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Method to produce durable pellets at lower energy consumption using high moisture corn stover and a corn starch binder in a flat die pellet mill

--Manuscript Draft--

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Abstract:	A major challenge in the production of pellets is the high cost associated with drying biomass from 30 to 10% (w.b.) moisture content. At Idaho National Laboratory, a high-moisture pelleting process was developed to reduce the drying cost. In this process the biomass pellets are produced at higher feedstock moisture contents than conventional method, and the high moisture pellets produced are further dried in energy efficient dryers. This process helps to reduce the feedstock moisture content by about 5-10% during pelleting, which is mainly due to frictional heat developed in the die. The objective of this research was to explore how binder addition influences the pellet quality and energy consumption of the high-moisture pelleting process in a flat die pellet mill. In the present study, raw corn stover was pelleted at moistures of 33, 36, and 39% (w.b.) by addition of 0, 2, and 4% pure corn starch. The partially dried pellets produced were further dried in a laboratory oven at 70 °C for 3-4 hr to lower the pellet moisture to less than 9% (w.b.). The high moisture and dried pellets were evaluated for their physical properties, such as bulk density and durability. The results indicated that increasing the binder percentage to 4% improved pellet durability and reduced the specific energy consumption by 30-50% compared to pellets with no binder. At higher binder addition (4%), the reduction in feedstock moisture during pelleting was <4%, whereas the reduction was about 7-8% without the binder. With 4% binder and 33% (w.b.) feedstock moisture content, the bulk density and durability values observed of the dried pellets were >510 kg/m3 and >98%, respectively, and the percent fine particles generated were reduced to <3%.
Author Comments:	Dear Editor, We earlier got an invitation to submit an article to JOVE journal based on our previously published papers in Energy Science and Engineering and Bioresource

	Technology. We take the opportunity to submit our original research article titled "Method to produce highly durable pellets at lower energy using high moisture corn stover and a corn starch binder in a flat die pellet mill" for review and publication in JOVE journal. Thanks for giving us this opportunity. Regards Jaya Shankar
Additional Information:	
Question	**Response**
If this article needs to be "in-press" by a certain date to satisfy grant requirements, please indicate the date below and explain in your cover letter.	

January 6th, 2016, Idaho Falls

To
JayDev Upponi
Science Editor
Journal of Visualized Experiments (JoVE)
Cambridge, MA, 02140

Dear JayDev,

We have now revised the manuscript based on Editor and reviewers comments and suggestions and resubmitted it for review and publication in the Journal of Visualized Experiments.

The changes we have made in the manuscript are indicated in different color for Editor and each reviewer comments. The following changes were made in the revised manuscript.

a) Added additional details on pellet diameter and expansion ratio to address the reviewer comments.
b) Added additional information in introduction section based on reviewer comments.
c) Updated the protocol section based on the reviewer comments.
d) Statistical analysis of the experimental data is included in the revised manuscript.
e) The figures have been updated in the revised manuscript.
f) Updated the results and discussion section based on reviewers comments and suggestions
g) Formatted the references based on the JOVE journal requirements.

Please let us know if you have questions on our revised manuscript submission. Thanks for giving us the opportunity to submit the revised manuscript.

We look forward to receiving the reviewer's comments and suggestions.

Sincerely,
Jaya Shankar Tumuluru, Ph.D.

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TITLE:
Method to produce durable pellets at lower energy consumption using high moisture corn stover and a corn starch binder in a flat die pellet mill

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KEYWORDS:
Corn stover, high moisture pelleting, process variables, starch binder, pellet properties, specific energy consumption.

SHORT ABSTRACT:
In this study, a protocol was developed to produce good quality pellets using a flat die pellet mill at reduced specific energy consumption testing high-moisture corn stover and a starch based binder. The results indicated that adding a corn starch binder improved the pellet durability, reduced percent fines and decreased specific energy consumption.

LONG ABSTRACT:
A major challenge in the production of pellets is the high cost associated with drying biomass from 30 to 10% (w.b.) moisture content. At Idaho National Laboratory, a high-moisture

pelleting process was developed to reduce the drying cost. In this process the biomass pellets are produced at higher feedstock moisture contents than conventional method, and the high moisture pellets produced are further dried in energy efficient dryers. This process helps to reduce the feedstock moisture content by about 5–10% during pelleting, which is mainly due to frictional heat developed in the die. The objective of this research was to explore how binder addition influences the pellet quality and energy consumption of the high-moisture pelleting process in a flat die pellet mill. In the present study, raw corn stover was pelleted at moistures of 33, 36, and 39% (w.b.) by addition of 0, 2, and 4% pure corn starch. The partially dried pellets produced were further dried in a laboratory oven at 70 °C for 3–4 hr to lower the pellet moisture to less than 9% (w.b.). The high moisture and dried pellets were evaluated for their physical properties, such as bulk density and durability. The results indicated that increasing the binder percentage to 4% improved pellet durability and reduced the specific energy consumption by 30–50% compared to pellets with no binder. At higher binder addition (4%), the reduction in feedstock moisture during pelleting was <4%, whereas the reduction was about 7–8% without the binder. With 4% binder and 33% (w.b.) feedstock moisture content, the bulk density and durability values observed of the dried pellets were >510 kg/m^3 and >98%, respectively, and the percent fine particles generated were reduced to <3%.

INTRODUCTION:

Biomass is one of the major energy resources in the world and is considered carbon neutral[1]. Bulk density of baled and ground agricultural biomass and chipped woody biomasses is low. Low bulk densities of baled biomass (130–160 kg/m^3), ground biomass (60–80 kg/m^3) and chipped woody biomass (200–250 kg/m^3) create storage, transportation, and handling issues[2,3].Densifying or compressing the ground biomass by using pressure and temperature increases the bulk density by about 5 to 7 times, and helps to overcome transportation and storage limitations[4]. Pellet mills, briquette presses, and screw extruders are densification systems typically used for biomass[4]. Breakeven transportation distance analysis on baled and pelleted biomass feedstock indicated that pellets can be transported 1.5 times farther than bales using a truck for the same cost[5].The transportation efficiencies of pellets increase with other modes of transportation like rail and cargo ships, since they are volume-limited compared to trucks that are limited by weight. Currently, in Europe the pellets produced from woody biomass are extensively used for bio-power generation. Canada and the United States are the major producers and suppliers of wood pellets to Europe[6]. Pellets produced from both woody and herbaceous biomass can be used for both thermochemical (cofiring, gasification, and pyrolysis) and biochemical conversion (ethanol) applications[7-9].

The qualities of pellets (density and durability) and specific energy consumption of the pelleting process are dependent on the pellet mill process variables, such as die diameter, die speed and length to diameter ratio of the die and feedstock variables, such as feedstock moisture content and composition[4]. Both of pellet mill process variables and feedstock variables influence the quality of the pellets and the specific energy used in the process. The die dimensions (i.e. length-to-diameter ratio) will influences the compression and extrusion pressure, and the die rotational speed controls the residence time of the material within the die. Moisture content is a feedstock variable that plays an important role by interacting with the biomass composition components (i.e. protein, starch, and lignin) due to high temperature and pressure encountered in the die. The presence of moisture increases the van der Waals forces, thereby increasing the attraction between the biomass particles[10]. In general, higher moisture in the biomass impacts the bulk density of the compressed product due to diametrical and lateral expansion as it exits the pellet mill or briquette press die[10]. Biomass composition, such as starch, protein, lignin, and other water-soluble carbohydrates, influences the binding behavior when subjected to pressure and temperature in densification equipment[11]. Some of the common composition reactions that are influenced by feedstock moisture, die temperature, and pressure are starch gelatinization, protein denaturation, and lignin glass transition. Generally, at temperatures of 100 °C or greater and a feedstock moisture content of greater than 30%, starch in food and animal feed gets gelatinized and influences textural properties like hardness[12]. Typically, the starch reactions are gelatinization, pasting, and retrogradation. Among these reactions, gelatinization has the greatest influence on pellet properties[13]. Starch is often included in food and non-food applications as a binder. For example, in the pharmaceutical tablet formulation starch is used as filler[4,14]. Protein in the biomass undergoes denaturation and forms complex bonds due to the high temperature and pressure experienced in the densification process[11]. In general, higher amounts of protein in biomass will result in a more durable pellet[15,16]. For example, alfalfa, which has a higher amount of protein, results in durable pellets at higher feedstock moisture content. The fat in the biomass reduces the friction forces and the extrusion energy during pelleting or briquetting[11,17]. In lignocellulosic biomass, the presence of lignin within plant material helps to form pellets without adding any binders[18]. Woody biomass has higher lignin content (29–33%) when compared to a herbaceous biomass, which typically consists of 12–16% lignin[4,19]. At lower feedstock moisture contents of about 10-12% (w.b.), glass transition temperature of the lignin is greater than 140 °C[20]; whereas, increasing the moisture content reduces the glass transition temperature[21]. According to Lehtikangas[22], the glass transition temperature of lignin at 8-15% (w.b.) moisture content is about 100-135 °C, but increasing the moisture content to >25% (w.b.) reduces the glass transition temperature to <90 °C.

Herbaceous biomass is available at higher moisture content depending on the harvesting method and harvesting time. In the case of single pass harvesting method the harvested material will have a moisture content >30% (w.b.)[23]. Biomass is typically dried to about 10% (w.b.) moisture content to make it aerobically stable and to prevent dry matter loss during storage. Lamers et al.[24] indicated that to preprocess biomass at 30% moisture content the total cost for both grinding (stage-1 & 2) and drying is about $43.60/dry ton, and about $15.00/dry ton is just for drying the biomass. Drying biomass takes about 65% of the total preprocessing energy, and pelleting takes about 8-9%.[18] Yancey et al.[25] has corroborated the fact that drying is the major energy consumer in biomass preprocessing. The experimental data and techno-economic analysis indicated that efficient moisture management is critical for reducing the biomass preprocessing costs. One way to decrease the drying cost and manage the feedstock moisture more efficiently is to use a high-moisture pelleting process coupled with a low temperature drying method. In the high-moisture pelleting process developed at Idaho National Laboratory, the biomass is pelleted at moisture contents greater than 28% (w.b.); the partially dried pellets produced which are still high in moisture, can be dried in energy efficient driers, such as grain or belt dryers[21]. One major advantage of high-moisture pelleting is that it helps reduce the drying cost, which in turn results in reduced total pellet production cost. Techno-economic analysis indicated that energy and production costs are reduced by about 40-50% using the high-moisture pelleting process compared to a conventional pelleting method[24,26]. The major reason for reduced pellet production cost is due to replacing a rotary dryer that operates at high temperatures of 160 to 180 °C with a grain dryer that operates at lower temperatures of about 80 °C or less[21]. The other advantages of replacing a rotary dryer with a belt or grain dryer are: 1) greater efficiency, 2) reduced fire hazard, 3) does not need high quality heat, 4) reduced volatile organic compound (VOC) emissions, 5) reduced particulate emissions, and 6) does not agglomerate high clay or sticky biomass[27]. The energy-intensive steam conditioning step in conventional pelleting , typically used to add moisture and activate some of the biomass components, is replaced with a short preheating step. This step helps reduce the feedstock moisture content as well as activate biomass components like lignin. The frictional heat developed in the pellet die also helps reduce the feedstock moisture content by about 5–8% (w.b.)[21,28]. In the high-moisture pelleting process, the pellet mill not only compresses the biomass, but also helps to reduce the moisture content during compression and extrusion. Many researchers have done experiments on pelleting of raw and chemically pretreated biomass at a wide range of moisture contents (7-45%, w.b.) using single, laboratory, pilot, and commercial continuous pelleting systems[10,25,29-41]. These researchers adjusted feedstock moisture content of the biomass to different desired levels to understand the effect of moisture content on quality attributes of the pellets.

Pellet quality attributes, bulk density and durability, are normative specifications according to the USA based Pellet Fuel Institute (PFI). However, according to the European Committee for Standardization (CEN) durability is a normative and bulk density is an informative specification[42]. Pellets with durability values >96.5% and bulk density >640 kg/m^3 are designated as super premium pellets based on PFI standards, whereas pellets with durability values >97.5% are designated as pellets with the highest grade. Both the CEN and PFI standards recommend pellets with different diameters. For example, PFI recommends a diameter in the range of 6.35-7.25 mm, whereas CEN recommends a diameter ranging from 6-25 mm and a pellet length less than or equal to 4 times the diameter[42]. Smaller diameter pellets (6 mm) are preferred for transporting longer distances considering they have higher packing densities[28]. For conventional pelleting processes, it is recommended to pellet biomass at low moisture contents to meet these density specifications desirable for transporting the pellets long distances[42]. Both CEN and PFI have additional pellet grades[42]. Tumuluru[28] and Tumuluru and Conner[41] indicated that high moisture pelleting processes developed at Idaho National Laboratory help to produce corn stover and wood pellets with different quality attributes (bulk density and durability) and specific energy consumption making them suitable for different transportation and logistics scenarios.

Most of the pelleting studies on biomass were done using a single pelleting system. Pelleting data on biomass using a continuous system at laboratory scale is limited. Studies on continuous pelleting systems will be useful to understand the effect of the pelleting process variables like die rotational speed, length to diameter ratio and die diameter on the quality attributes and specific energy consumption. The pelleting data on the continuous systems can be further used to scale up the process to pilot and commercial scale system. In general, a flat die pellet mill is used for conducting pelleting studies on woody and herbaceous biomass in a laboratory[4]. The working principle of the laboratory scale flat die, and pilot, and commercial scale ring die pellet mills are similar. All these pellet mills have a perforated hard steel die with one or two rollers. By rotating the die, the rollers exert force on the feedstock and force it through the perforations of the die to form densified pellets[4].

Our earlier studies on high-moisture pelleting of corn stover at feedstock moisture content of 28-38% (w.b.) without any binder addition resulted in lower durability values at higher feedstock moisture content[21,28]. Improving the durability of high moisture pellets after cooling and drying is important as it helps to prevent the disintegration of the pellets (loss of pellet quality) during handling, storage and transportation. The disintegration of pellets typically results in fines generation and loss of revenue for the pellet producers. Binders are typically used in the pelleting process to improve pellet quality, especially durability, and to reduce the specific energy consumption. Commonly used natural binders in the pelleting process are proteins and starch[4,28]. Starch undergoes gelatinization, whereas protein undergoes denaturation in the presence of heat, moisture, and pressure. Both of these reactions result in better binding and more durable pellets at lower energy consumption. The overall objective of this study were to develop and demonstrate a high-moisture pelleting process using corn stover with the addition of a binder to produce good quality pellets in terms of green durability (after cooling) and cured durability (after drying) at a lower specific energy consumption. The specific objectives for the study were to 1) conduct high-moisture pelleting of corn stover at different feedstock moisture contents (33, 36, and 39%, w.b.) and starch binder contents (0, 2, and 4%), 2) evaluate the physical properties (pellet moisture content, pellet diameter, expansion ratio, bulk density and durability (green and cured durability)), and 3) evaluate specific energy consumption of the pelleting process.

PROTOCOL:

NOTE: Corn stover bales were procured in the form of bales from agricultural farms in Iowa, USA. The procured bales were ground sequentially in two stages. In stage 1, the corn stover bales were ground using a grinder fitted with a 50.8-mm screen. In stage 2, the ground material from stage 1 was further ground by using a Bliss hammer mill fitted with a 4.8-mm screen. The material was tested for moisture content and bulk density and stored in air tight container for further pelleting tests. Pure corn starch was procured from a local market and is measured for the moisture content and bulk density. The moisture content and bulk density of ground corn stover and corn starch binder are given Table 1.

1. Pellet Mill

1.1 Use a laboratory scale flat die pellet mill equipped with a 10 HP motor for carrying out the pelleting tests (**Figure 1**)[21,28,38].

[Place *Figure 1* here]

1.2 Place flexible heating tape on the surface of the hopper and screw feeder then insulate them with glass wool to prevent heat loss. Connect the heating tape to a temperature controller to preheat biomass to desired temperature in the range of 30-130 °C.

1.3 Equip the pellet mill with a variable frequency drive (VFD). Connect the VFD of the pellet mill to the pellet mill motor. The feeder motor controller is a direct current motor controller to vary the feeding rate to pellet mill.

1.4 Connect a power meter to the pellet mill motor to record the power consumption. Manually choose a pellet die with an 8mm diameter opening and a length to diameter (L/D) ratio of 2.6.

1.5 Add a horizontal pellet cooler to the pellet mill to cool the warm pellets coming out of the pellet die. Connect the cooler to an exhaust system to circulate fresh air.

2. Feedstock Preparation

2.1 Take 2-3 kg of corn stover ground using a 4.8 mm screen. Measure the corn stover moisture content (see step 4.1) and bulk density (see step 4.3) (see **Table 2**).

2.2 Measure moisture content (see step 4.1) and bulk density (see step 4.3) of the pure (100%) corn starch binder procured from the local market.

2.3 Add corn starch binder to the ground corn stover (see **Table 2** for % binder addition)

2.4 Calculate the amount of water to be added to adjust the moisture levels of ground corn stover to 33, 36, and 39% (w.b.) using equation 1.

$$W_w = Ws \times \left(\frac{m_f - m_i}{100 - m_f}\right) \qquad (1)$$

NOTE: In equation 1, W_w is weight of water (g), W_s is weight of biomass sample (g), m_f : percent final moisture content of the sample (w.b.), and m_i: percent initial moisture content of the sample (w.b. %).

2.5 Add the calculated water to the corn stover/corn starch binder mix and blend it in a laboratory scale ribbon blender.

2.6 Store the moisture-adjusted corn stover/corn starch mix in a sealed container and place it in a refrigerator set at 4–5 °C to allow moisture to equilibrate.

3. High Moisture Pelleting Process

3.1 Take the corn stover/corn starch mix out of refrigerator and leave it at room temperature for about 1-2 hr to bring it to room temperature.

3.2 Load the material into the feed hopper of the pellet mill. Run the pellet mill at 60 Hz (440 rpm) die speed.

3.3 Feed the pellet mill uniformly by adjusting the feeding rate of the pellet mill to produce pellets in a steady state condition. Cool the pellets in the horizontal pellet cooler.

3.4 Separate fines generated in the pelleting process using a 6.2 mm screen. NOTE: Measure the moisture content and durability of the pellets after cooling[21].

3.5 Dry the cooled high-moisture pellets in a laboratory oven at 70 °C for 3–4 hr to reduce the final moisture content of the pellets to less than 9% (w.b.). NOTE: Measure the pellet moisture content, bulk density, and durability of dried pellets[21].

3.6 Log the power data into a computer during the pelleting process. NOTE: See Table 2 for pelleting test conditions and Figure 2 for pellet produced at 33, 36 and 39% moisture content and 4 % corn starch binder addition.

[Place *Table 2* here]

[Place *Figure 2* here]

4. Pellet Properties and Specific Energy Consumption

NOTE: ASABE standards[43] were used for measuring the moisture content, density, durability and percent fines of raw and pelleted materials.

4.1 Place about 25-50 g of the ground and pelleted corn stover samples in the laboratory oven set at 105 °C for 24 hrs. Weigh the sample before and after drying. Calculate the moisture content using equation 2. Conduct the experiments in triplicate.

$$Moisture\ content\ (\%, w.b.) = \frac{Wet\ weight\ of\ the\ sample\ (g) - Weight\ of\ sample\ after\ drying\ (g)}{Wet\ weight\ of\ the\ sample\ (g)} \times 100 \qquad (2)$$

4.2 Take a single pellet and smooth both the ends with Grit Utility Cloth. Measure the pellet diameter using Vernier calipers. Calculate expansion ratio of the pellet using equation 3[28]. Measure the diameter of the ten pellets.

$$\text{Expansion ratio} = \frac{D^2}{d^2} \qquad (3)$$

NOTE: In equation 3, D is the diameter of the pellet extruded (mm) and d is the diameter of the die (mm).

4.3 Use a plexi glass cylinder with a height of 155 mm and a diameter of 120 mm. Pour the pellets into the cylinder until it overflows and level the top surface with a straight edge. Weigh the cylinder with the material. Divide the weight of the cylinder with the volume of the cylinder to calculate bulk density. Repeat the experiments three times.

4.4 Hand sieve the pelleted-material using a 6.2 mm screen. Weigh the material that has passed through the screen. Calculate percent fines using equation 4.

$$\text{Percent fines} = \frac{\text{Weight of material that has passed through 6.2 mm screen}}{\text{Weight of the pelleted sample (fines+pellets)}} \times 100 \quad (4)$$

4.5 Place approximately 500 g of the pellets without fines into each compartment of the pellet durability tester. Tumble the pellets at 50 rev/min for 10 min. Sieve the tumbled material using a 6.2 mm screen. Use equation 5 to calculate the percent durability of the pellets.

$$\text{Durability} = \frac{\text{Weight of pellets after tumbling}}{\text{Weight of pellets before tumbling}} \times 100 \quad (5)$$

Note: Green durability is the durability of the pellets measured after cooling, and cured durability is the durability measured after drying the pellets at 70 °C for 3 hr.

4.6 Log the pellet mill power consumption using data logging software. Record the no load power (kW) data of the pellet mill by running the pellet mill empty at 60-Hz die speed. Use equation 6 to calculate the specific energy consumption (SEC).

$$\text{SEC} = \frac{(\text{Full load power (kW)} - \text{No load power (kW)}) * \text{time (hr)}}{\text{weight of biomass material (kg)}} \times 1000 = \frac{\text{kWhr}}{\text{ton}} \quad (6)$$

REPRESENTATIVE RESULTS:

Pellet Moisture Content

The moisture content of the biomass was reduced by about 5–8% (w.b.) after pelleting. This reduction is mainly attributed to frictional heat developed in the die, and preheating temperature and cooling of the high moisture pellets. Also, binders had an impact on the amount of moisture lost. At 0% binder, the loss of moisture was about 7–8%, which agrees with our earlier studies[21,28]; whereas, at 4% binder, the loss of moisture in the feedstock during pelleting was about 3–5% (**Figure 3**). The binder added to the biomass might have acted as lubricating agent. This may have reduced the frictional resistances and reduced the residence time of the material in the die channel causing the decrease in moisture loss. In previous studies die temperature measured immediately after pelleting using an infrared thermometer (Fluke, Model 561, Fluke Corporation, Everett, WA, USA) reached to about 100-110 °C[21]. Increasing the binder percentage reduced the moisture loss as the moisture might have been tightly bound to the starch granules. The high moisture pellets that were further dried in a laboratory oven at 70 °C for 3-4 hr had moisture contents >9% (w.b.), and these pellets were used to measure other physical properties like pellet diameter, expansion ratio, bulk density and durability. Statistical analysis of the pellet moisture content data indicated that there was an interactive effect of feedstock moisture content and binder addition on the pellet moisture content (**Table 3**). For pellets with no binder and 2% binder, an increase in feedstock moisture content caused an increase in pellet moisture content (Tukey's $p<0.05$), but this trend was not statistically significant at 4% binder (Tukey's $p \geq 0.05$; **Figure 3**).

[Place *Figure 3* here]

Pellet Diameter

The diameter of the pellets at 33% moisture content with and without binder addition was in the range of 8.4-8.7 mm after cooling (data not shown). Increasing the feedstock moisture content to 36 and 39% (w.b.) with added binder increased the pellet diameter to maximum values of 9.3 mm (data not shown). These pellets were further dried in a laboratory oven at 70 °C for about 3-4 hr. Drying resulted in a decrease in pellet diameter by about 0.3-0.4 mm. The major reason for a decrease in diameter after drying was due to contraction of the pellets. There was a statistically significant effect of the interaction between feedstock moisture content and binder addition on pellet diameter after drying (**Table 3**). At 33% feedstock moisture content the pellet diameter after drying was in the range of 8.3 to 8.5 mm, whereas increasing the feedstock moisture content to 36% or 39% increased the pellet diameter to about 8.7 mm (**Figure 4**). This increase was only statistically significant between 33%-39% when no binder was used (Tukey's $p<0.05$), likely because of the high deviations in the measurements.

[Place *Figure 4* here]

Expansion Ratio

Expansion ratio is calculated using the pellet diameter (equation 3). The expansion ratio values were higher for the pellets after cooling compared to after drying (data not shown). At 33% moisture content without and with binder addition, the expansion ratio values after cooling were in the range of 1.16-1.20. Further increasing the moisture content to 36 and 39% without binder addition increased the expansion ratio values to 1.35. The dried pellets had lower expansion ratios, which was mainly due to contraction of the pellets both diametrically and laterally. At 33% feedstock moisture content the expansion ratio values with and without binder addition were in the range of 1.11- 1.07 (**Figure 5**). Increasing the feedstock moisture content to 36 and 39% further increased the expansion ratio values to 1.10-1.18 (**Figure 5**); however, this was only statistically significant for 33% compared to 39% moisture content with no binder addition (Tukey's $p<0.05$; **Table 3**). In the case of pellet diameter and expansion ratio, adding a starch based binder increased these values at all of the feedstock moisture contents, but these differences were not statistically significant (Tukey's $p\geq0.05$). The expansion ratio results after drying corroborate the findings of earlier studies, where increasing feedstock moisture increased the expansion ratio and further decreased the bulk density values[28].

[Place *Figure 5 here*]

Bulk Density

The bulk density of the pellets made with a feedstock moisture content of 33% with and without binder and measured after cooling was in the range of 464-514 kg/m^3 (data not shown). At 36 and 39% feedstock moisture content without binder the bulk density values were in the range of 437-442 kg/m^3. Adding binder at these feedstock moisture contents reduced bulk density to <400 kg/m^3. Drying the high-moisture pellets in a laboratory oven at 70 °C for about 3 hours reduced the moisture contents of the pellets to less than 9% (w.b.). There was a slight increase in the bulk density values by about 50 kg/m^3 after drying. The probable reason for the increase in bulk density after drying could be due to lower inter-particle liquid bridges, which might have kept the particles closer with less-open structure. Oginni[45] observed that the bulk density of ground Loblolly pine decreased with an increase in moisture content. For pellets made with a feedstock moisture content of 33% with and without the binder addition, the bulk density of the pellets was in the range of 520-530 kg/m^3 (**Figure 6**). At higher feedstock moisture contents of 36 and 39% (w.b.), the bulk density of the dried pellets significantly decreased to <434 kg/m^3 and <437 kg/m^3, respectively. There was a statistically significant effect of the interaction between feedstock moisture content and binder addition on bulk density (**Table 3**). In general, bulk density decreased with an increase in starting feedstock moisture content. In addition, there is some indication that bulk density decreased with an increase in starch content (Tukey's p<0.05; **Figure 6**).

[Place *Figure 6* here]

Durability (%)

After cooling (green durability)

Figure 7 shows the durability of pellets after cooling (green strength) and after drying in an oven at 70 °C for 3–4 hours (cured strength). Higher durability values of high moisture pellets are desirable as it will result in less breakage during handling and storage due to shear and impact resistances. For the ANOVA, the interaction was significant between feedstock moisture content, binder percent, and drying (**Table 3**). The durability values of the pellets after cooling increased with an increase in binder content (**Table 3**; Tukey's p<0.05). At 33% (w.b.) feedstock moisture content, the durability values without binder were about 87.2%; whereas, with the addition of a 2 and 4% starch binder, the durability values increased to 93.2 and 96.1% (**Figure 7**). The trend was similar for the other feedstock moisture contents of 36 and 39% (w.b.). Without binder the durability values were about 80%; however, adding binder to the biomass increased the durability values. The durability increased to about 90% when pellets were made with a feedstock moisture content of 36% and 4% starch binder. At even higher feedstock moisture content of 39% (w.b.) the trend was similar, but the overall durability values decreased compared to the other feedstock moisture contents.

[Place *Figure 7* here]

After Drying (cured durability)

Drying of the high-moisture pellets in a laboratory oven at 70 °C for 3–4 hours resulted in curing of the pellets, thereby increasing the durability of the pellets. The durability values of the pellets made at 33, 36 and 39% (w.b.) feedstock moisture content increased to >92% (**Figure 7**). The durability values at 33% feedstock moisture content increased to about 98% after drying (**Figure 7**). These results match closely with the earlier work[21,28]. The durability values of the pellets made using a binder increased after drying (Tukey's p<0.05). At 33% feedstock moisture content and 4% binder, the final durability values observed were about 98%. The trend was similar at 36 and 39% feedstock moisture content, where the binder had a positive impact on the durability values (Tukey's $p<0.05$). At 39% feedstock moisture content with a binder addition of 2 and 4%, the durability values increased to about 94–95%.

Percent Fines

In the present study, the percent fines generated during pelleting were higher at 36 and 39% (w.b.) compared to 33% (w.b.) feedstock moisture content. Adding binders resulted in lowering the percent fines generated at all feedstock moisture contents when compared to tests with no binder addition (**Figure 8)**. Pelleting tests conducted with no binder showed the highest percent fines of about 11% at 39% (w.b.) feedstock moisture content. Adding 2 and 4% binder to the corn stover, decreased the percent fines generated during pelleting for 33% and 36% (w.b.) compared to pellets with no binder added. The lowest percent fines observed in this study were at 4% binder addition and 33% (w.b.) feedstock moisture content (approximately 3%).

[Place *Figure 8* here]

Specific Energy Consumption

The specific energy consumption was influenced by binder addition (**Figure 9**). With no binder, the specific energy at 33, 36, and 39% feedstock moisture content was between 118-126 kWhr/ton. Adding a 2% binder reduced the specific energy consumption to about 75-94 kWhr/ton. Further increasing the binder percentage to 4% further reduced the specific energy consumption to about 68–75 kWhr/ton for all feedstock moisture contents that were tested. Adding the binder at 2 and 4% reduced the specific energy consumption by about 20-40%.

[Place *Figure 9* here]

STATISTICAL ANALYSIS:

Statistical analysis was completed in JMP 10[44]. A two-way ANOVA was used to determine the effects of feedstock moisture content (33, 36, 39%) and corn starch binder (0, 2, 4%) on pellet moisture content ($n=3$), pellet diameter ($n=10$), expansion ratio ($n=10$), and bulk density ($n=3$). A three-way ANOVA was used to determine the effects of moisture content (33, 36, 39%), corn starch binder (0, 2, 4%), and drying (before drying, after drying) on durability ($n=3$). Residuals met the ANOVA assumptions for normality and homogeneity of variance. To meet these assumptions, pellet moisture content was transformed by raising the data to the 4th power. If the factors tested in the ANOVA were significant at $p<0.05$, Tukey HSD tests were used for post hoc pairwise comparisons.

FIGURE AND TABLE LEGENDS

Figure 1. Schematic of a laboratory scale flat die pellet mill at Idaho National Laboratory (adapted from Tumuluru[21]). Flat die pellet mill was used to conduct the high moisture corn stover pelleting tests with and without binder addition.

Figure 2. Photograph of the corn stover pellets produced with 4 % corn starch binder at different feedstock moisture contents.

Figure 3. Effect of feedstock moisture content (FMC) and starch binder on pellet moisture content after cooling (mean±1SD; n=3). Pelleting tests conducted without binder resulted in higher feedstock moisture content loss compared to tests conducted with binder. Different letters indicate significant differences using post hoc Tukey HSD tests ($p<0.05$).

Figure 4. Effect of feedstock moisture content (FMC) and corn starch binder on pellet diameter after drying (mean±1SD; n=10). Pellet diameter increased with an increase in feedstock moisture content and starch addition. Different letters indicate significant differences using post hoc Tukey HSD tests ($p<0.05$).

Figure 5. Effect of feedstock moisture content (FMC) and starch based binder on the expansion ratio of pellets after drying (n=10). Expansion ratio of pellets increased with an increase in feedstock moisture content and with binder addition. Different letters indicate significant differences using post hoc Tukey HSD tests ($p<0.05$).

Figure 6. Effect of feedstock moisture content (FMC) and starch binder on the bulk density of pellets after drying (mean±1SD; n=3). Lower feedstock moisture content of 33% (w.b.) and no binder resulted in the highest bulk density. Adding 2 and 4% binder at different feedstock moisture contents resulted in lower bulk density values. Different letters indicate significant differences using post hoc Tukey HSD tests ($p<0.05$).

Figure 7. Effect of feedstock moisture content (FMC) and starch binder on durability after cooling and after drying (mean±1SD; n=3). Durability values of high moisture corn stover pellets produced at 33, 36 and 39% (w.b.) feedstock moisture content increased with binder addition both after cooling and after drying. Different letters indicate significant differences using post hoc Tukey HSD tests ($p<0.05$).

Figure 8. Effect of feedstock moisture content and starch binder on the percent fines produced from the pelleted material. At feedstock moisture contents of 33, 36 and 39 % (w.b.) addition of binder reduced the percent fines in the pelleted material.

Figure 9. Effect of feedstock moisture content and starch binder on the specific energy consumption of the high moisture pelleting process. Specific energy consumption of the high moisture corn stover pelleting process reduced by about 20-40% with addition of 2 and 4 % starch based binder.

Table 1. Moisture content and bulk density of ground corn stover and corn starch binder.

Table 2. Experimental test conditions used in the present study.

Table 3. Statistical significance of the process variables based on analysis of variance (ANOVA).

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DISCUSSION:

The critical steps in the high moisture pelleting method to produce the desired durability pellets at lower specific energy consumption are: 1) drying the high moisture corn stover to the desired moisture levels (33-39%, w.b.), 2) percent binder addition and 3) feeding high moisture biomass uniformly into the pellet mill. Feedstock moisture and percent binder are process variables that influenced the pellet properties (density and durability of the pellets before cooling and after drying) and specific energy consumption of the pelleting process. It is recommended to test the moisture content of the feedstock before it is used for pelleting studies. Feeding of high moisture corn stover at 33, 36 and 39% (w.b.) uniformly to the pellet mill has an impact on quality and energy consumption. Modifying the pellet mill feeder with a variable frequency drive was essential to feed the biomass uniformly to the pellet mill.

Results from the present study indicated that adding binder to the high moisture corn stover did reduce the bulk density of the pellets marginally, but improved the durability significantly. Adding a starch based binder increased the moisture content in the pellets after compression and extrusion, but the increase was found not to be statistically significant in almost all the cases studied. The loss of moisture during pelleting was about 3 to 4% at the addition of 4% binder, whereas it was higher (7–8%, w.b.) without the binder. The addition of a binder to the corn stover might have 1) reduced the residence time of the material in the die and 2) reduced the frictional resistances in the die, thereby reducing the die temperature, which might have resulted in less moisture loss during compression and extrusion in the pellet die.

There was an increase in the pellet diameter after it was extruded from the pellet die and dried (**Figure 4**). This increase was greater at higher feedstock moisture content and with starch binder addition. The bulk density of the pellets was in the range of 510-530 kg/m^3 at 33% (w.b.) feedstock moisture content with and without a binder. Previous research has indicated that higher feedstock moisture content of about 38% (w.b.) results in lower bulk density, mainly due to expansion of the pellets as they exit through the die[21,28]. It is a common phenomenon that when high-moisture biomass material is extruded through the die under pressure it results in moisture flash-off[12,21]. The moisture flash-off gives way to the expansion of the pellet, both in the axial and diametrical direction. In general, the diametrical expansion is more prominent compared to axial expansion. Another reason for the expansion behavior of biomass after compression and extrusion through the pellet die could be that biomass fibers relax in the presence of moisture content. Ndiema et al.[46] and Mani et al.[18] indicated that release of the applied pressure in a die results in relaxation of the compressed biomass. The relaxation characteristics are dependent on many factors like particle size, feedstock moisture content and applied pressure. Also, in this study we have observed that the bulk density increases after drying, which could be due to lower inter-particle liquid bridges that might have kept the particles closer and produced a less-open structure. Oginni[45] observed that the bulk density of ground Loblolly pine decreased with increased moisture content.

Durability of the pellets was measured to understand the strength of the pellets. Generally, pellets are subject to shear and impact resistances during storage, transportation, and the handling process[4,47]. Kaliyan and Morey[48] suggested that durability of pellets produced immediately after production (green strength) is different than durability of the pellets that are stored for a few days after production (cured strength). Pellets with lower durability values break and increase the risk of storage issues, such as off-gassing and spontaneous combustion that could cause revenue loss for pellet manufacturers. According to the European Committee for Standardization (CEN) and the United States Pellet Fuels Institute (PFI) the recommended values of the durability are >96.5% for high quality or premium grade pellets[31]. In this study, the durability values increased to approximately 94-95% when pelleted with a starch binder at 39% moisture content compared to pellets made with no binder that had durability values in the range of 83-85% after drying. The pellets produced at 33% (w.b.) feedstock moisture content had durability values >96.5% and meet the international standards.

Moisture has different functions during biomass pelleting, including: 1) solid bridge formation between the biomass particles due van der Waals forces, 2) activating natural binders like protein, starch and lignin present in the biomass, and 3) promoting starch and protein based reactions like gelatinization and denaturation that have a strong impact on the textural properties, such as hardness[4-12]. In the case of lignocellulosic biomass, the main binding agent is lignin (woody biomass: 27–33%, herbaceous biomass: 12–16%)[4]. Lignin content in corn stover was determined to average around 16% based on a review of composition data, including literature sources and feedstock databases[49]. Lignin molecules, which have higher mobility at higher moisture content, act as an adhesive and result in stronger binding; however, at very high levels the moisture will act more like a lubricant resulting in less binding. In the present study, at very high moisture content of about 39% (w.b.) moisture might have acted more like a lubricant and resulted in low durability and more fines generation in the pellet production process. Higher durability values were observed by the addition of a binder at a higher feedstock moisture content of 36 and 39% (w.b.), which could be caused by gelatinization of starch in the presence of die temperature and feedstock moisture content. These gelatinization reactions can lead to the formation of cross linking of starch with the other biomass components.

The percent fines generated during the pelleting process is a good indicator for how well biomass will form pellets. Generation of fine particles during the pelleting process results in product and revenue loss to the pellet producer. Excessive fine generation during pelleting processes can also have an impact on quality attributes like density and durability. The fines generation during the pellet production process is influenced by biomass composition (i.e., starch, protein, lignin, and waxes), pellet mill process variables like length to diameter ratio (L/D ratio), die rotational speed, steam condition, preheating, and feedstock variables (i.e., feedstock moisture content, particle size and feed rate) [4]. The present results indicate that the addition of binder not only reduces the percent of fine particles generated, but also helps to improve the physical properties while reducing the specific energy consumption. Lower percent fines generated indicate that the biomass has a greater pelletability.

Tumuluru et al.[4] in their review on densification systems suitable to make biomass into a commodity type product indicated that adding binder helps to reduce the extrusion energy, which results in reducing the specific energy consumption. Typically, length to diameter (L/D) ratio controls the residence time of the material in the die and helps the binding of the biomass. Also, L/D ratio controls the extrusion energy and the specific energy consumption. Higher L/D ratio increases the residence time, which improves the physical properties of the pellets, but increases the energy necessary for extrusion. Adding a binder to biomass can help bind the biomass at lower L/D ratio and reduce the extrusion energy. In this study, constant length to diameter (L/D) ratio (2.6) was selected. Future research is aimed at understanding the effect of L/D ratio of the pellet die and its interaction with feedstock moisture content on pellet quality attributes.

The experimental data on biomass preprocessing (grinding, drying and pelleting) obtained from the Biomass National User Facility (https://www.inl.gov/bfnuf/) located at INL and associated techno-economic analysis indicated that drying biomass from 30-10% (w.b.) consumes a large amount of energy (unpublished data). The high moisture pelleting process developed at INL can help reduce the pellet production cost compared to a conventional pellet production method[24]. The present study indicated that adding a starch-based binder to a high moisture pelleting process improved the durability of the pellets to >92% after cooling at the feedstock moisture contents of 36 and 39% (w.b.), and it also reduced the specific energy consumption of the pelleting process by about 30-50%. Greater durability values of the pellets made at higher feedstock moisture is important as they can be handled efficiently by conveyors. Typically low durability pellets crumble to fines during handling and storage which results in revenue loss for the pellet producers. In addition, fines generated in the process can result in safety hazards like spontaneous combustion and off-gassing[28,42]. The specific energy reduction by approximately 40-50% using a binder outweighs the cost of the binder. Also, based on this study we can conclude that some of the starch wastes from food processing industries could be used for biomass pelleting for bioenergy applications. Currently, the high-moisture pelleting process was demonstrated using a laboratory scale flat die pellet mill. The protocol described here for the laboratory-scale pellet mill will be the basis for developing scale-up models and for testing the process in pilot-scale and commercial-scale pellet mills.

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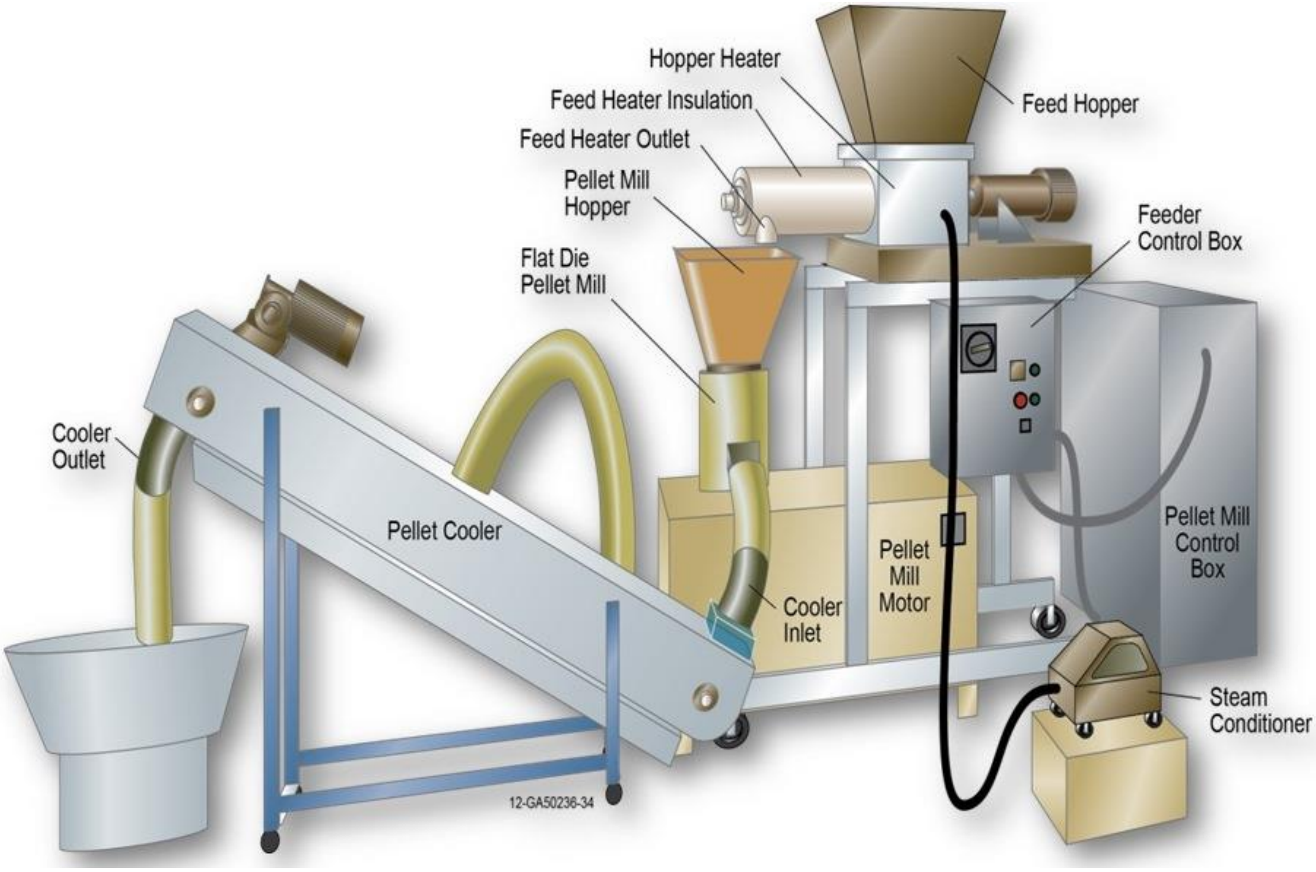
Figures Jove

Figure 1



Figures Jove

Figure 2

Figure 3

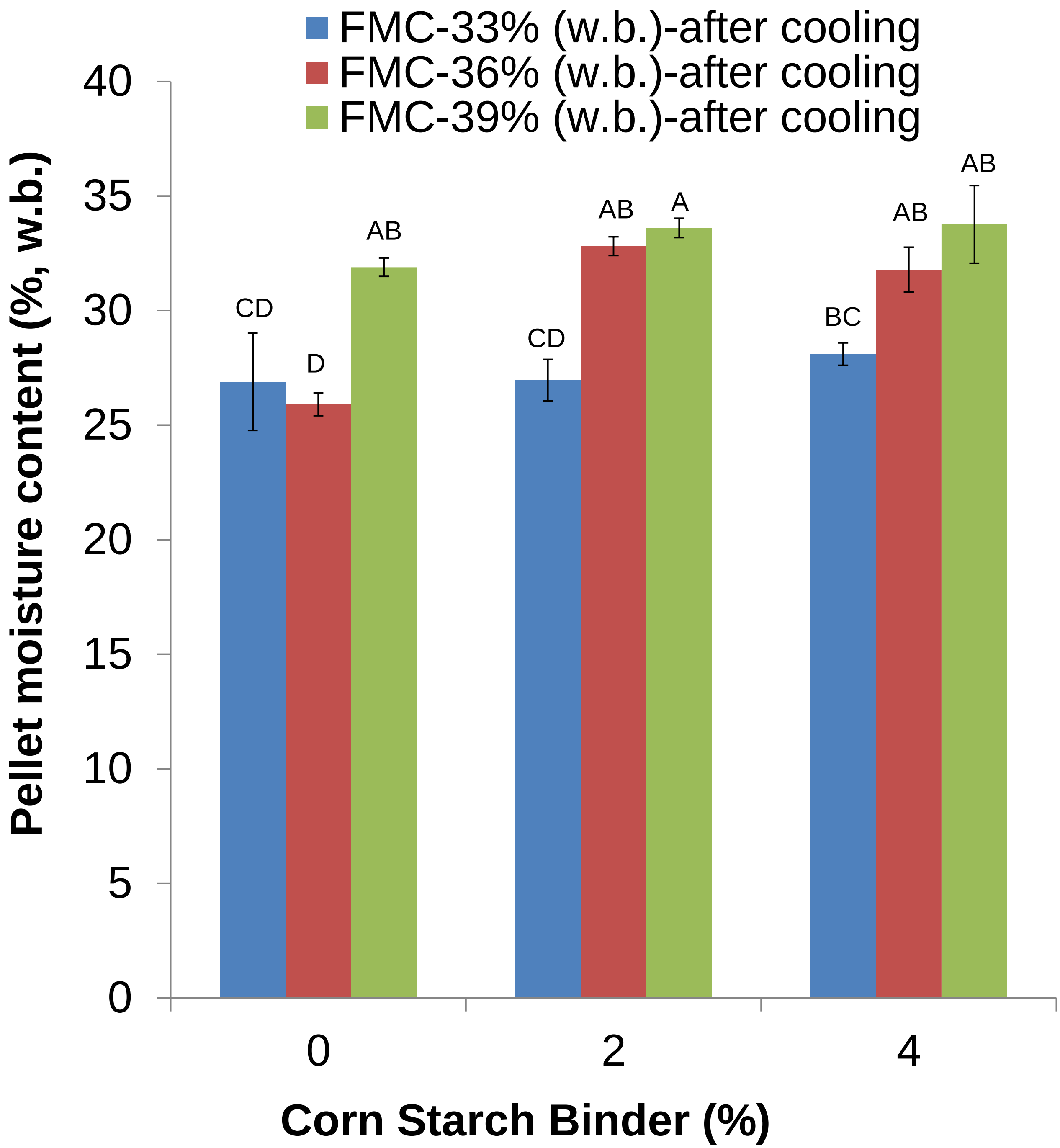

FMC-33% (w.b.)-after cooling
FMC-36% (w.b.)-after cooling
FMC-39% (w.b.)-after cooling
Pellet moisture content (%, w.b.)
0
5
10
15
20
25
30
35
40
CD
D
AB
CD
AB
A
BC
AB
AB
0
2
4
Corn Starch Binder (%)

Figure 4

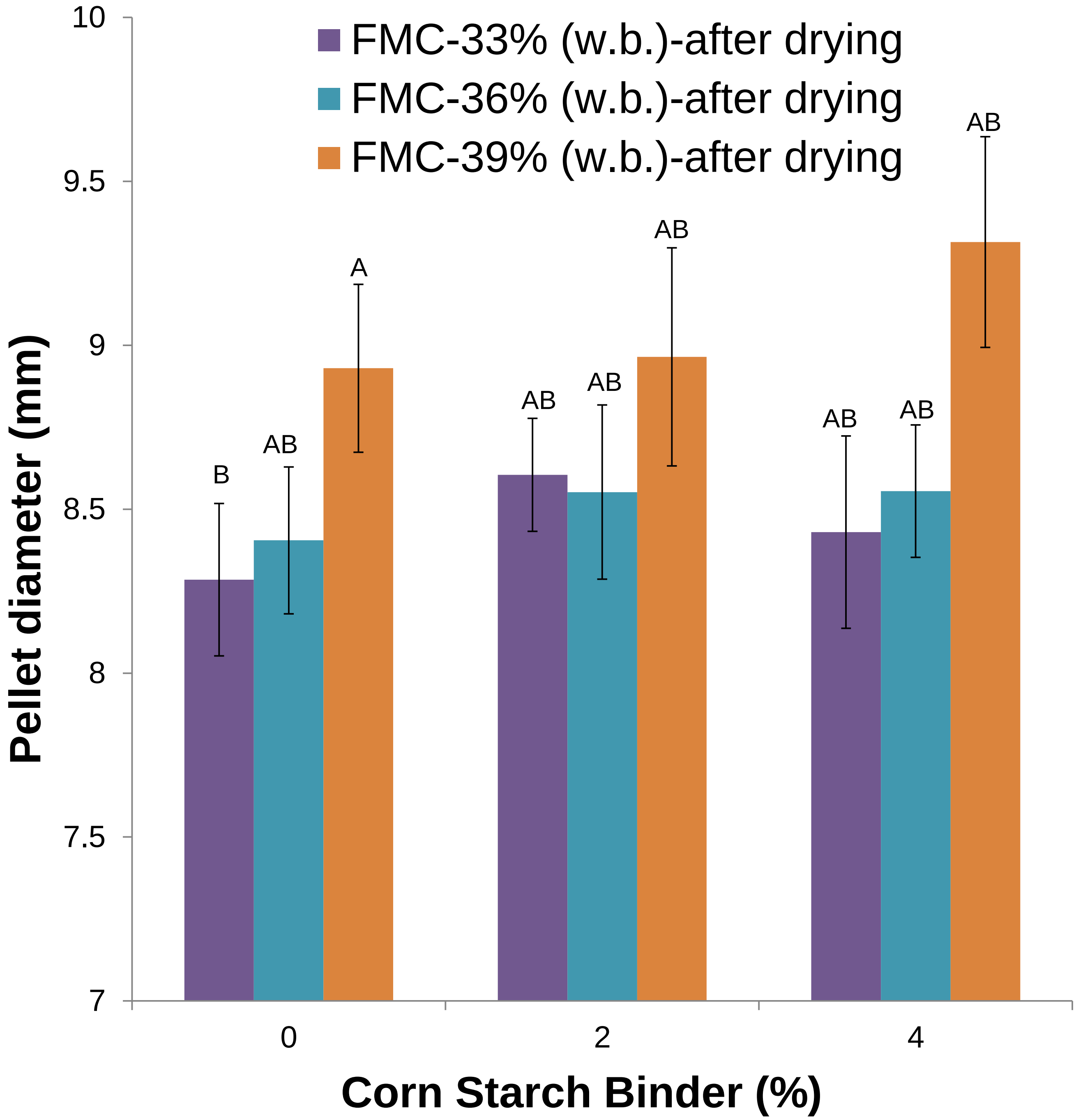

10
9.5
9
8.5
8
7.5
7
FMC-33% (w.b.)-after drying
FMC-36% (w.b.)-after drying
FMC-39% (w.b.)-after drying
B
AB
A
AB
AB
AB
AB
AB
AB
0
2
4
Pellet diameter (mm)
Corn Starch Binder (%)

Figure 5

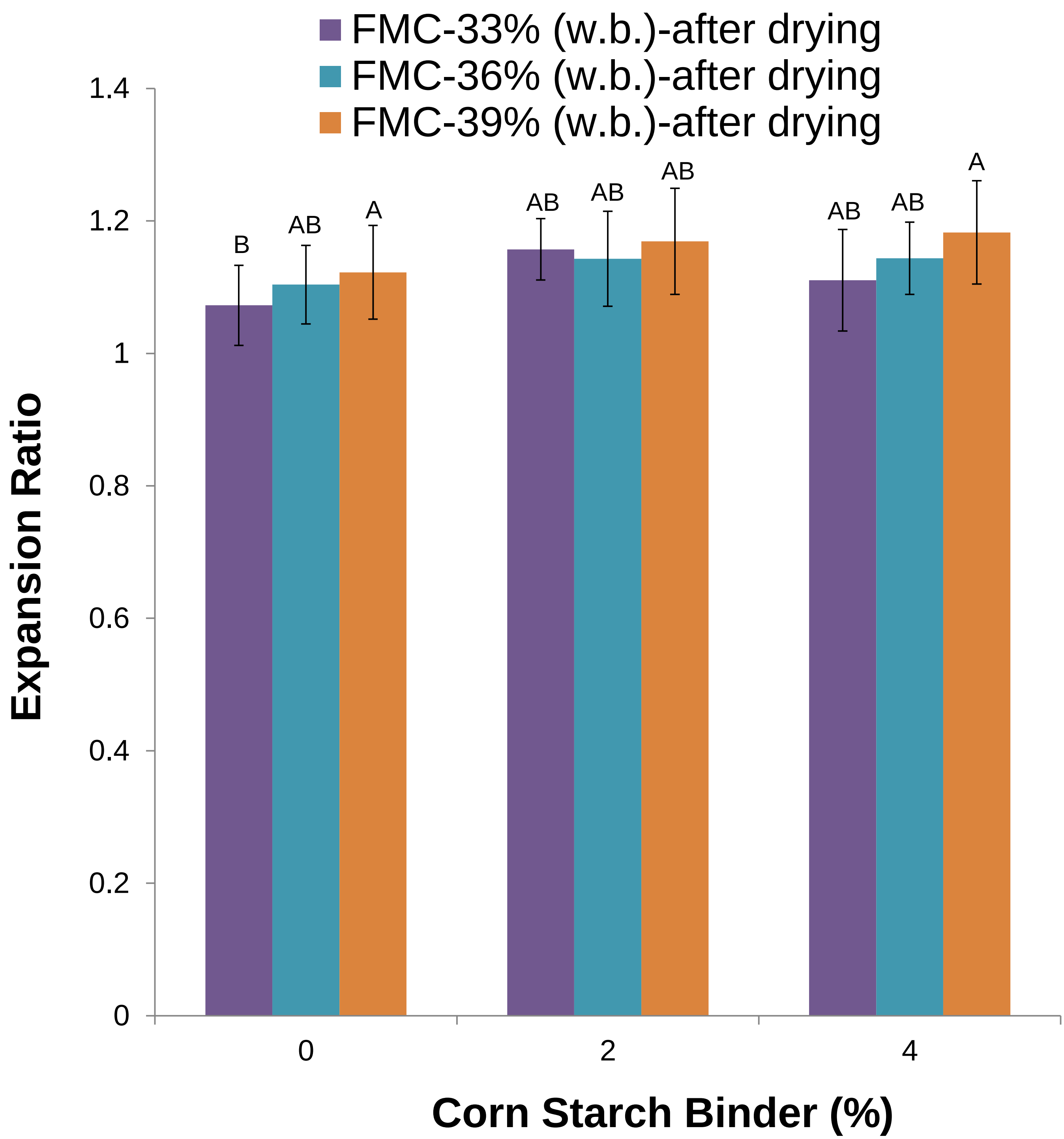

FMC-33% (w.b.)-after drying
FMC-36% (w.b.)-after drying
FMC-39% (w.b.)-after drying
Expansion Ratio
1.4
1.2
1
0.8
0.6
0.4
0.2
0
B
AB
A
AB
AB
AB
AB
AB
A
0
2
4
Corn Starch Binder (%)

Figure 6

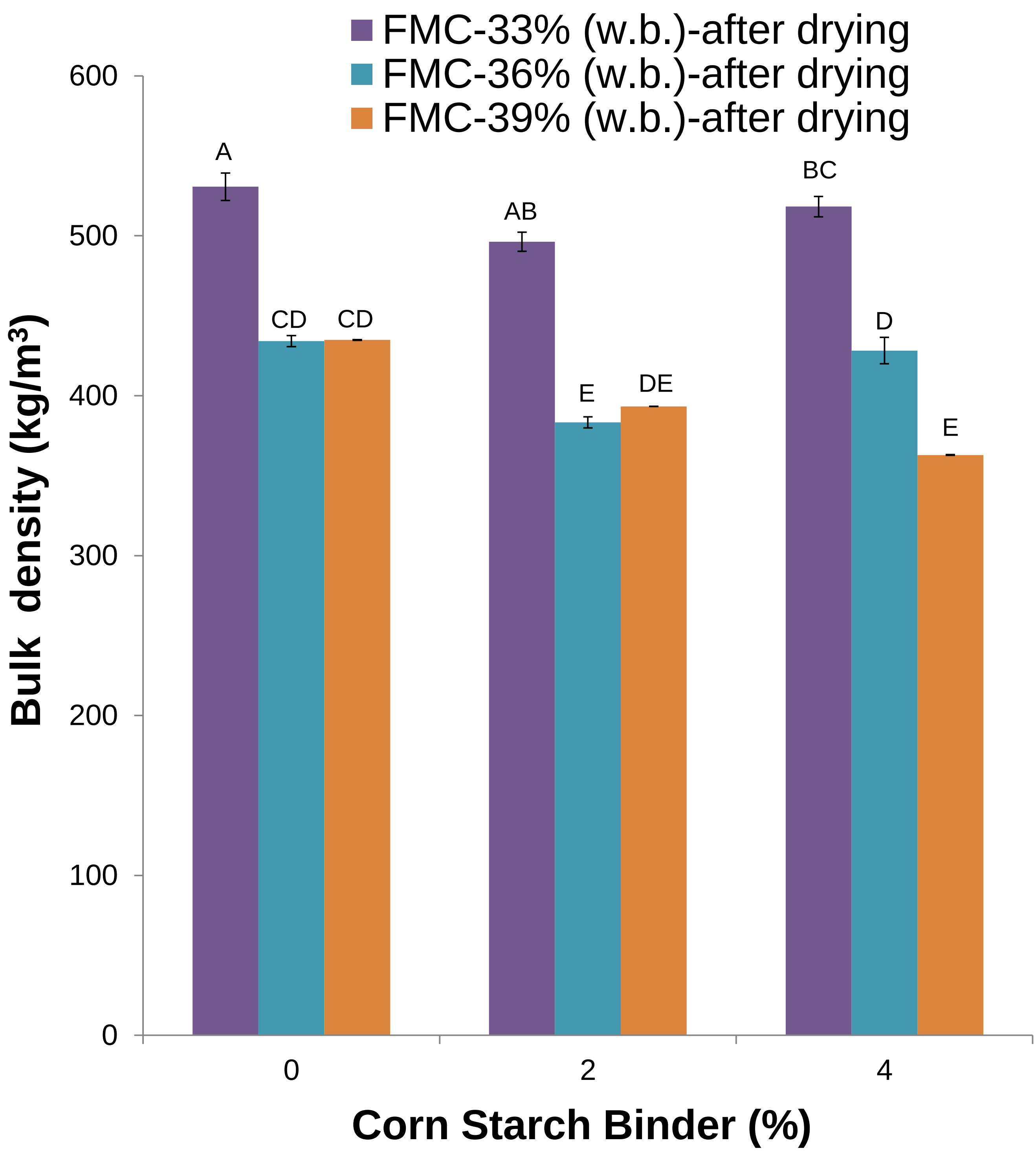

FMC-33% (w.b.)-after drying
FMC-36% (w.b.)-after drying
FMC-39% (w.b.)-after drying
600
500
400
300
200
100
0
A
CD
CD
AB
E
DE
BC
D
E
Bulk density (kg/m³)
0
2
4
Corn Starch Binder (%)

Figure 7

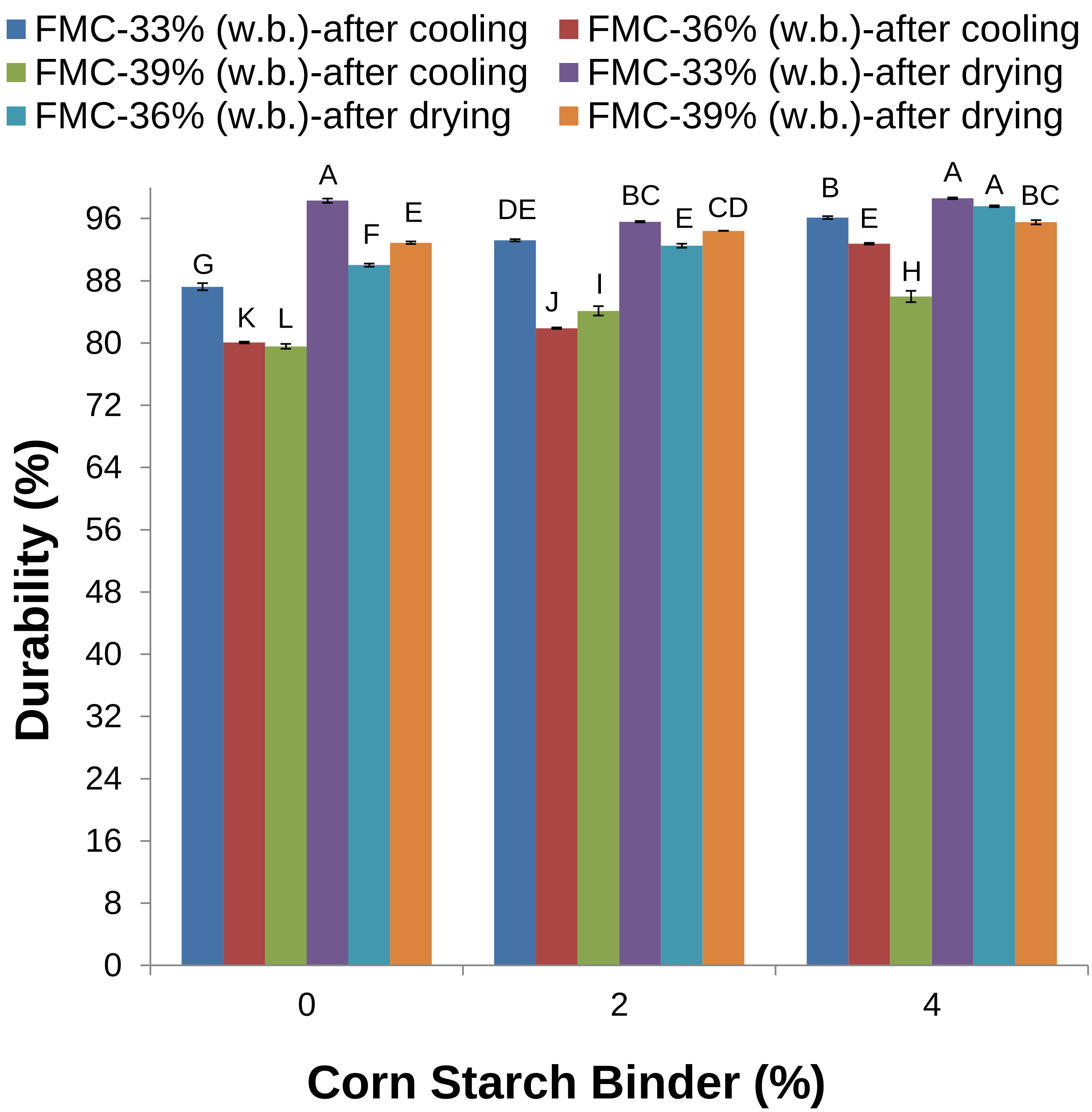

FMC-33% (w.b.)-after cooling
FMC-36% (w.b.)-after cooling
FMC-39% (w.b.)-after cooling
FMC-33% (w.b.)-after drying
FMC-36% (w.b.)-after drying
FMC-39% (w.b.)-after drying
Durability (%)
Corn Starch Binder (%)
0
2
4
0
8
16
24
32
40
48
56
64
72
80
88
96
G
K
L
A
F
E
DE
J
I
BC
E
CD
B
E
H
A
A
BC

Figure 8

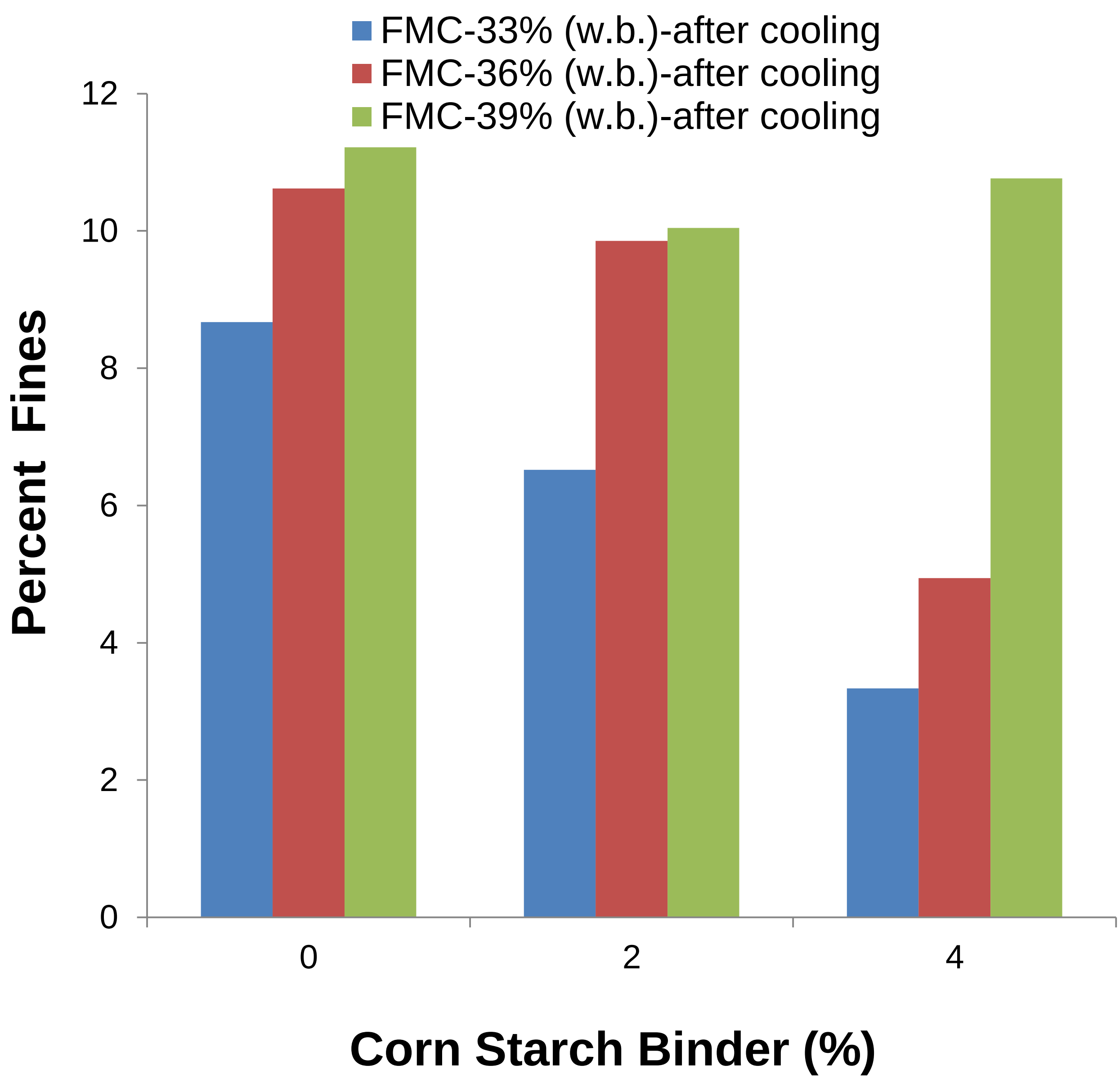
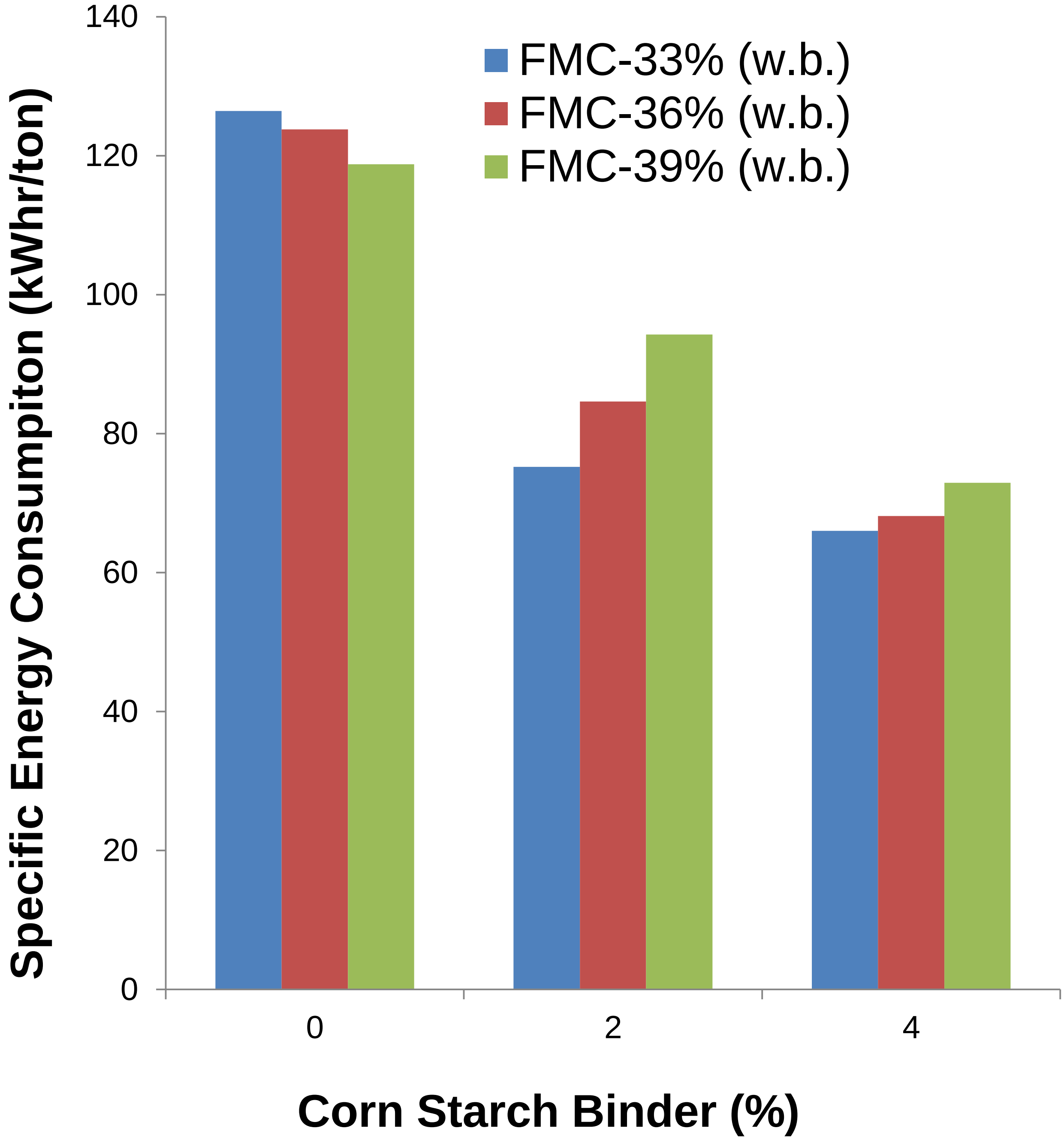

FMC-33% (w.b.)-after cooling
FMC-36% (w.b.)-after cooling
FMC-39% (w.b.)-after cooling
12
10
8
6
4
2
0
Percent Fines
0
2
4
Corn Starch Binder (%)

Figure 9



140
120
100
80
60
40
20
0
Specific Energy Consumpiton (kWhr/ton)
FMC-33% (w.b.)
FMC-36% (w.b.)
FMC-39% (w.b.)
0
2
4
Corn Starch Binder (%)

Table 1

Feedstock	Moisture content (%, w.b.)	Bulk density (kg/m^3)
Ground corn stover (4.8 mm screen)	8.39 (0.03)	111.0 (2.3)
Corn starch binder (%)	9.72 (0.02)	597.1 (31.3)

Table 2

Pelleting process variables	
Feedstock moisture content (%, w.b.)	33, 36, and 39
Corn starch binder (%)	0, 2, and 4

Table 3

Source of Variation	Measured Values				
	Moisture Content	Bulk Density	Durability	Diameter	Expansion Ratio
Feedstock Moisture Content (FMC)	<0.0001	<0.0001	<0.0001	0.0464	0.0184
Binder	0.0002	<0.0001	<0.0001	0.3833	0.1893
Drying	NA	NA	<0.0001	NA	NA
FMC*Binder	0.0019	0.0022	<0.0001	0.0240	0.0405
FMC*Drying	NA	NA	<0.0001	NA	NA
Binder*Drying	NA	NA	<0.0001	NA	NA
FMC*Binder*Drying	NA	NA	<0.0001	NA	NA

Name of Reagent/ Equipment	Company
Flat pellet mill	Colorado Mill Equipment, Canon City, CO, USA
Heating tapes	BriskHeat, Columbus, OH, USA
Thermocouples	Watlow, Burnaby, BC, Canada
Variable frequency drive	Schneider Electric, Palatine, IL, USA
Power meter	NK Technology, USA
Pellet cooler	Colorado Mill Equipment, Canon City, CO, USA
Data logging software	National Instruments Corporation, Austin, TX, USA
Durability tester	Seedburo Equipment Co., Des Plaines, IL 60018, USA
Hammer mill	Bliss Industries
Grinder	Vermeer
Horizontal mixer	Colorado Mill Equipment, Canon City, CO, USA
Blue Grit Utilty Cloth	3M
Insulation materail	McMaster Carr
Feeder controller	KB Electornics, INC
Dust exhaust system	Delta
Vernier Calipers	VWR® Digital Calipers
Binder	ACH Food Companies Inc., Memphis, TN, USA
Corn stover	Harvested in Iowa and procurred in bale form

Catalog Number	Comments/Description
ECO-10 pellet mill	
Silicon Rubber Heater, Etched foil elements	
J-type	
Altivar 71	
Model No: APT‑48T‑MV‑220‑420	
CME ECO-HC6	
Labview software	
Pellet durability tester	
CME ECO-HC6	
HG200	
ECO-RB 500	
Part No.05107-150J grade	
Flexible Fiberglass Insulation	
KBIC-DC-MTR Direct Current motor controller	
Model No: 50-763, Serial No: 2010 11OI1415	
Part Number: 12777-830	
ARGO 100 % pure corn Starch,	

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Author(s): Jaya Shankar Tumuluru, Craig C Conner, & Amber Hoover

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Name: Jaya Shankar Tumuluru
Department: Biofuels and Renewable Energy Technology
Institution: Idaho National Laboratory
Article Title: Original Research
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•Formatting (References): Please check that doi information has been added where applicable.

•Additional detail is required
-1.2-Approximately how long should the corn stover be ground using these different screens?
Reply: Typically in pellet production process the grinding is done sequentially (stage-1 & stage-2 grinder). The material what has gone through stage-1 fitted with a bigger screen (50.8 mm) is further passed immediately through stage-2 grinder fitted with a smaller screen (4.8 mm screen size). Information included in the revised manuscript (Page 5 lines 211-215).

-1.3-If this step is to be filmed, please provide additional details as to how to determine the moisture content.
Reply: Moisture content method is included in section 4 of protocol (page 7, lines 306; Page 8, line 307-310)

-2.2-Provide additional details as to where/how to attach the tapes/controllers to the hopper and screw feeder. In addition, what are the "desired temperatures"?
Reply: Additional information on tape heaters and controllers is included in the revised manuscript (page 6, lines 228-230).

-2.3/2.4-Please expand on how the frequency drive is "equipped", and the power meter "provided."
Reply: Included in the revised manuscript (page 6, lines 232-233).

-3.7-If this step is to be filmed, please provide additional details as to how to measure moisture content, etc.
Alternatively, include this step as a 3.6 Note.
Reply: Method for measuring the moisture content is included in the revised manuscript in section 4. This has been included as step 3.7 in the revised manuscript (Page 8, line 307-311).

-4.1-Please provide details as to how to assess moisture content.
Reply: Included in the revised manuscript (page 8, line 311)

•Unnecessary branding should be removed from steps 3.8 and 4.5 (LabVIEW).
Reply: Removed in the revised manuscript.

•Results: Please include error bars for all figures (Figure 5/6), and define whether they represent SEM or SD in the accompanying legends.
Reply: The error bars were included for graphs representing pelleting moisture content, pellet diameter, expansion ratio, bulk density and durability. In the case of percent fines and specific energy consumption, the error bars are not included as the data is from single pellet mill run from steady state pelleting condition. We have published this method in other high impact factor peer reviewed journals like Bioresource Technology, Biosystems Engineering and Energy Science and Engineering Journal.

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note that often DOIs are not listed with PubMed abstracts and as such, may not be properly included when citing directly from PubMed. In these cases, please manually include DOIs in reference information.
Reply: The manuscript has been carefully revised to JOVE requirements.

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Reviewers' comments:

Reviewer #1:
Manuscript Summary:
Both in the manuscript title and abstract, please include the pellet mill type used in the study (i.e., flat die pellet mill) because the conventional pelleting mill is a different model than the one used in the study.
Reply: This has been included in the revised manuscript at all the appropriate places.

Major Concerns:
-- One of the critical steps in pelleting is grinding of feedstock (P4, L153-154). The authors have ground the raw corn stover in two stages; however, it appears that the authors have not used the raw corn stover at 30% or higher moisture content. Instead, the authors ground a relatively dry corn stover and added water to obtain 30% or higher moisture samples for the study (P5, L179-180). In addition to the pelleting process, the grinding process has the ability to dry the biomass which was not discussed in the manuscript. Another important difference would be in the particle size distribution of the ground particles which would be different depending on the initial moisture content of raw feedstock. If the authors had ground the corn stover at 30% moisture or higher, the results presented in this manuscript would have been much different and would have affected all of the end results including the pelleting energy consumption and pellet properties. Let alone, the grinder energy consumption for grinding a high moisture corn stover (MC of 30% or more) would be huge. Thus, the usefulness of the study is highly questionable because the study was conducted using a dry raw corn stover than a high moisture corn stover at the grinding stage.
Reply: The emphasis of this manuscript to show that corn stover can make good quality pellets at high moistures. To prove this point we have tested the material by adjusting the moisture content to varying desired levels. This method has been accepted by the peer review community and many papers have been published using this method. References on this method have been included in the revised manuscript (page 4, lines 152-156). Many researchers have used this method to study the effect of moisture content on pellet quality properties. Our TEA study on conventional and high moisture pelleting has indicated that drying energy has a more significant impact on the cost of preprocessing compared to grinding (Lamers et al., 2015). Our recent tests of grinding corn stover using a commercial scale grinder associated with the Biomass National User Faciltiy at Idaho National Laboratory indicated that grinding corn stover at 30% moisture content (65 kWhr/ton) takes about 3 times more energy compared to corn stover at 10% (20 kWhr/ton) moisture content, whereas drying corn stover from 30 to 10% takes about 300-350 kWhr/ton using a rotary dryer. So reducing the drying energy is critical to reduce the preprocessing cost and in turn the pelleting costs. Our TEA analysis indicated high moisture

pelleting reduces the pelleting cost by about 40 to 50% compared to conventional pelleting (Page 3, lines 125-131).

-- No statistical analyses were conducted. Especially, the significant effect of moisture or binder addition needs to be discerned using suitable statistical analysis.
Reply: Statistical analysis is included in the revised manuscript (page 8, lines 345; Page 9, lines 348-355), in the representative results section and new table 3 in the revised manuscript.

-- No replication was done for the pelleting process per se. Although the pelleting process is a continuous operation, pelleting at a given moisture and binder combination was not repeated at different times. Since this is a lab-scale study, this should be doable, but why the authors did not consider it. I noted that pellet properties were replicated, but not the energy consumption measurement.
Reply: As pelleting has resulted in steady state production condition, we have done our experiments a single time. The pellets which were produced at steady state conditions were further analyzed for physical properties. The pellet properties like pellet moisture content, bulk density, durability were measured in triplicates whereas pellet diameter measurement was an average of 10 measurements. We have published this procedure in other peer reviewed journals like Bioresource Technology, Biosystems Engineering and Energy Science and Engineering Journal.

-- No data on pellet temperature or pellet mill die temperature are reported. Since the authors have attributed the drying of corn stover due to frictional heating during pelleting, the temperature data need to be added to the manuscript.

Reply: As the die was rotating during pelleting it was difficult to attach a thermocouple. We measured the die temperature using a laser gun and reported this in our earlier work. This will be something we will definitely incorporate in our future studies.

-- No pellet dimensions (diameter and length) are provided. The L/D ratio of the die is given (P11, L445), but the die diameter or die length is not given. These critical data need to be added to the manuscript.

Reply: In the revised manuscript we have added information on die diameter and die channel length in the protocol section (page 8, lines 311-318). Also we have included data on pellet diameter and expansion ratio calculated using pellet diameter in the revised manuscript (page 9, lines 382-391; ; page 9, lines 392-413).

Minor Concerns:
P2, L80: Please check and correct - "biochemical chemical conversion".
Reply: Corrected in the revised manuscript.

P2, L83-84: Please remove "and its composition" which doesn't fit well in this sentence.
Reply: Removed in the revised manuscript.

P3, L104: Please corcted in Corect the word "table".
Reply: Corrected to tablet in the revised manuscript.

P3, L110: Please use the correct verb in "make helps to form".

Reply: Statement corrected in the revised manuscript.

P4, L147: Please use "evaluate specific energy consumption for pelleting" instead of "evaluate its specific energy consumption". Note the use of "its" is not clear here.
Reply: Changed in the revised manuscript.

P4, L166-167: Why was there a need for preheating of biomass in the hopper/feeder? You have not discussed the preheating temperature that you have used in your study.
Reply: Pellet mill is equipped to preheat the biomass to different temperatures, but in the present study we did not preheat the biomass before pelleting.

P4, L169-170: What were the die speed and feeding rate that you had used for the study? I did not find these data in your manuscript.
Reply: The feeding rates were adjusted based on the pelleting conditions to get a steady state pellet production.

P5, L185-187: It is not clear how the "uniform feeding of the biomass to the pellet mill" was achieved in this study. Is this manually done? Or, do you want to say "uniformly feed the biomass to the feed hopper".
Reply: Corrected in the revised manuscript.

P5, L194: Please use the correct article in "an laboratory oven".
Reply: Corrected the article in the revised manuscript.

P7, L265: Please add the word "respectively" at the end of this sentence.
Reply: Added in the revised manuscript.

P8, L347-348: Why do you think there is a need for "adjusting the feedstock moisture to the right levels" in a commercial pelleting process? In general, the lower the feed moisture, the better the pellet quality. Is it mandatory to have a moisture of 30% to make quality pellets? Or, are you suggesting to dry the high moisture corn stover if the moisture is higher than the values studied in this work (i.e., >30 to 40%)? Please elaborate this bullet point #1 to make it clear.
Reply: This has been corrected in the revised manuscript.

P9, L353: Please change "Our studies have indicated" to "Our study indicates".
Reply: Changed in the revised manuscript.

P9, L363: You have introduced a term "pelleting efficiency" here. Please define it.
Reply: I have changed this statement to match what we have measured in this study.

P9, L376: Please delete the repeating reference number "29" in "21,26,29-29".
Reply: Deleted in the revised manuscript.

P9, L371-383: The expansion of pellets is also affected by the "stress relaxation" behavior of the fibers in addition to moisture flash-off effect. The stress relaxation effect has to be mentioned here. You can easily find suitable references from the literature to cite for the stress relaxation effect.
Reply: Additional explanation included in the revised manuscript (page 14, lines 574-579).

P10, L421-429: Please provide the data on "L, D and L/D of the pellet mill die" in this paragraph.
Reply: Additional details of the die dimensions are included in the revised manuscript (page 6, Lines 240-241).

P10, L431: Please remove "50% of the".
Reply: Removed 50% in the revised manuscript.

Figure 1: Why is the preheating temperature given as "0 C". I hope you have not frozen the biomass. If no preheating is involved, please say "Preheat: None".
Reply: Changed to Preheat: None in the revised manuscript (figure 2 in the revised manuscript).

Additional Comments to Authors:
What were some of the properties of the corn starch used as a binder in this study (e.g., moisture, particle size distribution, chemical makeup of the starch types in the corn starch, flowability, etc.)?
Reply: The corn starch that we used in the present study is commercial available product. The details of the commercial product are included in the material section (excel sheet). The details of moisture content and bulk density of the grdound corn stover and corn starch binder are included in Table 1 in the revised manuscript. Also as we feel that other details like chemical makeup of the starch, flowability may not be necessary as we are adding commercial corn starch binder at a very low percentage of 2 and 4 %

Reviewer #2:
Manuscript Summary:
N/A

Major Concerns:
My largest concern/question is that the results are somewhat counterintuitive - that a high moisture pellet can be made with high density and high durability. Even the samples with no binder had a reasonably high bulk density. As stated in the paper itself, it is generally accepted that pellets do not form, or do not form well, at high moisture ("Moisture in the biomass while in the densification process acts as a binder and increases the bonding via van der Waal's forces, thereby increasing the contact area of the particle, but when the moisture content exceeds the threshold levels it acts more like a lubricant and does not help with binding. In general, low moisture content in the biomass (5- 10%) will result in denser, stable, and more durable briquettes compared to those produced from biomass with a higher moisture content.").
Given that, it remains unclear exactly why this approach works even though it is against the conventional wisdom. I believe it would be appropriate to mention specifically what aspects of the pelleting procedure used here are necessary for obtaining a solid pellet at high moisture, particularly if previous research has been performed to determine what variables are important. For example, a previous paper (Tumuluru 2014 - Biosystems Engineering) made the pellets using an 8 mm die. Is this larger die size required? Or is this simply the case where the "conventional wisdom" was never fully investigated and was incorrect, and therefore pellets can be easily made at the high moisture under many different process conditions?
Reply: I think the conventional wisdom on pelleting is based on meeting the specifications needed for transportation where higher density and durability are desirable for long distance transportation. But the literature has indicated that a lot of researchers have done pelleting research by varying moisture content in the range of 7-45% (w.b.) (page 4, lines 152-156), but increasing the feedstock moisture

content results in lower density pellets. Pellets that are produced by the pellet industry have higher bulk density (>700 kg/m3) as they transport the pellets over longer distances. These densities are achievable at lower feedstock moisture content. The process we have developed helps to make pellets at higher moisture content with densities in the range of 350-550 kg/m3 based on the feedstock conditions selected and helps to reduce the pellet production cost (Lamers et al., 2015). According to the Pellet Fuel Institute (PFI), USA, and the European Committee for Standardization (CEN), durability and bulk density are a normative specification for grading of pellets (Tumuluru et al., 2010). Pellets with durability values >96.5% and bulk density >640 kg/m3 are designated as super premium pellets based on PFI standards; whereas, in the case of CEN, the durability and bulk density values should be >97.5% and >700 kg/m3 and are desirable for international transport. Both PFI and CEN have other density and durability standards for lower grade pellets (Tumuluru et al., 2010). In general, to transport pellets over shorter distances (e.g., interstate), very high density and durability pellets may not be required. This process gives an opportunity to customize the pellet production process to produce pellets with varying durability and density values to meet different transportation scenarios. The present study has indicated that pellets with different density and durability can be produced at different feedstock moistures and binder addition, which can have a significant cost impact on storage and transportation of biomass for different logistics scenarios (Page 4, Lines 158-174).

Tumuluru, J. S., Sokhansanj, S., Lim, C. J., Bi, X. T., Lau, A.,. Melin, S., Sowlati, T., Oveisi, E. Quality of wood pellets produced in British Columbia for export. Appl. Eng. Agric. 26, 1013–1020.

Minor Concerns:

1) I did not see it mentioned what the die properties are in the article. What is the die size and thickness?

Die dimension are included in the revised manuscript (page 6, lines 240-241).

2) Figure 3: Is the bulk density shown here dry bulk density (ie, kg dry matter / m3) or wet bulk density? This is unclear in the paper.

Reported data is "as is bulk density" after cooling and after drying. We did not correct the bulk density to 0% moisture content.

3) I am assuming Fig 3 is dry bulk density, given that the difference between the bulk density before and after drying is less than the difference in weight from the drying. However, in general, the bulk density decreased slightly after drying. If so, then why does the dry bulk density decrease after drying; do the pellets expand?

The main reason for an increase in bulk density after drying could be due to lower inter-particle liquid bridges, which might have kept the particles closer and produced less open structure. Oginni (2014) in his studies observed the bulk density of ground Loblolly pine decreases with increases in moisture content. Explanation included in the revised manuscript.

4) Presumably, the reason that there is less drying occurring when binder is added is because there is less electricity consumption, which means less energy being turned to heat. Do you agree with this explanation?

Yes, it can be a good explanation for less moisture loss when binder is added.

5) Likewise, given the difference in electricity consumption, is this due to a decrease in the power consumption or due to an increase in the throughput of the pelletizer?
We have seen both lower energy consumption in the pelleting as well as increased throughput when binder is used.

Additional Comments to Authors:
N/A

Reviewer #3:
Manuscript Summary:
General Comments:
*The paper presents some interesting research. The procedures are carefully described for the most part and the work appears to be carefully carried out. One thing that is missing is an indication of the number of replications either for the experiments or for the property determinations (e. g. moisture content, bulk density, durability, etc.). Error bars are shown in figures 2 through 4 indicating some calculation of variability, but not in figures 5 and 6. This needs to be clarified.
Reply: The error bars were included for graphs representing pelleting moisture content, pellet diameter, expansion ratio, bulk density and durability. In case of percent fines and specific energy consumption, the error bars are not included as the data is from single pellet mill run from steady state pelleting condition. We have published this method in other high impact factor peer reviewed journals like Bioresource Technology, Biosystems Engineering and Energy Science and Engineering Journal.

*The biggest concern I have is the rational for the research which is interestingly spelled out in the last paragraph of the discussion near the end of the paper in lines 431-433. "Most of the woody and 50% of the herbaceous biomass is available at feedstock moisture contents of >30% (w.b.) and biorefineries are not ready to use this material due to high preprocessing costs related to drying the material for conventional pelleting." First, this statement should be included in the introduction since it is not based on any data in the paper. And second, and more importantly, I don't think that 50% of herbaceous biomass, and particularly corn stover, is available at moisture contents >30% w.b. Most corn stover will likely be available at 20% or less moisture content. Since it is harvested in a narrow window in the fall and needs to be available throughout the year it needs to be less than 20% for storage. The primary rational for pelleting at moisture contents greater than 30% seems to be the advantages in drying after pelleting versus before pelleting. While drying may be necessary for woody biomass, I think it is much less so for herbaceous biomass.
Reply: This statement has been moved to introduction section and additional information has been included in the revised manuscript regarding the moisture content of the harvested biomass based on the harvesting methods (page 3, lines, 121-123).

*On lines 130-132, the authors state that rotary dryers operating at 160 to 180oC are more energy intensive than grain dryers operating at 80oC. I don't think this is necessarily true. It may be true that there are less problems drying pelleted material in a grain-type dryer at 80oC than undensified, bulky biomass in a rotary dryer at 160 to 180oC, but I don't believe the rotary dryer is inherently less efficient (more energy intensive).
Reply: Belt or grain dryers operate at lower temperature, and they have greater efficiency compared to rotary drum dryers (additional information included in the revised manuscript). Additional explanation included in the revised manuscript (page 4, lines 140-145)

*While binders appear to have some advantages, at least at these moisture contents, what do they cost and how does this impact the cost of pelleted material?
Reply: We have added binder at very low percentages. Our TEA analysis team is working on understanding how the binder addition will influence the cost. We feel cost information is not within the scope of the manuscript.
Specific Comments:
Line 208 - include the dimensions of the plexi glass cylinder.
Reply: Plexi glass cylinder information included in the revised manuscript.

Major Concerns:
N/A

Minor Concerns:
N/A

Additional Comments to Authors:
N/A

Reviewer #4:
Manuscript Summary:
The crop residue is one of important renewable biomass. The pelletization of crop residue has more challenges than woody biomass due to the physical properties and compositions. This manuscript investigated the effects of moisture content and binder on the corn stover pellet production. The information reported in this manuscript will be helpful for the agricultural biomass based pellet production. There are several questions needed to be addressed before published.
1. The details of the material used in this studied was not clearly described? What was the moisture content in the biomass before adjust the moisture content? What is the composition of biomass?

Reply: We have included the moisture content of the biomass in the revised manuscript. We do not have the chemical composition data as we do not feel it is within the scope of this paper; however, we have added information regarding typical lignin content in corn stover (page 14, lines 603-605).

2. How did the authors adjust the moisture content to 33, 36 and 39%? Equipment and methods?
Reply: Included in the revised manuscript (page 6, lines 257-262; page 7, lines 263-267).

3. Page 7 line 270 and line 286, they should be "Figure 4".
Reply: Change to the correct number in the revised manuscript.

4. Why did the bulk density increased greatly after drying the pellet for FMC-33% without binder?
There was a slight increase in the bulk density values by about 50 kg/m^3 after drying.
The probable reason for the increase in bulk density after drying could be due to lower inter-particle liquid bridges, which might have kept the particles closer with less-open structure. Oginni (2014) observed that the bulk density of ground Loblolly pine decreases with increases in moisture content. This information is included on page 10, lines 424-427.

5. In addition to the cost for binder, the cost of drying for the pellet might also increase due to the increase of moisture according to the results of this manuscript. This manuscript only investigated the

specific energy consumption. The energy consumption and economic evaluation for whole process including pretreatment, pelletization, cooling and drying should be also investigated.
Reply: The emphasis of this manuscript is only on the method of producing good quality pellets using binder. Our analysis team is working on analyzing the cost of adding a binder into the pellet production process. This cost analysis of the whole high moisture pelleting process is presented in our TEA paper published in Bioresource Technology Journal.

Major Concerns:
N/A

Minor Concerns:
N/A

Additional Comments to Authors:
N/A