substances. These include but are not limited to starch, protein, fat, cellulose, hemicellulose, and lignin. Both starch and protein are thought to undergo deformation at the higher processing temperatures of densification and form new bonds between particles (Kaliyan & Morey, 2010; Kaliyan & Vance Morey, 2009; Zeleznak & Hoseney, 1987). Feed material with higher proportions of starch and protein tends to produce better quality pellets than biomass with only cellulosic material (Sokhansanj, Mani, Bi, & Zaini, 2005).

Woody and herbaceous biomass is commonly characterized by the percentage of the macro molecular structures of cellulose, hemicellulose, and lignin. Table 2.2 shows the percentage composition of various constituents, the remaining percent consist of other minor components (not listed). Figure 2.4 shows the chemical structure of each of these substances. Cellulose is a polysaccharide composed of repeated β linked units of D-glucose. Hemicellulose is composed of a number of heteropolymers present along with cellulose in the plant cell wall. Lignin is a chemically complex structure most commonly found in wood, and is located in the cell wall between cellulose and hemicellulose (Albersheim et al., 2011). During densification, lignin is believed to act as a binder (Kaliyan & Morey, 2010).
Table 2.2: Typical lignocellulosic composition of selected biomaterials (Kaliyan & Vance Morey, 2009; Szczukowski, Tworkowski, Klasa, & Stolarski, 2002; Tumuluru & Wright, 2011)

<table>
<thead>
<tr>
<th>Item</th>
<th>% Composition</th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Celluloses</td>
<td>Hemicelluloses</td>
<td>Lignin</td>
<td></td>
</tr>
<tr>
<td>Salix (Willow)</td>
<td>55.9</td>
<td>14.0</td>
<td>13.8</td>
<td></td>
</tr>
<tr>
<td>Corn Stover</td>
<td>49.4</td>
<td>26.2</td>
<td>8.8</td>
<td></td>
</tr>
<tr>
<td>Switchgrass</td>
<td>43.8</td>
<td>28.8</td>
<td>9.2</td>
<td></td>
</tr>
<tr>
<td>Rice Straw</td>
<td>34.0</td>
<td>27.2</td>
<td>14.2</td>
<td></td>
</tr>
<tr>
<td>Birch Wood</td>
<td>40.0</td>
<td>25.7</td>
<td>15.7</td>
<td></td>
</tr>
<tr>
<td>Scots Pine</td>
<td>40.0</td>
<td>28.5</td>
<td>27.7</td>
<td></td>
</tr>
</tbody>
</table>

Note: variation is to be expected among cultivars, locations, growing conditions, etc.

Figure 2.4 Chemical Structures of (a) Lignin, (b) Hemicellulose, and (c) Cellulose (Wikimedia.org)

2.5. Mechanical Properties of Ground Biomass

Determination of the mechanical properties of ground biomass is important to better understand the relationship between ground material properties and the resulting pellet properties. Key parameters for developing this relationship include bulk modulus, compression index, spring-
back index, failure stress, shear modulus, loading rate, confining pressure, and unloading-reloading stress level (Kamath & Puri, 1997; Li & Puri, 2003). Table 2.3 provides a brief explanation of some of these variables. These properties can be determined and analyzed using a fundamental mechanical tester such as the Cubical Triaxial Tester (CTT).

Table 2.3 Explanation of fundamental mechanical parameters (Li & Puri, 2003)

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Unit</th>
<th>Significance</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bulk Modulus</td>
<td>N/m² (Pa)</td>
<td>Measure of the material’s resistance to volumetric deformation at a given isotropic pressure</td>
</tr>
<tr>
<td>Compression Index</td>
<td>- \frac{1}{\ln(Pressure)}</td>
<td>Quantifies compressibility of powder at a given isotropic pressure</td>
</tr>
<tr>
<td>Spring-back Index</td>
<td>- \frac{1}{\ln(Pressure)}</td>
<td>Quantifies powder’s ability to recover/relax after release of stress at a given isotropic pressure</td>
</tr>
<tr>
<td>Failure Stress</td>
<td>N/m² (Pa)</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>(Strength)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Shear Modulus</td>
<td>N/m² (Pa)</td>
<td>Measure of material’s resistance to change in shape at a given pressure difference in principal directions.</td>
</tr>
</tbody>
</table>

2.6. Cubical Triaxial Tester (CTT)

The Cubical Triaxial Tester allows for the measurement and study of true stress-strain behavior of particulate materials over a wide range of compression and extension conditions without the confounding effect of die-wall friction (Kamath and Puri 1997). The device consists of a chamber with six equal sized flexible boundaries that are able to be pressurized and depressurized. The flexible boundary CTT was developed for low and medium pressures.
Previously, it was used in the characterization of industrial powders but also has been used with ground biomass (Karamchandani, Yi, & Puri, 2015).

As mentioned in section 2.6, the CTT is used in the determination of mechanical properties of particulate materials. The device produces volumetric stress-strain curves, which are determined by measuring deformation changes of a powder system or granular sample for a pre-determined stress path. A typical pressure-strain data for switchgrass under a cyclic hydrostatic stress path is shown in Figure 2.5. The slope of this curve is used to determine the bulk modulus values. Further analysis of these stress-strain curves also provides the other aforementioned mechanical properties.

![Figure 2.5 A typical isotropic stress (Pressure) vs. volumetric strain response with a cyclic hydrostatic stress path (Karamchandani et al., 2015)](image)

2.7. Pelletization Process

Pelletization is achieved through the application of pressure from a mechanical force exerted by a roller overcoming the friction between ground biomass and die wall. Error! Reference source not found. depicts this process, where biomass is squeezed into a die by a roller which forces material to flow inward, causing elastic and plastic deformation which contribute to the compaction of the material (Kaliyan & Morey, 2010).
2.8. Properties of Densified Biomass

Once pellet formation has been completed, further testing is necessary to assess the quality of the product. This includes evaluating the pellet density, strength, and durability.

2.8.1. Pellet Density

Mani et al. (2006) found that individual pellet density of biomass ranges from 1,000 kg/m$^3$ to 1,200 kg/m$^3$. The bulk density of these pellets ranges from 550 kg/m$^3$ to 700 kg/m$^3$. A high bulk density is desired for ease and cost effectiveness of transportation, storage, and handling.

As shown in Table 2.4 Karamchandani et al. (2015) found untreated switchgrass had a single pellet density between approximately 700-1300 kg/m$^3$. Pellet density also did not significantly differ between the compared feedstock moisture contents or particle sizes (p>0.05) (Karamchandani et al., 2015).
Table 2.4 Summary of switchgrass pellet quality metrics (Karamchandani et al., 2015)

<table>
<thead>
<tr>
<th>Condition of Pellet Feedstock</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), column comparisons

2.8.2. Pellet Strength

There is no universally accepted standard for testing the strength of densified biomass (Kaliyan & Vance Morey, 2009). Part of the reason for this is that the term “pellet strength” can refer to compression strength, pellet hardness, or impact resistance.

Compression strength, also termed pellet hardness, is defined for this study as the force at breakage when a pellet is placed between two metal plates and compressed at a fixed strain (or displacement) rate while force and displacement are recorded (Stelte et al. 2012b). A universal testing machine (Instron model 3345, Norwood, MA) is suitable for this test. Applied force and displacement are recorded until the pellet breaks. Defined as Newtons, the force at fracture is recorded as the compressive strength of the pellet. The terms diametral and axial indicate the orientation of compressive testing. Diametral testing occurs along the pellet’s curved surface and axial testing is along the pellet’s axis. Karamchandani et al. conducted both axial and diametral strength tests of switchgrass pellets at two different values of both moisture content and screen size (Karamchandani et al., 2015). The results are shown in Figure 2.7 and Figure 2.8. The results noted a trend that switchgrass ground at the lower grind size of 3.175mm may form stronger pellets. The results also showed that increased moisture content may cause a decrease in pellet strengths. Both of these trends were not statistically significant (p>0.05)
2.8.3. Pellet Quality Standards

The Pellet Fuels Institute (PFI), a nonprofit association in the pellet industry, has a set standard for densified fuel. Three levels of fuel grade pellets exist, premium, standard, and utility. To meet the minimum standards, minimum or maximum thresholds of bulk density, diameter, pellet durability index (PDI), fines, ash content, length, moisture content, and chloride must be met. Notably, bulk density must be greater than 608 kg/m³, the PDI must be greater than 95%, and moisture content must be less than 10% (PFI, 2011).

2.8.4. Pellet Durability

During production, transportation, handling, and storage, pellets are subject to varying mechanical loads. For example, during product distribution, pellets are loaded onto trucks, subjected to vibrations and movements of travel, and/or transported in and out of bins and
boilers. It is important for pellets to retain their shape and density to maintain flowability and ease of handling. The gain in bulk density from pelleting is of reduced value if the pellet cannot hold its shape.

Durability is the parameter used to quantify and evaluate if a pellet can hold its shape. According to ASABE standard S269.5 (ASABE, 2012), durability is measured using the pellet durability index. The PDI is determined by tumbling a 500 g sample for 10 minutes in a dust tight container at 50 rpm. The box is of a specified size with a baffle in the middle. An example of a tumbling tester is shown below in Figure 2.9. ASABE standard S269.5 defines durability as the initial mass of pellets minus the material lost in tumbling divided by the initial mass multiplied by 100%. This is expressed in Equation (1).

\[ PDI = 100 \left( \frac{M_f}{M_i} \right) \]  

(1)

where \( PDI \) is pellet durability index as percent, \( M_i \) is initial mass of pellets, and \( M_f \) is final mass of pellet.

Studies offer a variety of different recommendations for durability standards. Kaliyan and Morey (2009) recommends a minimum 96% durability for feed pellet mills. Colley et al. considers durability high above 80%, low below 70%, and medium when in between (Colley, Fasina, Bransby, & Lee, 2006). Wilson argued that the Dural tester (Shahab Sokhansanj & Crerar, 1999) was a better device for measuring durability. The cited reasons include a greater range of results, smaller required sample size, and shorter test time (Wilson, 2010). The device operates at 1600 rpm and processes a 100 g sample in 30 seconds. Karamchandani measured pellet durability using a friabilator. A friabilator tumbles a 50 g sample of pellets at 55 rpm for ten minutes in a dust tight enclosure. Five replications were used (Karamchandani et al., 2015). For all durability measurements, the sample was sieved both before and after treatment before measuring the mass of the sample. In this way fines and crumbles of the same size were sifted out. The reported durability of switchgrass ranged from 87.7% to 93.5% (Karamchandani et al., 2015). Hilton et al. (2013) developed a subjective pellet quality standard where the operator judges the pellet quality on a scale of 1-10. The scale positively correlates to the ASABE durability standard S269.5 and provides a rapid way to assess results (Hilton et al., 2013).
2.9. Factors Affecting Pelletization

Several key process parameters that affect pelletization have been identified. These include particle size and shape, moisture content, and temperature. All of these have been shown to influence pellet quality.

2.9.1. Moisture Content

Moisture content plays an important role in the pellet formation of biomass as noted by several studies (Gilbert et al., 2009; Kaliyan & Vance Morey, 2009; Karamchandani et al., 2015; Nielsen et al., 2009). The optimum moisture content varies from species to species, but in general the optimum level is 5 to 10% wet basis for woods and 10 to 20% for grasses (Karamchandani et al., 2015; Stelte, Sanadi, et al., 2012). Moisture content above the optimum leads to increased clogging of the die and increases energy consumption. Additionally the magnitudes of mechanical properties such as strength and durability (Kaliyan & Vance Morey, 2009) are reduced. Similarly material that is too dry has poor mechanical properties presumably because moisture plays a role in the intermolecular binding.

Figure 2.9 Image of tumbling box fabricated in the Agricultural and Biological Engineering Department according to ASABE standard S269.5 specification to be used to test pellet durability.
2.9.2. Die Temperature

Die temperature has been shown to play a significant role in biomass densification (Gilbert et al., 2009; Nielsen et al., 2009; Rhén, Gref, Sjöström, & Wästerlund, 2005; Stelte et al., 2011b). Heat is generated from the friction between the biomass and the die. The average die temperature in these studies is approximately 90°C while the temperature of the exiting biomass is usually about 70°C (Stelte et al. 2012b). It has also been found that an increase in die temperature decreases friction in the press channel and also reduces energy consumption (Stelte et al. 2011a). In woody feedstock, this has been attributed to the migration of tall oil (rosin oil) to the surface of the press channel. The oil is believed to lubricate the die and reduce the pressure build-up in the pellet press (Stelte et al., 2011a).

2.9.3. Thermal Activation of Natural Binders

The addition of heat can soften or melt naturally present chemicals in biomass and allow them to act as glue that binds adjacent particles. The glass transition range is the point where the material becomes soft enough to move yet does not fully flow as a melted compound would. This range varies with moisture content but Zeleznak and Hoseney (1987) identified the glass transition of starch to be around 30°C and the onset of melting was approximately 80°C. The melting of the amyllose-lipid complex correlated to 100°C (Zeleznak & Hoseney, 1987). Reports for glass transition range of lignin vary, especially with regards to material type and moisture content. At 8% moisture content, the glass transition range was estimated to be 53°C-63°C for wheat straw and as high as 91°C for spruce (Stelte et al. 2012a; Stelte et al. 2012b).

Studies have also suggested that the glass transition of lignin at higher temperatures contributes to the hardening and improved mechanical properties of formed pellets (Gilbert et al. 2009; Kaliyan and Morey 2009; Stelte et al. 2011b). Gilbert et al. (2009) found that switchgrass/tar pellets formed at 166 bar (16.6 MPa) and 75°C had better density and tensile strength than a pellet formed at room temperature and 552 bar (55.2 MPa).

2.9.4. Particle Size

The size to which biomass is ground does play a significant role in the densification process. Material undergoes size reduction through the use of a size reduction device such as a knife-mill
and a screen controls the resulting particle size distribution. Due to an increase in specific surface area, friction in the pelletizer increases with decreasing particle size (Kaliyan & Vance Morey, 2009; Mani, Tabil, & Sokhansanj, 2006; Stelte et al., 2011a). Kaliyan and Morey (2009) also found that higher density briquettes were made from smaller particle size material. However, a study by (Serrano, Monedero, Lapuerta, & Portero, 2011) found that the opposite was true and bulk density actually decreased.

Karamchandani et al. (2015) conducted tests using corn stover, switchgrass, and willow particles ground at 6.35 mm and 3.175 mm screen sizes. It was found that particle size did not have a significant (p>0.05) effect on axial compressive strength or diametral tensile strength. Although pellets formed from the smaller screen size did have higher values, they were not significantly different (p>0.05). It was also found that spring-back index values slightly increased for the larger screen size at 95 kPa. Additionally, it was found for switchgrass pellets screen size did not have any significant effect (p>0.05) on pellet density or durability (Karamchandani et al., 2015). These findings are summarized in Table 2.4.

The quantity of fines (particles less than 0.5 mm in diameter) must be considered during pellet production as well; too large a fraction of fines negatively affects pellet quality and increases die friction. Stelte et al. (2012b) recommends the quantity of fines not exceed 10.0 to 20.0% unless a binding agent is added. Such agents are added during a preprocessing step.

2.10. Densification Preprocessing and Preconditioning

Generally, if resulting pellets are of an undesirable quality, changes to the incoming material can be made to improve the final product. For the purpose of this review, a preconditioning process is considered to be any step taken before pelletization with the intent of improving the quality of resulting pellets. This may be as simple as the inclusion of additives or as complex as steam explosion where the structure and appearance of the material are affected.

2.10.1. Additives

Occasionally, additives (additional materials such as binders or lubricants) may be added to the pellet process to improve the mechanical property of pellets. Typical additives for wood pellets researched include lignosulphonate, dolomite, starches, vegetable oil, and motor oil. The general
quantity for additives is between 1-3% by mass of the incoming material (Tumuluru & Wright, 2011). Depending on the material, binders may be used to improve strength, density, or durability. Lignosulphonates and starch have been shown to improve pellet durability, but decrease calorific value (Tarasov, Shahi, & Leitch, 2013). Lubricants reduce friction in the die, and are more commonly added for hardwood pellet production rather than softwood. Vegetable oil is the most common lubricant (Stelte, Sanadi, et al., 2012). The pellet fuels institute requires that any included additives as well as the type of material be included in quality marked products (PFI, 2011).

2.10.2. Steam Explosion

Steam explosion is a process in which a material is heated under high pressure using superheated 220°C steam, after which the steam pressure is quickly released. This causes the material to expand rapidly and undergo physical, chemical, and micro-structural changes. Zandersons et al. (2004) stated that steam explosion resulted in changes in cellulose structure and activation of lignin resulted in new bonds. These new bonds created through pretreatment are thought to improve pellet qualities.

Biswas et al. (2011) performed steam explosion on Salix (willow) to evaluate its effect on pellet properties. It was found that the treatment produced a nearly 100% durable pellet compared to the conventional pellet (Biswas et al., 2011). The abrasion and impact resistance were attributed to the melting of lignin on the surface of the pellet during pelletization (Biswas et al., 2011). Tooyserkani et al. (2012) performed a sulfur dioxide catalyzed steam treatment on spruce, Douglas fir, pine, and Douglas fir bark, and found that mechanical strength increased twofold, but the resulting pellet density was lower than untreated pellets.

2.10.3. Torrefaction

Torrefaction is the heating of biomass between 200°C to 300°C under zero oxygen conditions. It produces a dark, coal-like pellet that has reduced mass but also slightly reduced energy yield. As Ciolkosz and Wallace (2011) noted, the process has both positive and negative effects. The resulting product has a lower oxygen content, less hydrophilic, and higher caloric value than untreated biomass (Stelte et al. 2012b). It also requires as much as 50% less energy to pelletize,
yet also had a reduced strength (Bergman & Veringa, 2005; Ciolkosz & Wallace, 2011; Gilbert et al., 2009).

2.10.4. Thermal Preconditioning with Steam

The addition of steam before pelleting is common practice in both the feed and wood pellet industries. The feed industry uses pressurized steam conditioners to improve the hardness and density of feed pellets (Larsson, Thyrel, Geladi, & Lestander, 2008). Steam conditioning has been shown to improve pellet properties for both feed pellets and wood pellets (Boussaid, Esteghlalian, Gregg, Lee, & Saddler, 2000; Cutlip et al., 2008). Cutlip et al. (2008) found that conditioning feed material with steam led to an increase in pellet quality. Steam conditioning also increases moisture content, a property well documented to improve pellet quality. Filbakk et al. treated different types of softwood with 2 kg/hr of steam and 6 kg/hr of steam and found positive results from the increased steam (Filbakk, Jirjis, Nurmi, & Høibø, 2011).

Steam explosion and torrefaction are much more expensive than steam preconditioning, requiring specialized devices. These methods are also much more energy intensive than simple thermal preconditioning. Thermal preconditioning has the benefit of being cheaper and easier to implement and is the preprocessing step selected for this research.

2.11. Evaluation of Thermal Preconditioning with Steam on Switchgrass Pellet Quality

Research has been done on the effect of steam explosion pretreatment on pelleting (Biswas et al., 2011; Tooyserkani et al., 2012), however, both studies used a cylindrical piston press rather than a roller-die pelletizer. It was shown that similar studies had opposite results due to this difference in equipment (Stelte et al. 2012b). Lignin melts at a much lower temperature than the 220°C achieved by steam explosion (Tumuluru & Wright, 2011), and it is possible that similar success can be attained without steam explosion. Previous work has been done with the CTT to study compaction mechanics of untreated ground material (Li & Puri, 2003). Exploring the differences between the stress-strain behaviors of pretreated and control biomaterial will give a better picture of the effect of steam pretreatment, and to a lesser extent, the overall densification process. Thermal preconditioning is a process often used to improve quality in wood. It is used less frequently with regard to pelleting of bioenergy crops. Previous research studies have concluded
that elevated temperatures contribute to improved pellet qualities by the hypothesized softening of lignin and other natural binders.
3. Goal, Hypothesis, and Objectives

3.1. Goal
The goals of the proposed research are to utilize thermal preconditioning with dry steam and quantify the effects at the lab scale based on the metrics of density, strength, and durability.

3.2. Hypotheses
Hypothesis 1:

\( H_0 \): The dry steam conditioning of ground switchgrass prior to pelletization has no significant effect on pellet density at an \( \alpha \) of 0.05.

\( H_A \): The dry steam conditioning of ground switchgrass prior to pelletization has a significant effect on pellet density strength at an \( \alpha \) of 0.05.

Hypothesis 2:

\( H_0 \): The dry steam conditioning of ground switchgrass prior to pelletization has no significant effect on pellet axial (and diametral) strength at an \( \alpha \) of 0.05.

\( H_A \): The dry steam conditioning of ground switchgrass prior to pelletization has a significant effect on pellet axial (and diametral) strength at an \( \alpha \) of 0.05.

Hypothesis 3:

\( H_0 \): The dry steam conditioning of ground switchgrass prior to pelletization has no significant effect on pellet durability at an \( \alpha \) of 0.05.

\( H_A \): The dry steam conditioning of ground switchgrass prior to pelletization has a significant effect on pellet durability at an \( \alpha \) of 0.05.
3.3. Objectives:

To test those hypotheses, relevant physical properties of ground switchgrass and pellets should be determined and analyzed. Therefore, the objectives of this research are to:

1. Measure and quantify the physical properties of ground switchgrass at different steam-treated time-temperature conditions, including an untreated as control, and
2. Measure and quantify the physical and mechanical properties of formed pellets at different steam-treated time-temperature conditions, including an untreated as control.
4. Methodology

4.1. Overview and Flowchart of Methodology

This chapter explains the design and execution of this project to achieve the goals and objectives and test hypotheses. A flow chart detailing the entire process is shown in Figure 4.1. Preconditioning design specifications, as well as the source and planned material are listed. The intended equipment for respective experiments or measurements, as appropriate, is discussed. The overall methodology is organized into six phases, which are described below.

**Phase 1:** Phase 1 involves assessing viability of an autoclave (Model 902146501 Honey Brook, PA) as a preconditioning device. Preliminary tests were conducted using the autoclave in order to assess the effect on the increased moisture content, material temperature, and temperature distribution of ground biomass samples. Thermal conditioning time and the logistics of maintaining material temperature during bulk property measurement were also assessed.

**Phase 2:** Switchgrass was prepared and characterized for preconditioning and pellet formation in this phase. A shear cutting-based Munson SCC-10 (Utica, NY) size reduction machine ground the material into finer particles. At this time, physical properties of the untreated (control) material were measured. This includes determination of the material’s bulk density, moisture content using ASABE standard S358.2 (ASABE 2006), and particle size distribution using a sieve shaker.

**Phase 3:** At this stage, prior to dry steam conditioning, the ground switchgrass was preconditioned by adding water to attain moisture content of 17.5% and 20.0% (w.b.). A pycnometer (Quantachrome Instruments, MVP 2, Serial # 1149606701, Boynton Beach, Florida) with helium was used to determine the particle density. Bulk density and moisture content were determined as well. Pellets were then formed from both steam-treated and untreated materials. Pelletization was performed using a Pellet Pros® farm-scale pellet mill (Model VI84TTFB4026AA, Dubuque, IA).

**Phase 4:** In phase 4, the formed pellets were evaluated for density, strength, and durability. Pellet density was calculated by measuring the diameter and length of pellets using Vernier calipers (Mitutoyo, CD-6”CS, Aurora, IL). A universal testing machine (Instron model 3345,
Norwood, MA) was used to measure diametral tensile and axial compressive pellet strengths. Durability was determined using ASABE Standard S269.5 (ASABE 2006) and the friability tester.

**Phase 5:** Phase 5 focused on interpreting the data, analysis, and testing of hypotheses 1, 2, and 3 at significance level \( \alpha = 0.05 \).

![Flowchart of methodology](image)

Figure 4.1 Flowchart of methodology

4.2. Materials and Equipment
Baled switchgrass was the source material for these experiments. Switchgrass is faster growing and can be considered more renewable than most woody materials. It was grown in Julian, PA and the cultivar of the switchgrass is “Cave in Rock”. The material was ground in a size reduction machine, subjected to dry steam preconditioning, and then pelletized in a farm-scale
pellet mill. Mechanical properties of pre-pelletized and post-pelletized material were determined using a universal testing machine, pycnometer, as appropriate. These instruments were all available in the Department of Agricultural and Biological Engineering (ABE). Thermal preconditioning was conducted with an autoclave also available through ABE.

Figure 4.2 Switchgrass material, before (a) and after (b) size reduction using 6.35 mm screen size.

4.3. Experimental Design

Switchgrass was analyzed at three different steam temperature values, two moisture contents, and one screen size (Table 4.1) for a total of six treatments. During particle evaluation, a group considering the unconditioned, dry material was also included. Based on preliminary tests, limitations of the autoclave settings, and the transition ranges of natural binders, temperature levels of 100°C and 120°C were selected. The proceeding sections provided the rationale for the moisture contents and screen size used in this study. The different temperature levels were controlled by temperature settings using the Hard Goods cycle within the autoclave.

Table 4.2 shows the number of replications per treatment with each testing apparatus. Treatment replications were used 500 g to allow for sufficient testing and pelletization.
Table 4.1 Design of experiment

<table>
<thead>
<tr>
<th>Variables</th>
<th>Number of levels</th>
<th>Levels</th>
</tr>
</thead>
<tbody>
<tr>
<td>Screen Size (mm)</td>
<td>1</td>
<td>6.35</td>
</tr>
<tr>
<td>Moisture Content</td>
<td>2</td>
<td>17.5% w.b.</td>
</tr>
<tr>
<td></td>
<td></td>
<td>20.0% w.b.</td>
</tr>
<tr>
<td>Time and Temperature (°C)</td>
<td>3</td>
<td>Ambient/Untreated</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Five-minute treatment at 100°C (steady state)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Five-minute treatment at 120°C (steady state)</td>
</tr>
</tbody>
</table>

Table 4.2 Experimental plan for replications

<table>
<thead>
<tr>
<th>Property/test</th>
<th>Apparatus</th>
<th>Replicates</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>Preconditioning</td>
<td>Autoclave</td>
<td>5</td>
<td>Literature review and Preliminary runs</td>
</tr>
<tr>
<td>Densification</td>
<td>Pelletizer</td>
<td>5</td>
<td>Literature review and Preliminary runs</td>
</tr>
<tr>
<td>Particle Density</td>
<td>Pycnometer</td>
<td>5</td>
<td>(Karamchandani et al., 2015)</td>
</tr>
<tr>
<td>Bulk Density</td>
<td>Container of known volume</td>
<td>5</td>
<td>(Karamchandani et al., 2015)</td>
</tr>
<tr>
<td>Particle Size Distribution</td>
<td>Sieve shaker</td>
<td>3</td>
<td>(Karamchandani et al., 2015)</td>
</tr>
<tr>
<td>Durability</td>
<td>Durability tester</td>
<td>2</td>
<td>ASABE S269.5</td>
</tr>
<tr>
<td>Axial Compressive Strength</td>
<td>Universal Testing Machine</td>
<td>10</td>
<td>(Karamchandani et al., 2015)</td>
</tr>
<tr>
<td>Diametral Tensile Strength</td>
<td>Universal Testing Machine</td>
<td>10</td>
<td>(Karamchandani et al., 2015)</td>
</tr>
</tbody>
</table>

4.4. Material Processing

4.4.1. Size Reduction

The switchgrass was ground using a Munson SCC-10 shear mill (or knife mill). Ground material passing through the 6.35 mm screen size was collected as test material. The 6.35 mm size was
chosen based on previous studies (Karamchandani et al., 2015). A fan and dust collector (Cincinnati Fan, 50S/TI, Mason, OH) were used to collect excess fines from the process. These fines were discarded and only the material gathered in the first chamber of the system was used. It has been noted that too high concentration of fines on pellet quality (Stelte et al. 2012b). Figure 4.3 shows a picture of the apparatus.

![Figure 4.3](image.png)

**Figure 4.3** Size reduction machinery (Munson SCC-10) and particle collector (Cincinnati Fan, 50S/TI)

### 4.4.2. Moisture Preconditioning

Prior to dry steam preconditioning, the material’s initial moisture content was determined through oven drying of samples of switchgrass following the ASABE standard 358.3 (“Moisture Measurement — Forages,” 2012). The initial moisture content was then calculated and with Equation 2 was used to determine the quantity of water to be added to attain 17.5% and 20.0% (w.b.) moisture content. Subsequent testing of conditioned material revealed actual moisture contents at the time of testing was closer to 16% and 18% (w.b.), respectively. Equation 2 was derived by a mass balance equation of water in the material.

\[
M_w = \frac{M_d (mc_d - mc_0)}{1 - mc_0}
\]

where \(M_w\) is mass of water added (kg), \(M_d\) is desired mass (kg), as explained below, \(mc_0\) is initial moisture content, fraction (w.b.), and \(mc_d\) is desired final moisture content, fraction (w.b.).
Water was added by pouring the determined mass of water ($M_w$) into a bucket and mixed using a manual Mini-Inversina (Bioengineering, AG, Switzerland). The device is capable of mixing material in three dimensions, rather than in two dimensions along an axis. Due to the small container size, the material was mixed piecemeal at approximately 80 rpm for a total of two minutes each to ensure uniform mixing. This is consistent with past methodology involving the Mini-Inversina (Karamchandani et al. 2015). In each run, approximately 500 g of material was prepared. The material was then left in a sealed container for a period of 24 hours for moisture to diffuse uniformly throughout the sample.

4.4.3. Preconditioning with Dry Steam

Preconditioning with dry steam utilized the Hard Goods Cycle in an autoclave (M902146501, Beta Star, Honey Brook, PA) at different time and temperature settings. The Hard Goods cycle is typically used for the surface sterilization of equipment with steam. In this study, the ground switchgrass was spread in a thin, even layer of 1 cm thickness for steam exposure. This was the smallest bed depth to produce a treated batch of 500 g of material required for making sufficient quantity of pellets for subsequent quality assessment. Sample temperature was measured using a set of ten thermocouples and a Midi data logger (Graphtec, GL450, Irvine, California). The autoclave was set to a five-minute exposure time at the target, steady value temperature (100°C or 120°C) with a minimum ramp-up and cool down time (Figure 4.4). The five-minute hold time was chosen based on preliminary tests. The moisture content changes recorded during the five-minute treatment time showed a 2% decrease. Dry steam treatment temperatures of 100°C and 120°C were based on the literature review target of glass transition temperatures of natural binders present in the material. These temperatures represent values within and above the glass transition range of lignin (Uslu, Faaij, & Bergman, 2008).

Prior to steam preconditioning, moisture content of the entering material (17.5% or 20.0% w.b.) was adjusted using the methodology described in section 4.4.1. Approximately 500 g of material was split equally among three trays to an approximately even and level distribution. The three trays in the autoclave were oriented with two vertically (i.e., long dimension aligned front-back) in the back and one horizontally (i.e., long dimension aligned left-right) oriented near the middle. The layout of these trays can be seen in Figure 4.5.
Figure 4.4 Hard Goods Cycle held for 5-minute exposure time of target 100°C steady state temperature.

Figure 4.5 Layout of steam trays in autoclave
4.5. Physical Properties of Ground Switchgrass

The following section details the calculations of particle size distribution of the ground feedstock, as well as particle and bulk density. The particle size distribution was determined following the method from ASABE standard S319.3 (ASABE 2006). A 100 g ground biomass sample was used in a Ro-tap sieve shaker for 15 minutes using U.S. Standard Sieve numbers 5, 7, 10, 14, 18, 25, 35, 45, 60, 80, 120, 170, and 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, and 0.062 mm, respectively) for this analysis.

After the sieving was performed, the mass of each sieve with the retained material was measured and the retained sample weight was analyzed following ASABE standards. Three replicates were performed for each aforementioned moisture content condition. This was to ensure that the measured values were within the 95% confidence interval, and was verified in the following chapter.

A pycnometer (Quantachrome Instruments, MVP 2, Serial # 1149606701, Boynton Beach, FL) was used to measure the particle density of the ground material using ultra-pure helium. The particle density was calculated using the known volume of a reference cell and pressure drop due to the expanded volume with the addition of the volume of the sample cells. The volume of particles is calculated with the ideal gas law with respect to the pressure and volume measurements as expressed by the Equation 3 below:

\[ V_p = V_c - V_r \left( \frac{P_1}{P_2} - 1 \right) \]  

where \( V_p \) is the volume of the sample cylinder, \( V_c \) is the volume of the sample cell and is given by the manufacturer, \( V_r \) is the volume of the reference cell and is also provided by the manufacturer, \( P_1 \) is the gage pressure reading after pressurizing the reference cell, and \( P_2 \) is the gage pressure reading after including the sample cell in the pressurized circuit.

The bulk density was measured but spooning the material into a container of known volume. The mass of the filled container was then measured to calculate the mass of the feedstock.
4.6. Pellet Formation

Pellets were formed using a Pellet Pros (Model PP220, Dubuque, IA) farm-scale, batch pelletizer. It uses a 5 horsepower (3.7 kW) motor and consists of a 75 mm diameter roller and 150 mm diameter flat plate die. The die is 25 mm thick and has 6 mm diameter die holes, resulting in aspect ratio of 4.17. The inlet taper is 5 mm deep and has an angle of 22°, and the exit taper is 1 mm deep. The pelletizer was run at half speed, i.e. a frequency controller was used at 30 cycles per second. Figure 4.6 shows an image of this machine. The screws holding the rollers were hand tightened and then an extra quarter turn was added.

From the literature review, it was decided to use a 150 g, 70% Distiller's Dried Grains with Solubles (DDGS) and 30% switchgrass pre-mix to establish a consistent die-flow before pelletization (Ciolkosz et al. 2013). Pellets from the initial mixture are captured in a separate bucket and switched over to a sample collection bin when it becomes visually clear that the yellow DDGS is no longer present in the exiting pellets.

Before each usage, the pelletizer is cleared of any previously contained material by unscrewing the hopper, removing the roller and manually clearing the die with a hammer and cylindrical punch. Initial die temperature is measured then the test sample transferred from the autoclave into the running pelletizer by pouring. Material from two trays is combined into one tray and poured into the hopper of the pre-clogged, running pelletizer. Adding the third, and final, tray of treated material then follows this. Combining trays in the initial transfer maintains a better process flow, as confirmed with preliminary testing. The transfer from autoclave to pelletizer is completed in approximately three minutes. Material transfer into the pelletizer for the control group did not use trays but was poured from a bucket into the hopper. Resulting pellets were collected in a small bucket beneath the pelletizer. Pellets were placed in an air-conditioned lab at 20.0% relative humidity and approximately 20°C to cool for 24 hours.
4.7. Pellet Properties

Pellet quality was analyzed with a tumbling box device, a friabilator, and a universal testing machine. Electronic calipers were used for measuring pellet length and diameter. Following ASABE standard S269.5 (ASABE, 2012) the durability was tested using a tumbling box. A picture of the constructed device is shown in Figure 2.9. Additionally, durability was also measured using a friabilator, which can be seen in Figure 4.7. The friabilator allows for smaller sample sizes to be used, but is more gentle on the pellets than the box test. Both devices were used for comparison to previous studies. Last, pellet strength was measured using an Instron universal testing machine (Instron model 3345, Norwood, Massachusetts).
4.7.1. Single Pellet Density

After cooling for at least 24 hours, twenty pellets were randomly selected at each condition and filed along the ends. This sample size was chosen to ensure a 95% confidence interval. This interval was verified in the Results and Discussion (Chapter 5). These pellets were then assumed to be cylindrical and their volume was calculated by measuring their length and diameter with an electronic caliper accurate to within +/-0.01 mm (Mitutoyo, CD-6”CS, Aurora, IL). The diameter was taken as an average measured across the width of the pellet near the top, center, and bottom. Mass of the pellet was then measured to within +/-0.001 g and density was calculated.

4.7.2. Pellet Strength

Axial and diametral strengths were measured with a universal testing machine (Instron model 3345, Norwood, MA). Applied force and displacement were recorded until the pellet broke. A compressive loading was applied at a constant speed of 0.5 mm/min to maintain a quasi-static state. The force at fracture was recorded as the compressive strength of the pellet. Diametral tensile strength was measured by compressing the pellet along its curved surface and axial compressive strength by compression along the axis.

Twenty filed pellets were also used at each condition for strength testing. To ensure that values are within a 95% confidence interval, ten pellets were selected for diametral strength testing and ten for axial strength testing, giving ten replications for each test. This interval was verified in the Results and Discussion (Chapter 5).
Equation 4 was used to calculate the diametral tensile strength. A schematic for the diametral strength is shown in Figure 4.10a below. Similarly, Equation 5 was used to calculate axial strength and Figure 4.9a shows the schematic for the axial strength layout.

\[
\sigma = \frac{2P}{\pi LD} \tag{4}
\]

\[
\sigma = \frac{4P}{\pi D^2} \tag{5}
\]

Where \(P\) is the maximum load observed, \(L\) is the pellet length and \(D\) is the pellet diameter (average of three measured diameters along the length of each pellet).
4.8. Pellet Durability

Pellet durability was determined with two different methods, by using a tablet friability tester (Figure 4.7) and a tumbling box method described in ASABE standard S269.5 (Figure 2.9).

The friability test used five replicates of 50 g tumbled in a dust tight enclosure at 55 rpm for 10 minutes (Karamchandani et al., 2015)(Pandeya & Puri, 2013). A number 5 screen size (4 mm) sieve was used to separate mass retained after the process. Figure 4.7 shows an image of the friabilator used for this test.

The box durability test used a 500 g sample tumbled in a durability tester constructed according to ASABE standard S269.5. Pellets were tumbled in the box for 10 minutes before the remaining mass was separated with a number 5 (4 mm) sieve.

Durability for both tests was calculated using the Equation 6 below.

\[
\text{Durability} = \frac{\text{Mass of pellets before testing}}{\text{Mass of pellets after tumbling}} \times 100\%
\] (6)

4.9. Data Analysis:

The Instron outputs data into comma separated value files and all other data were collected in a lab notebook. MINITAB (Version 17.2, State College, PA) was used to perform hypotheses tests at 95% confidence level (\(\alpha=0.05\)).
The above methodology was used to best assess the relationship of thermal preconditioning with dry steam on switchgrass pellets. These methods were selected based on literature review, past research, and available equipment. Analysis of these results is presented in the following Results and Discussion chapter.
5. Results and Discussion

The physical properties of ground switchgrass, as well as the quality metrics of the formed pellets are examined. The results of tests on switchgrass ground with 6.35 mm screen and switchgrass pellets are shown below. Ground switchgrass was conditioned to 17.5% (w.b.) and 20.0% (w.b.) moisture content. Four metrics were used to evaluate the quality of pellets. The pellet strength was measured both in the axial and diametral directions. Pellet durability was evaluated using two different methods; the tumbling box test defined by the ASABE standard S269.5 (ASABE, 2012) and the friability test used by Karamchandani et al. (2015). A comparison of the pellet durability tests is also performed.

5.1. Physical properties of ground switchgrass

The values of particle density, bulk density, and physical properties of the ground switchgrass material before pelletizing are shown in Table 5.1 below. The moisture content values were selected based on preliminary runs and literature review (Karamchandani et al. 2015).

Table 5.1 Physical Properties of ground switchgrass

<table>
<thead>
<tr>
<th>Moisture Content (w.b) and Temperature</th>
<th>Particle Density (N=5) (kg/m³)*</th>
<th>Bulk Density (N=5) (kg/m³)*</th>
<th>Median Diameter (D₅₀) (N=3) (mm)*</th>
</tr>
</thead>
<tbody>
<tr>
<td>Unconditioned Control</td>
<td>1170.24 ± 38.52 a</td>
<td>130.62 ± 6.05 b</td>
<td>0.65 ± 0.02 a</td>
</tr>
<tr>
<td>17.5% (w.b.) Control</td>
<td>1177.50 ± 39.53 ab</td>
<td>122.22 ± 3.14 c</td>
<td>0.58 ± 0.02 bc</td>
</tr>
<tr>
<td>20.0% (w.b.) Control</td>
<td>1169.08 ± 36.50 ab</td>
<td>137.62 ± 4.90 ab</td>
<td>0.58 ± 0.01 bc</td>
</tr>
<tr>
<td>17.5% 100°C</td>
<td>1100.73 ± 43.12 b</td>
<td>139.23 ± 2.35 a</td>
<td>0.56 ± 0.01 c</td>
</tr>
<tr>
<td>20.0% 100°C</td>
<td>1126.91 ± 61.71 b</td>
<td>137.31 ± 3.84 ab</td>
<td>0.60 ± 0.02 abc</td>
</tr>
<tr>
<td>17.5% 120°C</td>
<td>1097.61 ± 16.90 b</td>
<td>121.55 ± 3.75 c</td>
<td>0.63 ± 0.04 ab</td>
</tr>
<tr>
<td>20.0% 120°C</td>
<td>1088.09 ± 50.59 b</td>
<td>139.44 ± 3.06 a</td>
<td>0.57 ± 0.01 c</td>
</tr>
</tbody>
</table>

* Same superscripts are not significantly different (p>0.05) while different superscripts are significantly different (p<0.05)
5.1.1. Particle Density

The results of the particle density test are shown in Table 5.1. From these results, moisture conditioning within the control group did not have an impact on particle density (p>0.05). This indicates that the absorption of water during material preconditioning does not have a measurable effect on the density of individual particles. Compared to the reported values of a similar study (Karamchandani et al., 2015), a similar decrease with the addition of moisture was noted and these values also were statistically different (p>0.05).

Switchgrass that was both moisture and dry steam conditioned had a statistically different particle density than unconditioned untreated switchgrass. Decrease in particle density was more pronounced in 120°C conditioning. In this case, the steam-conditioned sample particle density was reduced by almost 10%. This suggests that there can be volatile components that heated ground switchgrass loses during the steam conditioning. This could happen while voids are introduced due to binder migration as suggested (Stelte, Sanadi, et al., 2012).

5.1.2. Bulk density

The results of bulk density testing are shown in Table 5.1. Comparison of average bulk density is not similar to that of particle density. The bulk density data do not seem to correlate to either the moisture content or dry steam treatment. This is in contrast to the conclusions of Karamchandani et al. (2015) that moisture content did not have a significant (p>0.05) impact on bulk density. It is worth noting that the 17.5%-120°C steam conditioned material was observed to have visibly longer particles after size reduction. This difference in particle shape produced from size reduction may have contributed to the observed difference in bulk density at this treatment level.

The bulk density test is impacted by a number of additional factors compared to the particle density. Irregularities in particle shape, non-uniformity of particle sizes, and particle size distribution all may influence bulk density results.

5.1.3. Particle Size Distribution

Figure 5.1 shows the observed particle size distributions (mean values with N=3) amongst all treatment levels.
Figure 5.1 Mean particle size distributions for all treatment levels.

The observed particle size distributions were quite similar across all levels. The ground switchgrass conditioned at 17.5%-120°C shows to have more of larger particles than the rest of thermal treatment levels, again correlating to the noted difference in ground particle size for that particular batch of material. On the other hand, ground switchgrass without steam conditioning has finer particles Table 5.1 shows the mean and standard deviation values for median diameter $(D_{50})$. Differences between $D_{50}$ values are statistically different ($p<0.05$), however comparisons between variables and control groups do not seem to reveal any notable relationships. The observed differences do not seem to relate to changes in moisture content or the effects of dry steam preconditioning.

5.2. Pellet density

Average pellet density is given in Table 5.2. Mean pellet densities varied from 1010.73 to 1187.50 kg/m$^3$. The observed pellet densities were 100-200 kg/m$^3$ greater than pellet densities reported in a previous study (Karamchandani et al., 2015). This increase in pellet density can be
attributed to the refinement of pelleting methodology and procedure, including the additional pre-clogging step, as well as possible compositional differences between switchgrass used in this and the published study.

Table 5.2 Pellet Density

<table>
<thead>
<tr>
<th>Moisture Content (w.b) and Temperature</th>
<th>Pellet Density (N=20) (kg/m$^3$)*</th>
</tr>
</thead>
<tbody>
<tr>
<td>17.5% (w.b.) Control</td>
<td>1187.50 ± 39.53</td>
</tr>
<tr>
<td>20.0% (w.b.) Control</td>
<td>1059.08 ± 36.50</td>
</tr>
<tr>
<td>17.5% 100°C</td>
<td>1010.73 ± 43.12</td>
</tr>
<tr>
<td>20% 100°C</td>
<td>1026.91 ± 61.71</td>
</tr>
<tr>
<td>17.5% 120°C</td>
<td>1097.61 ± 16.9</td>
</tr>
<tr>
<td>20% 120°C</td>
<td>1028.09 ± 50.59</td>
</tr>
</tbody>
</table>

*No significant difference (p>0.05)

Figure 5.2 shows an interval plot of mean pellet density across all six formed pellet conditions. The lack of statistical difference among groups (p>0.05) indicates dry steam preconditioning did not affect pellet density. It should be noted that the differences between moisture content levels (2.5%) also did not seem to affect pellet density.
Figure 5.2 Interval plot of pellet density with 17.5% and 20.0% w.b. moisture content, for untreated and steam temperature of 100 and 120°C treatment condition

5.3. Pellet Strength
Mean diametral tensile strength ranged from 2.8-4.9 MPa, while mean axial compressive strength ranged from 5.2-6.1 MPa. It was also noted that the standard deviation for the axial strengths was greater than that of the diametral strengths. In some cases the standard deviation for the axial compressive strength approached 50%.

Table 5.3 shows the results of axial and diametral strength tests on formed switchgrass pellets. Mean values for both dry steam unconditioned 17.5% and 20.0% moisture content show no significant difference between the two for pellet strength (p>0.05). Due to the non-uniformity of the pelleting process, the number of replicates is ten to maintain confidence interval level.

Distribution of voids, micro- and macro-cracks within the pellet structure, and irregular curvature of pellets are all sources of variability during strength testing.
### Table 5.3 Pellet strength results

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Moisture Content (w.b. %)</th>
<th>Diametral Tensile Strength (MPa, N=10)*</th>
<th>Axial Compressive Strength (MPa, N=10) *</th>
</tr>
</thead>
<tbody>
<tr>
<td>Untreated Control</td>
<td>17.5</td>
<td>2.9 ± 0.7</td>
<td>5.2 ± 1.8</td>
</tr>
<tr>
<td></td>
<td>20.0</td>
<td>2.9 ± 0.8</td>
<td>5.6 ± 2.2</td>
</tr>
<tr>
<td>100 (for 5 minutes)</td>
<td>17.5</td>
<td>2.8 ± 1.1</td>
<td>5.4 ± 2.5</td>
</tr>
<tr>
<td></td>
<td>20.0</td>
<td>4.0 ± 1.9</td>
<td>5.5 ± 2.5</td>
</tr>
<tr>
<td>120 (for 5 minutes)</td>
<td>17.5</td>
<td>3.6 ± 0.9</td>
<td>5.3 ± 2.1</td>
</tr>
<tr>
<td></td>
<td>20.0</td>
<td>4.2 ± 1.4</td>
<td>6.1 ± 2.4</td>
</tr>
</tbody>
</table>

*No significant difference (p>0.05)

Previous tests (Karamchandani et al. 2013) also show no significant difference for pellet strength at different moisture contents. It is worth noting that the average pellet strengths were higher by at least 1.0 MPa (25%) across all categories than previously reported values. This again, similar to the pellet density findings, suggests improvement in pellet formation techniques and/or material composition and property differences.

5.3.1. Axial compressive strength

Figure 5.3 shows similar confidence intervals for all observed variables. Figure 2.7 shows that the axial compressive strengths reported by Karamchandani et al. (2015), exhibit high standard deviations for mean values. Statistical analysis indicates that neither time-temperature steam treatment had a significant effect (p>0.05) on the axial strength of the formed pellets.
Figure 5.3  Confidence interval (95%) plot of pellet axial compressive strength with 17.5% and 20.0% (w.b.) moisture content, for untreated and steam temperature treatments of 100 and 120°C (N=10).

The large variance of axial strength test data is likely related to the limitations of the test sensitivity as well as how the pellets were formed. The filed pellets’ deviation from the idealized cylindrical shape likely increased the variance in measured results. For the pellets used in strength and density observations, only pellets strong enough to be filed were selected. The filing was necessary for accurate length measurements, and meeting the required assumption of the pellet sample being cylindrical for the strength calculation. Additionally, filing the ends for proper alignment is necessary to ensure application of pure axial force and determine the true compressive strength.

The ability of the pelletizer roller to apply stress in the axial direction is related to the amount of material in front of and behind it. Therefore, during pellet formation, there exists dynamic variation in axial stress (Yates, 2013), which also likely contributed to the pellet to pellet variability. The use of a pre-mix helps provide consistency in the flow of material. There is also
a possible stress variation due to differences in applied forces within the pelletizer particularly in the radial direction. This is noticeable when cleaning out the die as material in the outer rim is more difficult to hammer out than material in the middle and inner die holes for the pelletizer used in this study. These factors may contribute to some pellets being of much higher strength than the others.

These previously mentioned contributing factors may all affect pellet strength. Considering the range of pellets produced, it may be meaningful that the strongest pellets in the control groups were less strong than those in three of the four dry steam groups (17.5%-100°C; 20.0%-100°C, 20.0%-120°C). It is possible that the dry steam preconditioning softened the natural binders enough to produce a stronger pellet, but the high variance of the test might have masked the results.

5.3.2. Diametral strength

![Diametral strength graph](image)

Figure 5.4 95% confidence interval plot of pellet diametral tensile strength with 17.5% and 20.0% w.b. moisture content, for untreated and steam treatment at 100 and 120°C.
Figure 5.4 shows the 95% confidence interval plot comparison for the mean of diametral tensile strengths across moisture content and thermal conditioning levels. Student’s t-test indicates no significant difference at $\alpha = 0.05$, but, at the 120ºC temperature treatment, the diametral strengths for the 17.5% and 20.0% are approximately 0.5-1 MPa higher than the diametral strengths of the unconditioned and 100ºC treatments. Perhaps, at higher temperature treatments, the diametral strength will increase even more. Stelte et al. (2012b) discussed that the tall oil in woody biomass migrates to the outer surface of the pellet during the pelleting process. The migration of additional binders to the outside of the pellet may explain the observed increase in strength at the 120ºC. This increase may have been observed in the axial direction but was masked by the relatively lower sensitivity of the test. The strongest pellets observed pellets were at the 120ºC 20.0% (w.b) treatment level.

5.4 Durability

As given in the Methodology chapter, pellet durability was determined using two different methods: the tumbling box method described in ASABE standard S269.5 (ASABE, 2012), and the friabilator method used by Karamchandani et al. (2015). Mean friability values ranged around 93% while mean box durability values were 86.6% for the control group and 77.9% for the dry steam treatment group.
Table 5.4 Durability of Switchgrass Pellets

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Moisture Content (w.b. %)</th>
<th>Box Durability (N=2)</th>
<th>Friabiliator Durability (%) (N=5)*</th>
</tr>
</thead>
<tbody>
<tr>
<td>Untreated</td>
<td>17.5</td>
<td>87.2% (86.4, 87.9)</td>
<td>94.0±1.94a</td>
</tr>
<tr>
<td></td>
<td>20.0</td>
<td>85.9% (88.5, 83.2)</td>
<td>94.0±0.70a</td>
</tr>
<tr>
<td>100 (for 5 minutes)</td>
<td>17.5</td>
<td>78.8% (86.8, 70.8)</td>
<td>92.9±1.12a</td>
</tr>
<tr>
<td></td>
<td>20.0</td>
<td>77.3% (80.0, 74.7)</td>
<td>93.1±0.61a,b</td>
</tr>
<tr>
<td>120 (for 5 minutes)</td>
<td>17.5</td>
<td>77.5% (82.8, 72.1)</td>
<td>94.9±0.56a,c</td>
</tr>
<tr>
<td></td>
<td>20.0</td>
<td>78.0% (75.6, 80.3)</td>
<td>93.6±0.87a</td>
</tr>
</tbody>
</table>

* Same superscripts are not significantly different (p>0.05) while different superscripts are significantly different (p<0.05)

5.4.1. Box Durability

The results of the box durability test on pellet samples are shown in Table 5.4. Pellet durability is not substantially affected by the change in moisture content between 17.5% and 20.0%. It is worth noting that sample 1 at 20.0% (w.b.) is below the metric for “good” durability of 85% (Stelte et al. 2012b), but, due to the limited sample size, it is difficult to draw conclusions about the population represented by the sample.

The use of a box durability tester requires 500 g of pellets per test which means the need for two to three pellet formation runs as each run produced approximately 200-300 g of pellets. The purpose of the box durability test is to provide a comparison to standard industrial methodology. Two replicates were measured for this comparison. The 500 g required sample size highlights the large pellet-to-pellet variability that the lab scale pelletizer produced. This is especially seen
across the two tested batches of the 17.5%-100°C treatment, with as high as 15% difference in between replicates.

While no statistically significant differences were detected, the box durability results seem to indicate a decrease in durability for dry steam preconditioning (overall mean of 77.9% and treatment mean values from 77.3% to 78.8%). Mean durability values for dry steam treated pellets were 7-10% lower than those of the untreated control (overall mean of 86.6% and treatment mean values of 85.9% and 87.2%). As intended, the box durability test was more aggressive than the friability test.

5.4.2. Friabilator Durability

The mean friabilator values ranged from 92.9-94.9% and standard deviation was as high as 1.94%. Table 5.4 shows the pellet friability results. The calculated friabilator durability values of the pellets are greater than those of the tumbling box durability test. Friability testing is also used to evaluate tablets and is less aggressive than the box test. The mean friability amongst all six treatment conditions was near 94%. Figure 5.5 shows the confidence interval plot of the friability for the six pellet conditions tested. Pairwise comparison does not show statistically significant differences among groups; except dry steam treated 20.0%-120°C compared with 17.5%-100°C.

Figure 5.5 95% confidence interval (CI) plot of pellet friability with 17.5% and 20.0% w.b. moisture content, for untreated and steam temperature of 100 and 120°C treatment condition.
6. Summary, Conclusions, and Future Recommendations

In this study, dry steam preconditioning of ground switchgrass was evaluated as a means of impacting pellet quality metrics. Physical properties of the control (i.e., unconditioned) and thermally conditioned pellets as well as the ground material were evaluated. Tests were conducted for biomass conditioned with dry steam at two different time-temperature conditions (100°C for 5 minutes and 120°C for 5 minutes) and two different moisture contents (17.5% and 20.0%, w.b.). Density, axial compressive strength, diametral tensile strength, friabilitator durability, and box durability tests were conducted to assess pellet quality. The following sections summarize key conclusions.

6.1. Physical Properties of Ground Biomass

The size distribution, D_{50}, particle density, and bulk density of control and thermally conditioned ground biomass were determined. The key conclusions are noted below.

6.1.1 Bulk Density

- Bulk density differences do not seem to relate to the addition of water or dry steam preconditioning.

6.1.2 Particle Density

- Adding water to the sample does not impact particle density (p>0.05)
- Adding water and steam preconditioning does particle density. The decrease is statistically significant (p<0.05). Loss of volatile compounds during steam treatment would result in lower density particles.

6.1.3 Particle Size Distribution

- Observed D_{50} values were similar for moisture conditioned and dry steam conditioned material (p>0.05).
6.2. Pellet Properties

Single pellet quality metrics of density, axial compressive strength, and diametral tensile strength and bulk pellet quality metrics of friability and box durability were determined. The key conclusions are noted below.

6.2.1 Pellet Density

- The moisture content at the selected levels of 17.5% and 20.0% did not have a significant effect (p>0.05) on pellet density compared to pellets formed using the unconditioned material.
- Density of pellets formed after dry steam preconditioning at the 100°C or 120°C steady temperature levels for five minutes were not significantly different (p>0.05) compared to pellets formed using the unconditioned material.

6.2.2 Pellet Strength

- Pellets formed after dry steam preconditioning at both the 100°C and 120°C steady temperature levels for five minutes had higher diametral tensile strength. However, these values were not significantly different (p>0.05) than pellets formed using the unconditioned material.
- The axial strength of dry steam preconditioned pellets at 100°C and 120°C steady temperature levels of five minutes were not significantly different (p>0.05) than pellets formed using the unconditioned material.
- The moisture content at the selected levels of 17.5% and 20.0% (w.b.) did not have a significant effect (p>0.05) on axial compressive strength or diametral tensile strength.

6.2.3 Pellet Durability

- Pellet friability was greater than 90% amongst all tested conditions. Friability values had a range of 91% to 96%. Pellet friability values were not significantly different (p>0.05) for treated pellets compared to the control.
• Box durability values were all greater than 70%. Dry steam preconditioning seemed to have a negative effect on the box durability of formed pellets, with a 7%-10% decrease in retained pellet mass compared to the pellets formed using unconditioned material.

6.3. Recommendations for Future Work

The present research evaluated the effect of dry steam preconditioning the physical properties of ground switchgrass and formed pellets. Based on this study, the following recommendations are proposed:

• The hydrostatic triaxial compression (HTC) test and conventional triaxial compression (CTC) test should be performed on the dry steam preconditioned material. This may be a useful prescreening metric for future studies of steam preconditioning.
• The effect of wet steam preconditioning on formed pellets should be studied.
• A temperature well above the onset of melting for lignin (140ºC) should be considered. The targeting of glass transition range did not seem to have the desired effect and higher temperature treatment may improve binder migration.
• For small scale batch pelletization, implementation of more aggressive screening step may reduce pellet to pellet variability. RoTap sieving should be explored as an alternate to the hand sieving used for this study.
• For durability evaluation of a small scale pellet formation using non-woody biomass densification, the box durability test as a metric should be investigated further to include smaller mass sample sizes.
References


ASABE Standards. 2012b. ASABE S358.2 Moisture Measurement – Forages. ASABE, St. Joseph, MI.


Faostat, United Nations (2012, November 30). FAOSTAT. Rome, Italy: FAO.


