

Article

# **Investigation of Process Variables in the Densification of Corn Stover Briquettes**

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Abstract: The bulk density of raw corn stover is a major limitation to its large-scale viability as a biomass feedstock. Raw corn stover has a bulk density of 50 kg/m³, which creates significant transportation costs and limits the optimization of transport logistics. Producing a densified corn stover product during harvest would reduce harvest and transportation costs, resulting in viable pathways for the use of corn stover as a biomass feedstock. This research investigated the effect of different process variables (compression pressure, moisture content, particle size, and material composition) on a densification method that produces briquettes from raw corn stover. A customized bench-scale densification system was designed to evaluate different corn stover inputs. Quality briquette production was possible using non-reduced particle sizes and low compression pressures achievable in a continuous in-field production system. At optimized bench settings, corn stover was densified to a dry bulk density of 190 kg/m³. Corn stover with a moisture content above 25%wb was not suitable for this method of bulk densification, and greater cob content had a positive effect on product quality.

**Keywords:** biomass; compression pressure; density; logistics; moisture content; pellet

#### 1. Introduction

Corn stover is a widely available biomass resource in the United States, particularly in the Midwest where corn is the predominant grain product. The USDA estimates that approximately 75 million dry tons of corn stover are available annually to support a biomass industry [1]. Although widely available, the low bulk density of corn stover poses a major challenge for large-scale acceptance as a biomass feedstock. The loose bulk density of chopped corn stover ranges from 40 to 80 kg/m³ [2], which creates large inefficiencies during harvest, transport, and storage phases of production. Transportation vehicles loaded with corn stover are restricted by volume capacity more than weight capacity, therefore a cost-effective means of increasing corn stover bulk density is critical to the feasibility of large-scale production [3]. Ideally, the material would be densified during harvest to minimize in-field transport wagons and trips, as well as maximize off-site hauling potential. Current self-unloading trailers suitable for off-site transportation of densified biomass have volumes of 65 to 90 m³. Based on the common legal weight restrictions of 36,290 kg for tractor-trailer combinations and the base weight of the tractor and trailer, the minimum bulk density of a briquetted biomass needed to fill these trailers to their weight capacity ranges from 270 to 370 kg/m³. This means a roughly four-fold increase in density from chopped corn stover is needed to optimize transportation efficiency.

Several densification methods have been used to overcome the poor bulk density of chopped corn stover: grinding, baling, briquetting, and pelleting [4]. Grinding operations are used in pre-processing for other densification systems and involve reducing the particle size of a material to increase bulk density [4]. Hammer mill performance with corn stover has an output bulk density ranging from 130 to 160 kg/m³ [5]. While an improvement over loose bulk density, grinding operations alone do not increase the density enough to maximize transportation efficiency to the 270 to 370 kg/m³ target range, and grinding operations are energy intensive [5].

Square baling operations provide an alternative to grinding by compressing material into a chamber with a reciprocating plunger and wrapping the material with strings to maintain a densified form. A study by Iowa State University (Ames, IA, USA) during the 2009 and 2010 harvest seasons produced baled corn stover wet bulk densities of 207 kg/m³ (156 kg/m³ dry bulk density) [6] with low energy requirements. Baling increases bulk density over loose stover and is more energy efficient than grinding operations [7], but requires an additional unit handling operation each time the bales are moved.

A final method of densifying corn stover is briquetting and pelleting. These operations apply extreme pressure and, in many cases, heat to compress materials into a self-retaining shape. A study using an instrumented testing machine yielded corn stover particle densities of 1220 kg/m³ [4], and ring-die pellet mill tests produced bulk densities of 550 to 610 kg/m³ [7]. While these systems offer greater density compared to baling and grinding systems, they require high energy inputs to complete [4,7] and are not feasible to accomplish in the field because they require sub-millimeter particle sizes to complete the process.

This research sought to improve the practicality and cost-effectiveness of briquette densification systems for large-scale biomass production by reducing requirements for energy and particle size reduction while also maintaining the robustness of densification practices across a naturally wide range of material moisture content and particle size properties that occur normally within industrial corn stover harvesting. Additionally, this paper presents a blended densification system which combines the

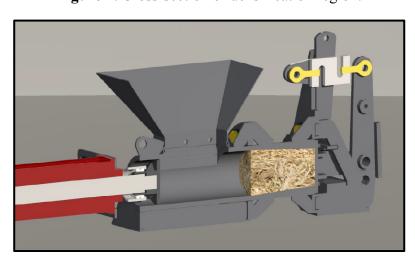
best features of various existing densification systems including a minimal requirement for size reduction and preprocessing found in baling systems as well as a bulk flowable final product which is similar to traditional high density pellets. This paper explores how various process variables impact briquette characteristics; a discussion of the energy component may be found in Thoreson [8].

Previous literature regarding biomass densification [4,7,9] suggests briquette densification characteristics are highly sensitive to compression pressure, material moisture content, and material particle size. In order to study the effects of these densification process variables, a customized bench-scale densification system was designed to evaluate different corn stover inputs. Four tests were conducted: (1) a three-way interaction experiment to assess the effects of compression pressure, material moisture content, and material particle size on dry material density; (2) a moisture effects experiment to determine the interactions of material moisture content, compression pressure, and die taper angle on dry particle density; (3) a material types experiment to evaluate the effect of corn stover material contents; and (4) a bulk density experiment to assess the densities of multiple briquettes at different moisture contents.

#### 2. Materials and Methods

# 2.1. Briquette Densification Bench Development and Basic Operation

A customized, bench-scale densification system was developed to conduct the experiments (Figure 1) [8]. This experimental briquetting system produced large cylindrical briquettes with a base diameter of 127 mm and a height of 200 mm. The chamber peak diameter of the briquette was determined by the die taper angle. For density calculations the final briquette dimensions were measured after being extracted from the compression chamber to account for material expansion. The system was designed to be capable of compression pressures ranging from 0 to 17 MPa. A tapered die was selected because it distributed a larger amount of axial force (resistance) to the material than a straight, cylindrical die (Figure 2). An instrumented die cap was installed to stop material flow and measure the cap force to quantify the process variable effects on force distribution.



**Figure 1.** Cross-section of densification region.

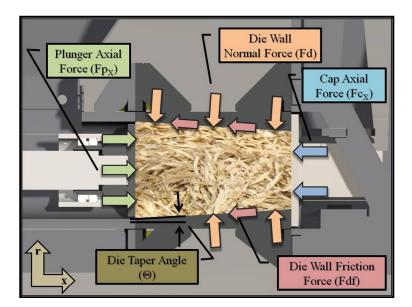


Figure 2. Free body diagram of the die region of the densification system.

To create briquettes, material was fed through the inlet on the top of the cylindrical chamber. Once in the chamber, a plunger pressed the material from the chamber into the tapered die. The process was repeated with additional material until the target briquette compression pressure (0 to 17 MPa) was reached. A separate ejection press is used to extract the finished briquette. The system was controlled by a macro-embedded Excel worksheet [10] on a personal computer. This computer was connected to a Measurement Computing 1408-FS data acquisition card, which controlled system functions and read data from two load cells and a string potentiometer to control and log compression pressure and plunger position.

## 2.2. Material Harvest and Classification

Corn stover utilized in this research was collected in the vicinity of Ames, IA during autumn of 2009 and 2010. These materials represented the range of materials that can be collected in common Midwest corn harvesting operations, including different material moisture contents, particle sizes, and corn stover material contents. Harvesting methods produced three classifications of material with different proportions of stalk, leaf, husk, and cob:

- (1) "Corn Stover." This material was harvested with a standard "all crop" header which cuts the plant just below the corn ear and harvests all material above the cut point. The output material primarily consisted of stalk and leaf;
- (2) "Material Other than Grain" (MOG). This material was harvested using a conventional corn header which generally harvests just the ear and husk. The output material primarily consisted of leaf, husk, and cob;
- (3) "Pure Cobs." This material was harvested using a conventional corn header and combine with a secondary attachment for output material processing. The output material primarily consisted of cob.

A range of different moisture contents were generated in each of these three classifications by using samples from different harvest dates or by moisture conditioning the material. Moisture conditioning

was limited to natural air drying of experimental samples before processing. Natural air drying by allowing samples to rest in an open ambient environment reduced the risk of over drying associated with oven drying procedures. Subsamples were collected periodically to evaluate the current moisture of the experimental samples and samples were processed once they reached a target moisture content.

Stover samples were dried at 60 °C for 72 h, and final weights were measured immediately following drying. Moisture content was quantified using by the drying procedure from ASABE S358.2 [11]. The observed moisture content for each sample is listed in the experiment description (Section 2.3) and is reported on a wet basis (wb). Particle sizes were classified into three categories by size reduction method:

- (1) "As Received." This material classification passed through an integrated chopper on a combine, but underwent no further size reduction;
- (2) "Wood chipper." This material classification was "As Received" above and subjected to further size reduction using a Vermeer HG200 wood chipper with dual parallel screens set at 70 and 111 mm;
- (3) "Hammer mill." This material was classification was "As Received" above and subjected to further size reduction using an Arts-Way 60HP stationary hammer mill with a 19 mm round-hole screen.

Particle size was quantified using the screening procedure outlined in ASABE S424.1 [12]. The range of geometric mean particle sizes based on multiple test replications are reported for each experiment in Section 2.3. Moisture content is a significant factor in particle size reduction and in all cases for a unique size reduction method increased moisture content led to an increased geometric mean particle size. Physical briquette properties were measured by the operator and mechanical data was measured by the densification bench data acquisition system. Particle density was determined by manually measuring the volume of a briquette and measuring the weight using a digital scale. Density was reported on a dry material basis. Qualitative assessments of briquette properties, such as flaking, smoothness, and durability, were also made.

#### 2.3. Experiment Design

# 2.3.1. Three-Way Interaction Experiment

The objective of the three-way interaction experiment was to determine the main and interaction effects of compression pressure, material moisture content, and material particle size on dry particle density. Three levels of compression pressure (7.0, 10.5, 14.0 MPa), two levels of material moisture content (8.3%<sub>wb</sub>, 54.5%<sub>wb</sub>), and three levels of material particle size (methods 1 (40 to 42 mm), 2 (22 to 35 mm), 3 (19 to 23 mm) as described in Section 2.2 above) were tested. This created 18 individual treatments (briquettes) that were replicated three times.

# 2.3.2. Moisture Effects Experiment

The moisture effects experiment was developed to study the main and interaction effects of material moisture content, compression pressure, and die taper angle ( $\theta$  in Figure 2) on dry particle density. Three levels of material moisture content ( $13.0\%_{wb}$ ,  $24.8\%_{wb}$ ,  $47.6\%_{wb}$ ), two levels of compression

pressure (8.8, 14.0 MPa), and two levels of die taper angle (3.6°, 7.2°) were tested and replicated three times for a total of 36 briquettes produced. As received corn stover material was used for all replications of this test. The increased minimum compression pressure was chosen based on the poor performance of the low (7.0 MPa) compression pressure during the three-way interaction experiment. The mean particle size of the corn stover was held constant at 40 mm throughout this experiment.

# 2.3.3. Material Types Experiment

Different proportions of corn stover components could produce different densification characteristics. Three different corn stover material classifications (corn stover, MOG, pure cobs as described in Section 2.2 above) were assessed with two levels of compression pressure (7.0, 14.0 MPa), and two levels of material particle sizes (methods 1 and 3 as described in Section 2.2 above). The experiment was replicated three times for a total of 36 briquettes produced. Material for this experiment was densified directly from harvest, so the material moisture content of the samples was not controlled and ranged from 10%<sub>wb</sub>–20%<sub>wb</sub>.

## 2.3.4. Bulk Density Experiment

In order to understand the bulk density of these briquettes, an experiment was designed to determine the bulk density of the material for different material moisture contents. Bulk density measures the density of several briquettes combined rather than the density of a single briquette. Briquettes were produced at a specified treatment and loaded into 0.19 m³ cylindrical barrels with a diameter of 61 cm and a height of 65 cm until the barrels were approximately 60% full. Briquette bulk sample volume and weight were then measured to determine the characteristics of the briquetted stover. The final briquette volume varied based on the test due to expansion after release from the compression chamber (Section 2.1). The experiment was designed with two levels of material moisture content (8.3%<sub>wb</sub>, 54.5%<sub>wb</sub>) and was replicated three times for a total of six barrels of briquettes. In all tests a compression pressure of 10.5 MPa was used and the material type was corn stover. As received material was used with no additional size reduction before the test was conducted. The large moisture content difference was chosen to cover the viable range of extremes from both abnormally wet and dry harvest seasons that are common in the collection of agricultural residues.

## 2.4. Data Analysis

All statistical tests were conducted using Minitab software [13]. All experiments discussed utilized full-factorial experiment designs which allowed the use of the factorial analysis tool (ANOVA) to determine the variation caused by each treatment factor. Factorial ANOVA was used to determine the significance of each treatment variable on the selected output factor. Confidence interval plots were used to determine if differences were present between treatment variable levels. Confidence intervals assume a normal distribution of the data and use critical values following a t-distribution.

#### 3. Results and Discussion

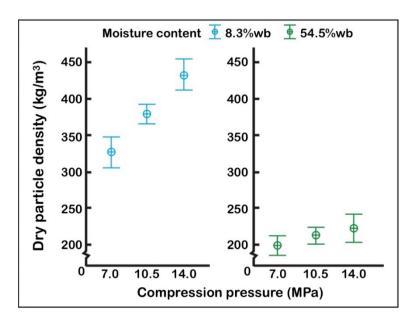
## 3.1. Three-Way Interaction Experiment

# 3.1.1. Dry Particle Density

During this experiment, briquettes were produced with dry particle densities between 170 and 470 kg/m<sup>3</sup>. All three treatment factors (compression pressure, particle size reduction method, and material moisture content), had an effect on dry particle density (p < 0.05). There was also an interaction effect from moisture content and compression pressure (p < 0.01).

At  $8.3\%_{wb}$  moisture content, compression pressure showed a positive output particle density ranging from 330 to 430 kg/m<sup>3</sup> across 7.0 to 14.0 MPa (Figure 3). This density was caused by an increase in briquette weight from the additional compression cycles when material was added without producing an increase in briquette axial expansion (the amount the briquette expands after ejection from the die). No differences were found between the compression pressure treatment levels at  $54.5\%_{wb}$  moisture content on dry particle density ( $\alpha = 0.05$ ). While the factorial ANOVA showed a potential relationship between the particle size reduction method and dry particle density, only two isolated treatment differences were observed between particle size treatments levels during the experiment ( $\alpha = 0.05$ ).

**Figure 3.** Treatment factor effects on dry particle density. Compression pressure and moisture content are averaged over all particle sizes. Means are represented by the crossed circle and error bars indicate the 95% confidence interval for the mean.



Increasing moisture content from 8.3%<sub>wb</sub> to 54.5%<sub>wb</sub> decreased the mean dry particle density by about 45% from 380 to 210 kg/m<sup>3</sup> (averaged over all compression pressure levels and material particle size treatments). This decrease can be attributed to two main factors observed during this experiment: briquette dry weight and briquette axial expansion. The elastic behavior of the briquette was also altered with increased moisture content, as evidenced by the increase in briquette axial expansion over increasing moisture content (Figure 3). Both of these factors directly contributed to a reduction in dry particle density with increased material moisture contents.

## 3.1.2. Qualitative Effects

The effect of compression pressure on the briquette quality can be observed by visual inspection (Figure 4). While the overall size and shape of the briquettes was about the same, the surface texture of the 14.0 MPa briquette (Figure 4B) was much smoother and held together better than the 7.0 MPa briquette (Figure 4A).

**Figure 4.** Qualitative comparison of briquettes produced under varying parameters. All briquettes shown were produced with corn stover (classification 1 in Section 2.2) unless otherwise indicated. (**A**) "as received" briquette produced under 7.0 MPa in the three-way interaction experiment; (**B**) "as received" briquette produced under 14.0 MPa in the three-way interaction experiment; (**C**) "hammer milled" briquette produced in the three-way interaction experiment; (**D**) briquette produced at 47.6%wb moisture content in the moisture effects experiment; (**E**) briquette produced using MOG in the material types experiment; (**F**) briquette produced using pure cubs in the material types experiment.



The difference between the tested particle size reduction methods was also visually noticeable. The hammer-milled briquette (Figure 4C) shows slightly more flake separation, whereas the previous two "as received" briquettes (Figure 4A,B) are more uniform across the briquette length. Briquette surface stiffness was also reduced for smaller particle sizes. The apparent flake separation and reduced stiffness of hammer milled briquettes will likely have a negative effect on briquette durability and the final bulk density of the product.

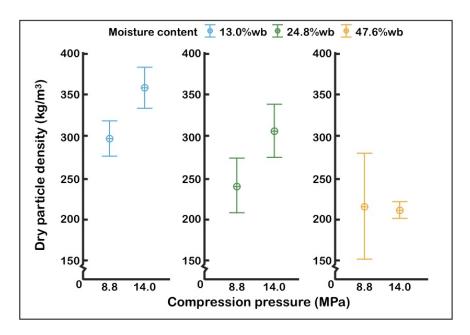
# 3.2. Moisture Effects Experiment

# 3.2.1. Dry Particle Density

This experiment produced briquettes ranging in dry particle density from 170 to 400 kg/m<sup>3</sup>. Two treatment factors, material moisture content and compression pressure, affected the dry particle density (p < 0.01). Die taper angle did not affect the dry particle density and there were no interaction effects.

Compression pressure demonstrated similar effects to those observed during the three-way interaction experiment. Increasing levels of compression pressure produced dry particle density gains at moisture contents at and below 24.8%<sub>wb</sub> moisture content (Figure 5).

**Figure 5.** Treatment factor effects on dry particle density. Means are represented by the crossed circle and error bars indicate the 95% confidence interval for the mean.



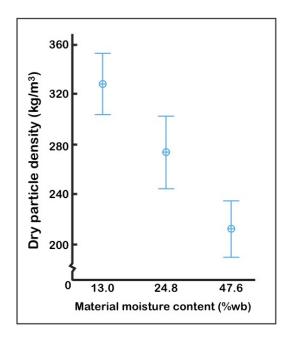
Following a similar trend displayed during the three-way interaction experiment, material moisture content had a negative effect on dry particle density. From 13.0%<sub>wb</sub> to 47.6%<sub>wb</sub> moisture content, dry particle density decreased by approximately 35% (Figure 6).

While the final relationship between moisture content and dry particle density was similar to the three-way interaction and moisture effect experiments, the reasoning behind the relationship was different. During the three-way interaction experiment, increased moisture content led to decreased briquette dry weight and increased axial expansion, which resulted in reduced dry particle density values. During the moisture effects experiment, an increase in dry briquette weight was only observed between 13.0%<sub>wb</sub> and 24.8%<sub>wb</sub> moisture content and not with further increase in moisture content to 47.6%<sub>wb</sub> (Figure 7).

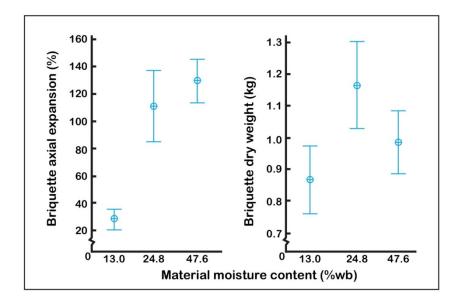
Weight increases were small (33% increase between 13.0%<sub>wb</sub> and 24.8%<sub>wb</sub>) in comparison to the changes in briquette axial expansion over different moisture contents (Figure 7). A pronounced difference was observed between 13.0%<sub>wb</sub> and 47.6%<sub>wb</sub> moisture content, where axial expansion increased by 360%. This expansion negates the density gains the increased dry particle weight offered. Consequently, while certain moisture content levels might allow for more dry material weight to be

densified, any density gained in that manner will be lost upon ejection due to the increased elasticity of briquettes made at increased moisture content.

**Figure 6.** Moisture content effect on dry particle density averaged over all levels of compression, pressure, and die taper angle. Means are represented by the crossed circle and error bars indicate the 95% confidence interval for the mean.



**Figure 7.** Moisture content effects on axial expansion and dry briquette weight. Means are represented by the crossed circle and error bars indicate the 95% confidence interval for the mean.



# 3.2.2. Qualitative Effects

The effect of material moisture content on briquette quality can be visually observed (Figure 4). The obvious visual difference was the amount of axial expansion observed on the high moisture briquette (47.6%<sub>wb</sub> moisture content, Figure 4D) compared to low moisture briquettes (*i.e.*, 13.0%<sub>wb</sub> moisture

content, Figure 4B). Dry briquettes can be handled by hand as a single unit, while wet briquettes cannot be moved without completely supporting the briquette. Without support, the wet briquette completely falls apart and handles like loose corn stover.

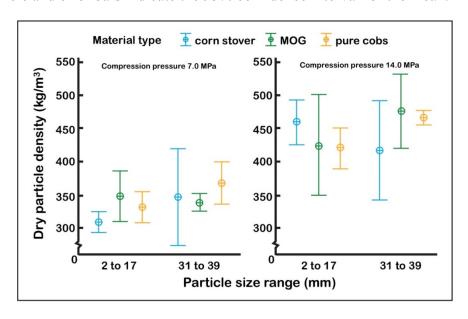
# 3.3. Material Types Experiment

# 3.3.1. Dry Particle Density

This experiment produced briquettes with dry particle density ranging from 300 to 500 kg/m<sup>3</sup>. Two treatment factors, compression pressure and particle size reduction method, had an effect on dry particle density (p < 0.05). Material type did not have an effect on dry particle density, however, the interaction effect of material type and material particle size did affect the dry particle density (p < 0.05).

No dry particle density differences were caused by material type (Figure 8;  $\alpha$  = 0.05). Similar to the previous experiments, the effect of material particle size was inconsistent across each material type and compression pressure treatment. However, particle size did have an effect on pure cob materials at 14.0 MPa compression pressures. With this compression, hammer milling the cobs resulted in an 11% increase in dry particle density.

**Figure 8.** Treatment factor effects on dry particle density. Means are represented by the crossed circle and error bars indicate the 95% confidence interval for the mean.



Compression pressure was the only variable that demonstrated consistent differences between treatment levels ( $\alpha = 0.05$ ) on dry particle density. Averaged across every material type and particle size reduction method, increasing compression pressure from 7.0 to 14.0 MPa increased the dry particle density by 30%. This can be directly attributed to a 30% increase in briquette weight with no change in the briquette axial expansion.

## 3.3.2. Qualitative Effects

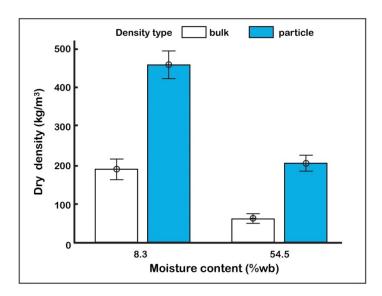
Flake divisions were clearly visible for every corn stover briquette produced (*i.e.*, Figure 4B). The surface texture of MOG (Figure 4E) and pure cobs (Figure 4F) was significantly smoother and stiffer.

Flake visibility was reduced with MOG and nearly impossible to identify with the pure cob briquettes. MOG and pure cobs formed a more cohesive briquette compared to corn stover. Based on these observations, increasing the cob content improved the overall briquette quality.

# 3.4. Bulk Density Experiment

The bulk density experiment showed material moisture content effected dry bulk density. A large decrease was observed between particle density and overall bulk density. For 54.5%<sub>wb</sub> moisture content stover, the dry density decreased approximately 70% going from particle to bulk density while the 8.3%<sub>wb</sub> moisture content stover had a decrease of approximately 60% (Figure 9). This inefficiency in translating particle density to bulk density was created by large air-filled spaces between the briquettes. Overall, the low moisture material had a superior dry bulk density (mean 190 kg/m³) relative to the high moisture material (mean 64 kg/m³). High moisture material offers no improvement over non-densified corn stover, which ranges between 40 and 80 kg/m³ [2].

**Figure 9.** Material moisture content effect on density. Means are represented by the crossed circle and error bars indicate the 95% confidence interval for the mean.



## 3.5. Summary of Key Findings

Compression pressure was tested from 7.0 to 14.0 MPa and positive, fairly linear trends were demonstrated with respect to dry particle density. At optimized treatment variables, dry particle density increased 50% (310 to 460 kg/m³) when increasing compression pressure from 7.0 to 14.0 MPa.

The experiments showed the most important effects on all of the output factors were caused by material moisture content. Material moisture content was tested on corn stover ranging from approximately 8%<sub>wb</sub> to 55%<sub>wb</sub> throughout the testing period. From 8%<sub>wb</sub> to 25%<sub>wb</sub>, the briquette quality transitioned from good to poor. At optimized treatment variables, dry particle density decreased from 368 to 214 kg/m³ (42%) when increasing material moisture content from 13.0%<sub>wb</sub> to 47.6%<sub>wb</sub>. Material bulk density would significantly decrease due to the extreme amount of briquette expansion at moisture contents greater than 25%<sub>wb</sub>, indicating that future in-field densification systems based from this research will require materials dryer than 25%<sub>wb</sub>.

Material particle size did not have consistent significant effects on particle density. Qualitatively, decreasing particle size led to a greater number of flake divisions and a softer briquette. For this type of densification, any particle size reduction beyond the integrated chopper on the combine is not recommended.

While material type had minimal effects on dry particle density, obvious qualitative differences were noted between treatments. Based on briquette stiffness and flake divisions, overall briquette quality was improved with increased cob content. Harvesting systems that can increase cob content would be advantageous to this type of densification system. Further extrapolation of these advantages can be found in Thoreson [8].

## 4. Conclusions

Based on briquette density and visual briquette quality, the following process variable settings should be used to maximize densified product quality:

- Only "as received" material particle sizes (passed through an integrated combine chopper only) should be used to reduce briquette flake divisions;
- Maximum compression pressure (14.0 MPa) was shown to maximize particle density. Future work should evaluate greater compression pressures and evaluate the relationship between compression pressure and total energy requirements in order to quantify an optimal balance of pressure and energy for a commercial implementation;
- Only moisture contents below 25%<sub>wb</sub> should be used to maximize briquette density and quality. When crop conditions exceed 25%<sub>wb</sub> alternative densification methods should be implemented;
- Corn stover contents with a greater cob content will improve briquette quality.

While this densification method did not increase the corn stover density enough to optimize off-site transportation, the improved bulk density was competitive with baled corn stover densities. Depending on the type and moisture of corn stover harvested, material can be continuously densified to approximately 190 kg/m³ dry bulk density (210 kg/m³ wet bulk density). Based on maximum legal trailer dimensions available without a specialized permit, this densified corn stover product allows trucks to haul approximately 18 to 19.5 metric tons using a single, 16.1 m (53 foot), live bottom trailer. By comparison, up to 18 to 20 metric tons of large square bales (0.9 m × 1.2 m × 2.4 m) can be hauled using a single, 16.1 m (53 foot) flatbed or drop deck trailer.

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# **Author Contributions**

Authors Thoreson and Webster were responsible for execution of experiments and analysis of data presented in this paper. Research oversight was provided by Darr throughout the completion of this work. Author Kapler provided additional data analysis and technical editing during the preparation of this manuscript.

## **Conflicts of Interest**

The authors declare no conflict of interest.

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