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Investigation of Steam Explosion Pretreatment of Sawdust and Oat Straw to Improve Their Quality as Biofuel Pellets

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Abstract: Steam explosion pretreatment of sawdust and oat straw under mild, medium, and severe conditions was conducted to improve the quality of pellets generated from these feedstocks. This work examined the effects of temperature, time, and moisture content on the mechanical properties of biomass pellets. From the ANOVA conducted, the *p*-values of the regression models for all the response variables (dimensional stability, tensile strength, and pellet density) studied were significant ($p < 0.05$), except for the pellet density of steam-pretreated oat straw pellets. The interaction of these three factors did not significantly affect the response variables of oat straw pellets. In addition, the higher heating value (HHV) of treated biomass increased up to a maximum of about 9.5% and 7% as compared with the non-treated sawdust and oat straw, respectively. In addition, an increment of about 3.6-fold and 3.1-fold in pellet tensile strength of steam-pretreated sawdust and oat straw was observed, respectively. Microstructural examination of the pellets from steam-pretreated biomass revealed that the material contained particles that were more closely bonded and featured a cemented surface with fewer pores when compared to particles from untreated oat straw and sawdust.

Keywords: steam explosion; pretreatment; pelletization; sawdust; oat straw; pellet quality

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1. Introduction

Achieving a sustainable, viable renewable energy supply will reduce our dependence on fossil fuels, but this remains a major challenge [1]. As the price of energy fluctuates, and concern over climate change has increased, bioenergy has emerged as an economically favorable alternative to fossil fuels that also provides environmental and energy security benefits [2]. Alternative energy generation can also be achieved using different kinds of renewable sources such as water, wind, and sun, but the industries based on sustainable materials, fuels, and chemicals depend mostly on lignocellulosic biomass [3]. Accelerating the use of biomass-derived energy can reduce emissions of greenhouse gases and supply a competitive market with fossil fuel, the cost of which is increasing daily. Biomass can be used as a solid fuel by direct combustion to produce electrical power through steam generation, or through gasification, which produces both combustible gas and syngas. Lignocellulosics are copiously available, moderately evenly distributed worldwide, and may ease the conflict in use between energy and food [4]. The use of agricultural and forest residues such as oat straw and sawdust for energy production relieves the pressure on food resources compared with the use of the more readily convertible starch-based biofuels,

while also adding value to low-value crop and forest residues [5]. Oat is a worldwide crop, and its production stands at about 25 million tonnes [6]. Canada is one of the leading suppliers of oat, and globally, oat trade makes up the majority. Oat is a major cereal crop in the Canadian province of Saskatchewan, with average yearly production in recent years of more than 1.5 million tonnes, on approximately 600,000 ha (1.5 million ac). Oat production has been steady in Saskatchewan. Recently, in the United States, food market oat demand has been active, as well as for the livestock market. Most of the Canadian oat trade is with the United States, with Canada being their major supplier [6]. In addition to the utility of oats as human food and agricultural animal feed, residues from oat production including straws are useful as biofuel feedstocks [7].

Using wood residues as a renewable energy source has come a long way in the Canadian forestry industry. Forest residues such as bark, wood chips, branches, treetops, and sawdust have become feedstocks for electricity cogeneration facilities and advanced steam facilities. New bioenergy products have emerged such as syngas, cellulosic ethanol, wood pellets, biodiesel, biocarbon, and bio-oil for use in lumber drying kilns [8]. Important tree species in Saskatchewan include white spruce, trembling aspen, black spruce, and jack pine. There are four large sawmills in Saskatchewan along with several smaller sawmills; the total annual production capacity of these mills is more than 1.3 million m^3 (545 million board feet) of spruce–pine–fir (SPF) lumber [9]. White spruce (*Picea glauca*) is a widely distributed boreal forest conifer in Canada. In Saskatchewan, this species is distributed across the expanse of the boreal forest region, mainly in moist, well-drained silty soils. Accordingly, white spruce makes up a major proportion of the total allowable annual harvest in Saskatchewan (16% of the total and 28% of the coniferous allowable cut) [9].

The massive production of the crop and forest products results in a great deal of biomass residue, and the majority of these residues are simply left on the field after grain harvest or in the forest, respectively. A very small proportion of this biomass is used, principally as agricultural animal feed and bedding, or as mulch. Straw biomass can be more completely utilized as a feedstock for the production of biofuels and bioproducts [10]. The use of such abundant biomass sources that produce low net carbon dioxide emissions can be part of a solution to decreasing greenhouse gas levels associated with the production and use of fossil fuel resources [10].

Some of the difficulties associated with the exploitation of untreated biomass that can impede their large-scale application include low bulk density, high moisture content, hydrophilicity, and inherently low calorific value. These constraints impact the logistics, storage, and energy efficiency of these feedstocks [11–13]. Several pretreatment strategies including physicochemical and biological pretreatment methods have been investigated to overcome this obstacle caused by lignocellulose matrix to cellulose degradation. As a result of the high economic cost and severity of most of these pretreatment application methods, their utilization on an industrial scale is very limited [14–17].

Steam explosion is a physiochemical pretreatment process that breaks down lignocellulosic biomass by the application of high-pressure heat, which results in the formation of organic acid, and shearing forces causing moisture expansion and explosive decompression are the two stages involved in the steam-explosion process. These processes modify the biomass components through hydrolysis of hemicellulosic components (resulting in the release of mono- and oligosaccharides), alteration to the chemical structure of lignin, and enhancement of the crystallinity index of cellulose. These conditions allow the lignocellulosic biomass structures to unlock and can enhance the fermentable carbohydrate yield of subsequent enzymatic hydrolysis steps [18].

Biomass pretreatment modifies the structure of the biomass feedstock, which facilitates pelletization [19,20]. To improve the quality of biomass pellets, chemical binders combined with pretreatment are required, but these negatively impact the total cost of densification [21,22]. Several biomass pretreatments have been investigated to facilitate the pelletization of agricultural biomass. Douglas fir was subjected to high-pressure saturated steam treatment by Lam et al. [23] to enhance its pellet quality. This revealed that the

moisture absorption rate of pellets reduced from 0.0152 to 0.0125 mL/min, indicating an improvement in storability.

Retention time during steam explosion has a major effect on the amount of degradation of the products that are observed, which must be minimized. Moreover, pressure is another important parameter that is correlated to temperature and influences the kinetics of the production of degradation products and hydrolysis of cellulose fractions. Additionally, the difference between the atmospheric pressure and that of the reactor is proportional to the severity of the shearing forces applied to the biomass when the pressure is suddenly and explosively released [24]. Much literature can be found related to the effects of pressure and retention time on steam explosion of biomass, but few previous studies have examined the effect of initial moisture content of the feedstock. The objective of this study is to fill this knowledge gap by determining the effects of initial moisture content of the feedstock during steam explosion pretreatment on the fuel and physiochemical properties of biomass.

2. Materials and Methods

2.1. Feedstock Preparation

Oat straw and white spruce sawdust were used in this study. The oat straw was obtained from a black soil zone within the Rural Municipality of Douglas No. 436 around Maymont, SK, Canada. The variety of the oat straw is Morgan, harvested in mid-October 2019, swathed, and baled with a conventional combine. The white spruce sawdust was collected from NorSask Forest Products Inc. (Meadow Lake, SK, Canada) in June 2020. The samples were kept in a dry chamber to be used for this study. Oat straw and white spruce sawdust samples had a moisture content of approximately 10% (w.b.) and 42% (w.b.) as received, respectively. Oat straw samples were further size reduced using a hammermill of sieve 3.2 mm. Samples were preconditioned to different moisture contents (25%, 35%, and 45% w.b.), by carefully spraying a calculated amount of water required for the targeted sample moisture content and gently stirring to obtain a homogeneous mixture. The preconditioned samples were stored in a sealed plastic bag in a conditioned room at 4 °C for a period of 48 h before determination of sample moisture content and usage for experimentation.

2.2. Steam Explosion Experiment

The experiment was conducted at the Canadian Feed Research Center (North Battleford, SK, Canada). Steam explosion pretreatments were carried out in a closed batch unit (Figure 1). The steam explosion system is composed of a 30 kW steam generator (HSI Model # STH-1640-30-4E) with a capacity to produce steam up to 3448 kPa (500 psi) (HSI Hydro Steam Industries, Franklin Park, IL, USA) (Figure S1a). It also comprises a 20.3 cm diameter pressure vessel (chamber) with a volume capacity of 40 L and pressure up to 3323 kPa (482 psi) (Figure S1b). It also consists of a cyclone that opens at the bottom to allow the discharge of treated samples (Figure S1c). Pressure is controlled through the manual opening and closing of the main steam feeding valve to the vessel and the time is controlled using a timer. Pressure was monitored using a software pressure gauge connected to a laptop. Pressure was regulated by opening and closing of the steam inlet valve using an electric/pneumatic valve.

The samples were steam exploded at three temperatures (160 °C, 180 °C, and 200 °C) and three samples of moisture contents (25%, 35%, and 45% w.b.) for three retention times (5, 7, and 9 min). These conditions were chosen based on the severity of operating conditions, that is, mild, medium, and severe steam explosions. Approximately 1000 g of biomass was loaded through the ball valve located at the hopper of the pressure vessel. The steam was generated inside the boiler, and when the steam reached the required reaction temperature, then the ball valve was manually opened to let the saturated steam be transferred to the reactor chamber (vessel) to treat the sample for a certain period.

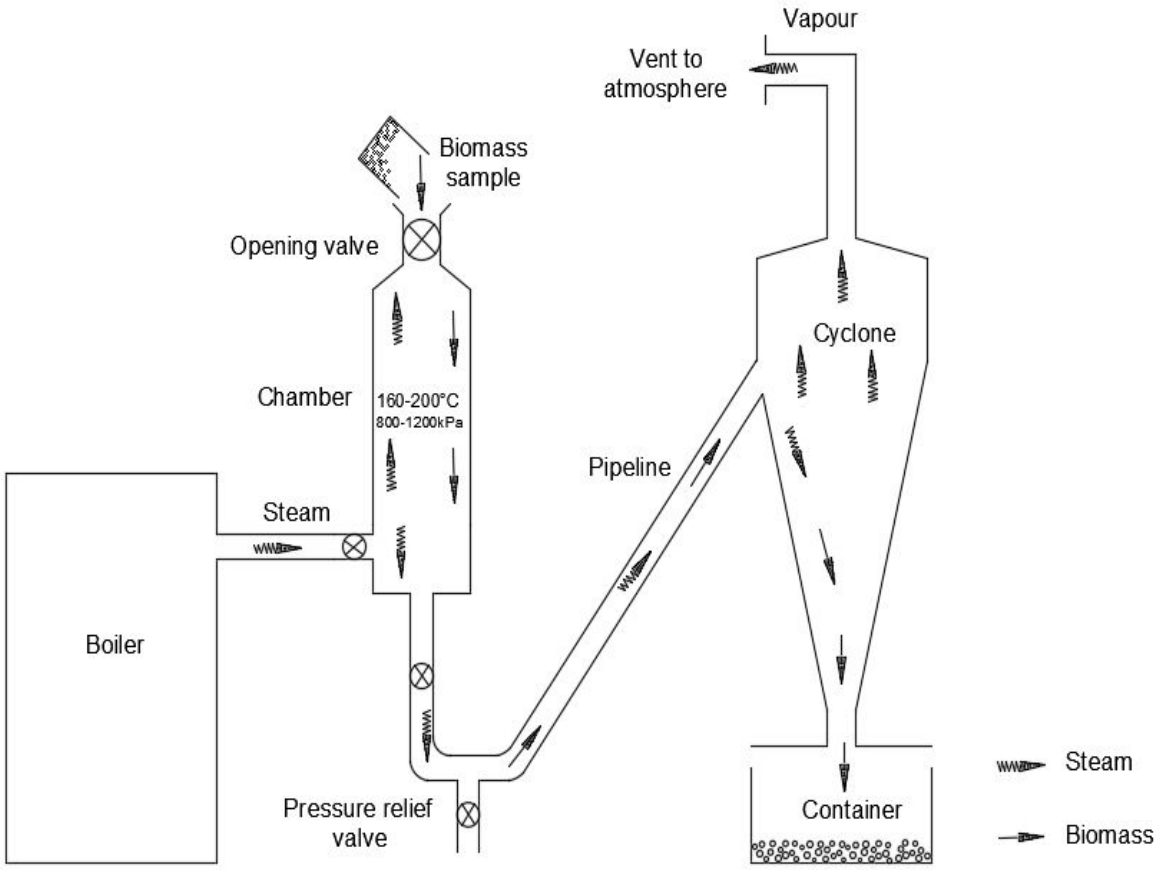

Figure 1. Schematic diagram of the steam explosion system used in the experiments.

2.3. Experimental Design of Steam Explosion Pretreatment

A factorial design of three factors, temperature (°C), retention time (min), and sample moisture content (%), at three levels was investigated. All experiments were conducted in three replicates. Analysis of variance (ANOVA) was conducted for all the response variables (dimensional stability, pellet tensile strength, and pellet density).

2.4. Pelletization of the Samples

Pelletization of steam-exploded samples and untreated samples were carried out using a single pellet unit firmly fixed to an Instron tester (Model No. 3366, Instron Corp., Norwood, MA, USA) preset at 4000 N maximum downward force to produce the pellets. The assembly consisted of three parts: (1) a cylinder, (2) a heating tape looped all over the external of the cylinder, and (3) a piston. The body of the cylinder was heated up to about 95 °C by turning on the electric power; during that time, a compressive pressure of approximately 126 MPa at a rate of 50 mm/min was provided to the plunger for a holding time of 1 min, then the pellets were ejected and cooled. Approximately 0.70–0.75 g of the samples, at a moisture content of about 8.5% (w.b.), were introduced into the cylindrical die which was then compressed by the plunger to make pellets. Approximately ten pellets were produced for each pretreatment condition, then stored separately in an airtight plastic container for other experiments.

2.5. Pellet Unit Density and Dimensional Stability

After producing the pellet, its unit density was determined. The unit density is described as the ratio between the mass of a pellet and its volume. A digital vernier caliper was used to measure the diameter and length of individual pellet in this study, with an accuracy of $\pm$0.01 mm; on the other hand, a digital weighing balance was used to measure single pellet weight with an accuracy of $\pm$0.001 g. The dimensional stability, which is

described as the volumetric difference in percent after producing pellets (s_0) and after 14 d of storage (V_{14}), was computed as given in Equation (1).

$$\text{Dimensional stability } (\%) = \frac{V_0 - V_{14}}{V_0} \times 100 \quad (1)$$

2.6. Diametral Compression Test

The tensile strength of the sawdust and oat straw pellets were evaluated by diametral compression test conducted using the Instron tester. Resistance to failure and dust generation as a result of handling (transportation and storage) is a good sign of the tensile strength of pellets. Kashaninejad and Tabil [25] performed a similar test using the same equipment to determine the tensile strength of pellets. In this study, a diamond-cutting wheel attached to a Dremel rotary tool (Robert Bosch GmbH, Stuttgart, Germany) and a scalpel were used to cut the pellets diametrically into 2.5 mm specimens. The specimen was placed on its edge on the lower plate of the test rig in the Instron tester, which was padded, and the upper plunger provided a compressed 1000 N load cell at a crosshead speed of 1 mm/min until failure happened. The tensile strength of pellets was calculated using Equation (2).

$$\delta_x = \frac{2F}{\pi dl} \quad (2)$$

where δ_x is tensile strength (Pa); F is fracture load (N); d is specimen diameter (m); and l is specimen thickness (m).

2.7. Moisture Absorption

The moisture absorption test of pellets from steam explosion treated and untreated oat straw grinds and white spruce sawdust were carried out after densification to investigate their water uptake. In this study, moisture absorption analysis was conducted in a controlled environment chamber (Espec SH-641 Benchtop chamber, ESPEC Corp., Osaka, Japan) by estimating the overall moisture uptake of various samples under 90% relative humidity at a temperature of 25 °C. A worst-case scenario was assumed while selecting the conditions for the analysis. This implies the conditions from seasons in Canada for storage. Pellet samples were left in the controlled environment for approximately 72 h until the moisture content was constant to evaluate the equilibrium moisture content (EMC). Other researchers used similar conditions for the calculation of EMC of biomass or their pellet form [23,26,27].

2.8. Characterization of Raw Material and Pellets

The untreated and steam-treated oat straw and sawdust pellets and grinds were characterized to rate the effects of each treatment.

2.8.1. Moisture Content and Solid Yield

The samples moisture content as received was determined using ASAES358 [28]; about 25 g of samples were oven-dried at a temperature of 103 °C for 24 h. In addition, the grind samples' moisture contents were determined using AACC Standard 44-15A [29], where 3 g of sample was oven-dried at 130 °C for about 90 min. The moisture content of samples after drying were also determined using the ASAES358 method. These moisture content tests were conducted in three replicates. Solid yield recovery of white spruce sawdust and oat straw were determined on dry solid basis as the percentage of samples recovered after the steam explosion experiment.

2.8.2. Particle Size

Determination of the particle size was performed using the ASAE S319 sieving method [5,30–32]. A Ro-Tap mechanical sieve shaker (W.S. Taylor Inc., Mentor, OH, USA) was used for this experiment. The selection of sieve series was based on the variety of

particle sizes in the samples. In this work, U.S. sieve numbers 16, 20, 30, 50, 70, and 100 (sieve mesh opening: 1.190, 0.841, 0.595, 0.297, 0.210, and 0.149 mm, respectively) were used. Approximately 100 g of steam-treated and untreated biomass samples were examined in triplicate for particle size distribution. As indicated in the ASAE Standard S319, the sieve shaker was allowed to operate for about 10 min. Equations (3) and (4) were used to calculate the geometric mean diameter (d_{gw}) and standard deviation of particle diameter (S_{gw}), respectively.

$$d_{gw} = log^{-1}[\frac{\sum_{i=1}^{n}(W_i log d_i)}{\sum_{i=1}^{n} W_i}] \quad (3)$$

$$S_{gw} = \frac{1}{2} d_{gw} \left[log^{-1} S_{log} - \left(log^{-1} S_{log}\right)^{-1}\right] \quad (4)$$

where d_{gw} is the geometric mean diameter of particles by mass in mm; n is the number of sieves + 1 pan; d_i is the nominal sieve aperture size of the ith sieve in mm; W_i is the mass on the ith sieve in grams; S_{log} is the geometric standard deviation of log-normal distribution by mass in the common (base 10) logarithm.

2.8.3. Bulk and Particle Density

Bulk density of treated and untreated oat straw grinds and white spruce sawdust were measured using a standard 0.5 L cylindrical cup (SWA951, Superior Scale Co. Ltd., Winnipeg, MB, Canada). The samples were introduced to the center of the cylindrical container through a funnel. Material flowing through the funnel was aided by stirring the grind with a steel roller. Excess sample at the top of the cylindrical cup was removed using a steel roller in a zig-zag pattern after completely filling it. The bulk density of treated and untreated oat straw grinds and white spruce sawdust in kg/m^3 were determined by calculating the mass per unit volume.

Particle density of treated and untreated oat straw grinds and white spruce sawdust were measured using a gas displacement pycnometer (AccuPyc 1340, Micromeritics Instruments Corp., Norcross, GA, USA) at a temperature of 22 ± 0.9 °C. Data were acquired in replicates of 10 for an individual sample.

2.8.4. Elemental Analysis

The amounts of carbon, hydrogen, nitrogen, and sulfur (CHNS) present in the samples were determined using the PerkinElmer Elementar CHNS analyzer (Vario EL III, Elementar Americas Inc., Ronkonkoma, NY, USA). The oxygen content in the sample was calculated by difference. Firstly, the equipment was calibrated using sulfanilic acid as a standard. Then, 4−6 mg of the samples was collected in an aluminum foil container and combusted for analysis.

2.8.5. Higher Heating Value and Ash Content

A bomb calorimeter (6400 Automatic Isoperibol, Parr Instrument Company, Moline, IL, USA) was used to determine the higher heating value (HHV) of the steam-exploded treated and untreated pellets. Approximately 0.5 g of the samples were burnt in an oxygen-filled metal cylinder submerged in a known volume of water, all held within a thermally insulated chamber. The test was conducted in three replicates for all samples.

2.8.6. Microstructural Examination

Scanning electron microscope (SEM Phenom-World, Eindhoven, The Netherlands), together with a stereoscope (Wild M_3Z, Wild Heerbrugg, Gais, Switzerland) having a magnification of 200×, paxcam3 camera (Midwest Information Systems, Villa Park, IL, USA), and Intralux 500 light source, was used to examine the surface morphology of the ground biomass and pellet. A scalpel was used to cut the pellet sample longitudinally and coated with gold to give a gold layer of 10 nm thickness employing a vacuum sputter coater (Q150T ES, Quorum Technologies, Sussex, UK).

3. Results and Discussion

3.1. Moisture Content and Solid Yield

After steam explosion pretreatment, the moisture content of oat straw samples was between 69.4% and 80.4% (w.b.), while white spruce sawdust samples ranged from 68.5% to 84.4% (w.b.). Samples that were treated at 200 °C had a mean moisture content that was significantly (p = 0.05) higher than samples treated at 160 °C and 180 °C. In addition, the average moisture content of samples treated for 9 min was significantly (p = 0.05) higher than samples treated for 7 min and 5 min. This is a result of the longer duration the samples remained in the vessel, thereby allowing for more absorption of moisture. Adapa [33] reported an average moisture content of 76.7% (w.b.) for oat straw when the digester was operated at 180 °C for 4 min. The initial moisture content of the sample is a determining factor in the severity of steam explosion pretreatment, since it impacts the ability of heat and chemicals to permeate the biomass [34]. The moisture content of steam-exploded samples after drying was between 7.8% and 9.2% (d.b.) for oat straw, while dried sawdust samples ranged from 8.1% to 10.4% (d.b.). Table S1 shows the severity factor as determined using the equation given by Chornet and Overend [35]. In the current work, solid yield of white spruce sawdust samples varied between 61% and 85%, while that of oat straw samples varied between 72% and 90%. Lam [36] reported a solid yield range between 51% and 84% when Douglas fir was steam exploded at 200 °C and 220 °C, respectively. The mean solid yield was significantly (p = 0.05) lower for samples treated at 200 °C, compared to samples treated at 180 °C and 160 °C. In the current study, it was observed that the loss of fine particles during drying amounted to less than 1 g, which corresponds to under 5% of the sample weight. In a similar study, Kobayashi et al. [37] reported 50% lower losses of solid yield for woody biomass from compressed hot water treatment at 240 °C. Normally, losses are due to inefficiencies in recovery of materials combined with the loss of volatiles during steam explosion [38,39].

3.2. Particle Size Analysis

The average particle size of steam-exploded oat straw and white spruce sawdust was significantly smaller than that of the non-treated biomass. This may be because steam explosion pretreatment application disintegrates the lignocellulosic structure of the biomass [30], resulting in decreased shear strength and easy grindability. Geometric mean particle sizes of 0.586 mm and 0.656 mm were recorded for untreated white spruce sawdust and oat straw, respectively. The particle size of steam-exploded biomass particles decreased with increasing severity of the steam explosion pretreatment. The average particle size of oat straw samples after steam explosion was between 0.565 mm and 0. 456 mm, while the corresponding values of white spruce sawdust samples was between 0.512 mm and 0.389 mm. Adapa et al. [5] observed similar results using steam explosion of oat straw. In their work, the average particle size of oat straw was reduced from 0.566 mm to about 0.367 mm when it was treated at 180 °C for 4 min. In addition, Lam [36] found similar results using steam explosion of Douglas fir (*Pseudotsuga taxifolia*) wood chips treated at 200 °C and 220 °C for 5 min, resulting in a decrease in the mean particle size from 0.42 mm to 0.40 mm (200 °C) and 0.35 mm (220 °C). Increasing reaction severity led to the increase in percentage of smaller particles and fines in each sample. A Shapiro–Wilk test ($p > 0.05$) indicates that the particle size of the grinds from the various reaction severities were approximately normally distributed. The particle sizes of the ground material should be normally distributed (Shapiro–Wilk test: $p > 0.05$) and should also have close to zero skewness with a lower peak than would be expected for the normal and wider distribution of data. This is reflected in negative Kurtosis values. The particle size distribution of the ground biomass from all screen sizes were skewed along the y-axis, and had a lower peak compared to a normal distribution. Figure 2 shows the normal size distribution of the untreated and treated sample particles at different severities. Wider particle size distribution is known to be suitable for densification [40]. During densification, the finer

particles rearrange and occupy the void space between the larger (coarse) particles, which results in the production of denser and more durable pellets [41].

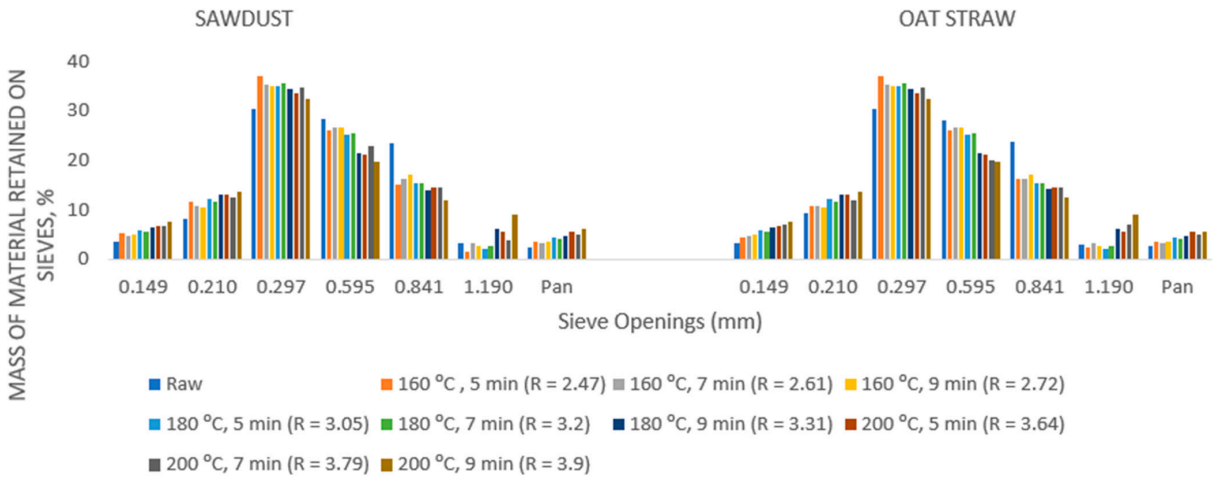

Figure 2. Size distribution of the untreated and treated sample particles at different severities.

3.3. Properties of Pellets from Non-Treated and Steam-Exploded Samples

The pellet tensile strength, dimensional stability, and unit density of the sawdust and oat straw from the untreated sample fuel pellets were 0.68 MPa and 0.34 MPa, 1011.02 kg/m^3 and 1009.67 kg/m^3, and 1047.95 kg/m^3 and 1031.74 kg/m^3, respectively, as shown in Table 1. When biomass is heated, lignin softens and melts, which provides thermosetting binder resin properties on the heated material and produces pellets with good dimensional stability and higher density [41]. Pretreated samples had higher initial pellet densities compared to untreated ones. Pellet shrinkage was observed for the pretreated pellet samples after 14 d, while the untreated pellet samples overall expanded in the longitudinal directions and diametrically.

The results (Table 1) also show that as the treatment severity increased, the pellets treated by steam explosion showed a volumetric expansion after relaxation. Dimensional stability provides data on how stable the formed pellets can be during the transportation process and under storage conditions. Smaller values are associated with increased pellet stability. A positive value indicates that there was either diametral or longitudinal expansion, while a negative value shows that there was either diametral or longitudinal contraction after 14 d. The results from the current study are in accordance with previous research showing that steam treatment prior to densification improves the compressibility of wood particles and greatly decreases the internal stresses that accumulate during compression. In addition, the tensile strength increment with increasing severity could be attributed to the disruption of the lignocellulosic structure, which is associated with increased degradation of the hemicellulose and lignin, thereby acting as a natural binder through adhesion, forming solid bridges between biomass particles and thus increasing pellet tensile strength. The bulk density of untreated oat straw and sawdust was significantly higher than the sample treated by steam explosion (Table 1). This could be because steam explosion pretreatment application breaks up the arranged and compact lignocellulosic structure of biomass, leading to lower bulk densities. Similar results have been obtained by other researchers [33,42]. The bulk density of treated oat straw ranged from 51.8 kg/m^3 to 82.1 kg/m^3. The bulk density of treated sawdust ranged from 97.4 kg/m^3 to 129.9 kg/m^3. The particle density of sawdust and oat straw treated by the steam explosion was significantly higher than the untreated sample. This is because steam explosion disintegrated the biomass into finer components, thereby disorganizing its lignocellulosic structure. The particle densities of steam-exploded oat straw and sawdust ranged from 947 kg/m^3 to 1444 kg/m^3 and 1172 kg/m^3 to 1414 kg/m^3, respectively.

Table 1. Properties of pellets from non-treated and steam-exploded samples.

			Sawdust					Oat Straw				
Temp (°C)	**RT (min)**	**M.C. (%)**	**Pellet Density (kg/m^3)**	**D.S. (%)**	**Tensile Strength (MPa)**	**Bulk Density (kg/m^3)**	**Particle Density (kg/m^3)**	**Pellet Density(kg/m^3)**	**D.S. (%)**	**Tensile Strength (MPa)**	**Bulk Density (kg/m^3)**	**Particle Density (kg/m^3)**
NT	-	-	1047.95	8.93	0.68	158.00	1051.56	1031.74	11.07	0.35	127.87	882.23
160	5	25	1060.71	8.07	1.05	129.90	1172.45	1154.99	10.76	0.99	82.12	947.42
160	5	35	1090.33	7.77	0.92	123.60	1209.32	1054.34	10.02	0.92	79.92	996.35
160	5	45	1077.59	7.59	1.28	125.83	1278.78	1154.81	9.38	0.97	68.24	1005.34
160	7	25	1135.76	6.96	1.39	112.90	1280.97	1157.88	8.65	1.02	66.23	1030.24
160	7	35	1072.36	6.78	1.40	116.12	1281.76	1167.44	8.43	1.25	63.42	1001.22
160	7	45	1072.33	6.21	1.47	117.23	1300.12	1172.28	8.39	1.30	67.87	1092.75
160	9	25	1094.65	5.43	1.88	110.53	1352.22	1148.31	7.58	1.55	63.32	1069.45
160	9	35	1115.64	5.78	2.16	110.64	1367.17	1151.46	7.39	1.43	62.71	1110.66
160	9	45	1103.91	5.89	2.33	110.52	1370.23	1153.56	7.32	1.46	60.28	1153.47
180	5	25	1121.58	4.46	2.59	127.41	1356.34	1168.82	6.59	1.68	59.19	1166.33
180	5	35	1131.34	4.24	2.45	112.43	1373.56	1172.89	6.42	1.56	57.97	1174.90
180	5	45	1111.57	4.57	2.48	116.72	1377.78	1172.41	6.63	1.62	58.52	1180.88
180	7	25	1122.61	4.50	2.27	110.58	1381.96	1190.31	6.75	1.65	57.93	1224.32
180	7	35	1133.31	4.43	2.02	114.36	1389.43	1225.07	6.62	1.64	56.84	1217.12
180	7	45	1144.26	4.03	2.13	113.19	1391.19	1191.72	6.43	1.73	57.91	1263.89
180	9	25	1130.01	3.93	2.82	102.37	1400.66	1202.59	6.25	2.03	56.21	1283.35
180	9	35	1167.04	3.70	3.36	106.42	1402.34	1210.21	5.23	1.98	55.82	1257.67
180	9	45	1153.85	3.48	3.21	108.21	1407.33	1195.25	5.24	2.12	56.25	1263.55
200	5	25	1184.66	2.55	3.55	108.68	1406.20	1219.43	4.55	2.11	54.16	1359.98
200	5	35	1111.58	2.58	3.42	105.62	1402.59	1208.22	4.44	2.09	53.18	1367.23
200	5	45	1131.30	2.32	3.09	107.83	1405.34	1214.58	3.62	2.33	54.17	1328.86
200	7	25	1145.54	2.66	3.32	106.11	1408.32	1197.96	3.51	2.63	54.46	1383.43
200	7	35	1134.72	2.49	3.22	101.45	1406.87	1259.83	3.05	2.54	55.56	1371.55
200	7	45	1132.48	1.36	3.57	101.66	1407.90	1237.87	2.43	2.64	55.29	1408.34
200	9	25	1147.43	1.65	3.89	101.62	1411.47	1246.93	2.95	3.13	54.83	1411.23
200	9	35	1225.97	1.08	4.04	100.85	1415.62	1190.15	2.04	2.98	51.82	1420.45
200	9	45	1148.58	1.62	3.95	97.42	1414.05	1253.21	2.45	3.34	53.12	1444.09

Temp = temperature; RT = retention time; M.C. = moisture content; N.T. = untreated; D.S. = dimensional stability.

3.4. Elemental Composition Analysis

Table 2 shows that pretreatment severity affects the elemental composition of biomass samples on a dry matter basis. The carbon, hydrogen, sulfur, and nitrogen content of the untreated oat straw and sawdust was 44.24%, 6.05%, 0.11%, and 0.41% and 47.49%, 6.66%, 0.02%, and 0.01%, respectively. After the steam pretreatment, the carbon and the nitrogen contents of the samples significantly increased ($p < 0.05$) as the severity increased, while hydrogen content decreased. Some researchers reported similar results from their works on steam-pretreated biomass [43–45]. Angles et al. [46] also reported that the process of steam explosion causes the lignin to carbonize and even condense, which results in an increase in the carbon content of the pretreated samples. Generally, during steam explosion, smaller hydrocarbon molecules with low energy density volatilize, which increases the energy density of the residual carbon-rich solids. In this study, it was assumed that the reduction in hydrogen and oxygen is due to the formation of carbon dioxide and water. Hydroxyl groups are connected to the backbones of the biomass structural components. As the steam penetrates the biomass samples, debranching reactions continue and release acetic and uronic acids which hydrolyze hemicelluloses. Furthermore, as the severity of the treatment increases, amorphous cellulose could be partially depolymerized [47–49]. The ash content of the oat straw before steam explosion pretreatment is 5.32%, which is much higher than the sawdust (less than 1%). Generally, cereal straw biomass samples have higher ash contents than woody biomass [50–52]. As shown in Table 2, the ash content for sawdust and oat straw increased from 0.14% to 0.61% and 5.32% to 6.49%, respectively, as the severity factor increased. The increase in ash content after steam explosion pretreatment is due to the loss of other components of biomass as also reported by some researchers [51,53]. Although Han et al. reported a reduction in ash content from 1.6–4%, this may be due to the removal of ash during the release of steam [54].

Table 2. Elemental composition and final moisture content of steam-exploded and non-treated biomass grinds (dry matter basis).

Biomass	R	M.C. (%)	C (%)	H (%)	N (%)	S (%)	O (%)	Ash (%)	H/C	O/C
Sawdust	NT	8.69 ± 0.16	47.47 ± 0.46	6.66 ± 0.12	0.11 ± 0.02	0.21 ± 0.01	45.41	0.14	0.14	0.96
	2.47	8.62 ± 0.12	47.96 ± 0.23	6.49 ± 0.07	0.15 ± 0.01	0.20 ± 0.01	44.91	0.29	0.13	0.94
	2.61	8.60 ± 0.13	47.98 ± 0.18	6.51 ± 0.11	0.21 ± 0.02	0.23 ± 0.01	44.78	0.29	0.13	0.93
	2.72	8.55 ± 0.26	48.24 ± 0.21	6.48 ± 0.06	0.26 ± 0.10	0.18 ± 0.02	44.54	0.30	0.13	0.92
	3.05	8.51 ± 0.08	48.45 ± 0.11	6.45 ± 0.03	0.30 ± 0.01	0.27 ± 0.03	44.21	0.32	0.13	0.91
	3.20	8.49 ± 0.14	48.53 ± 0.13	6.43 ± 0.10	0.33 ± 0.01	0.22 ± 0.03	44.11	0.38	0.13	0.91
	3.31	8.41 ± 0.20	48.61 ± 0.08	6.39 ± 0.06	0.32 ± 0.01	0.20 ± 0.01	44.06	0.42	0.13	0.90
	3.64	8.33 ± 0.26	49.54 ± 0.15	6.40 ± 0.12	0.35 ± 0.03	0.23 ± 0.02	42.94	0.54	0.12	0.87
	3.79	8.27 ± 0.17	49.67 ± 0.10	6.38 ± 0.05	0.38 ± 0.01	0.22 ± 0.01	42.77	0.58	0.12	0.86
	3.90	8.24 ± 0.22	50.10 ± 0.09	6.35 ± 0.08	0.42 ± 0.04	0.24 ± 0.04	42.28	0.61	0.12	0.84
Oat straw	NT	8.41 ± 0.18	44.24 ± 0.22	6.05 ± 0.11	0.41 ± 0.02	0.11 ± 0.02	43.87	5.32	0.14	0.99
	2.47	8.39 ± 0.20	44.36 ± 0.26	6.04 ± 0.08	0.39 ± 0.01	0.26 ± 0.04	43.59	5.36	0.13	0.98
	2.61	8.20 ± 0.31	44.39 ± 0.09	6.01 ± 0.03	0.38 ± 0.03	0.18 ± 0.02	43.37	5.67	0.13	0.97
	2.72	8.19 ± 0.22	44.48 ± 0.17	5.99 ± 0.01	0.37 ± 0.02	0.14 ± 0.01	43.16	5.86	0.13	0.97
	3.05	8.14 ± 0.23	44.87 ± 0.28	5.98 ± 0.12	0.39 ± 0.02	0.15 ± 0.02	42.64	5.97	0.13	0.95
	3.20	8.12 ± 0.05	44.89 ± 0.12	5.92 ± 0.09	0.35 ± 0.05	0.11 ± 0.05	42.65	6.08	0.13	0.95
	3.31	8.01 ± 0.06	45.05 ± 0.03	5.89 ± 0.02	0.33 ± 0.01	0.10 ± 0.02	42.41	6.22	0.13	0.94
	3.64	7.99 ± 0.17	45.57 ± 0.11	5.87 ± 0.10	0.36 ± 0.02	0.11 ± 0.01	41.80	6.29	0.12	0.92
	3.79	7.98 ± 0.32	45.66 ± 0.07	5.88 ± 0.04	0.37 ± 0.01	0.11 ± 0.01	41.61	6.37	0.12	0.91
	3.90	7.98 ± 0.06	45.65 ± 0.08	5.84 ± 0.01	0.41 ± 0.01	0.11 ± 0.01	41.50	6.49	0.12	0.90

R = severity factor; M.C. = moisture content (d.b.); C = carbon; H = hydrogen; N = nitrogen; S = sulfur; O = oxygen; H/C = hydrogen to carbon ratio; O/C = oxygen to carbon ratio; and NT = non-treated.

3.5. Higher Heating Values (HHV) of Pretreated and Untreated Biomass

The determination of HHV was essential due to the expected use of biomass treated by steam explosion for home heating or for the production of electricity. The HHV of the untreated sawdust and oat straw was 18.02 MJ/kg and 16.84 MJ/kg, respectively. Severity of the treatment condition influences the HHV, as shown in Figure 3. The HHV of treated biomass increased up to a maximum of about 9.5% and 7% compared to the untreated sawdust and oat straw, respectively. Higher moisture content negatively affected the HHV of the solid pellet, while increased temperature positively affected the HHV. Moisture presence in fuel causes smoke and promotes partial combustion. Bhattacharya et al. [55] studied the effect of moisture content on efficiencies of biomass-fired cookstoves and reported a reduction in efficiency in all stoves with an increase in fuel moisture content from 10% to 25%. In a similar manner, Yibo et al. [56] reported that the burning rate of biomass in a semi-gasified cookstove decreased by 33.3% from 30.0 g/min at 5.9% moisture content to 20.0 g/min at 22.1% moisture content, and the cooking power was reduced by 36.1% from 1910 W at 5.9% moisture content to 1220 W at 22.1% moisture content. Lam et al. [23] and Kumar et al. [57] observed an increase in HHV, ash, and lignin content when whitewood biomass was steam-pretreated. Adapa [33] reported that the HHV of oat straw increased from 16.40 MJ/kg to 17.80 MJ/kg after steam explosion pretreatment at 180 °C, using a steam pressure of 900 kPa for 4 min. Any chemical, structural, or physical changes in the composition of lignocellulosic biomass (cellulose, hemicellulose, and lignin) could potentially lead to changes in the HHV of the biomass. As stated earlier, during steam explosion, small hydrocarbon molecules with low energy density volatilize, thereby leaving behind the remaining components that increase energy density. In addition, carbonization of the biomass sample during steam explosion pretreatment resulted in higher energy content. Steam explosion affects the HHV of the formed pellets by affecting the raw biomass structural compositions, and this is achieved by autohydrolysis, which removes the extractives with low volatility resulting in increased HHV [58,59].

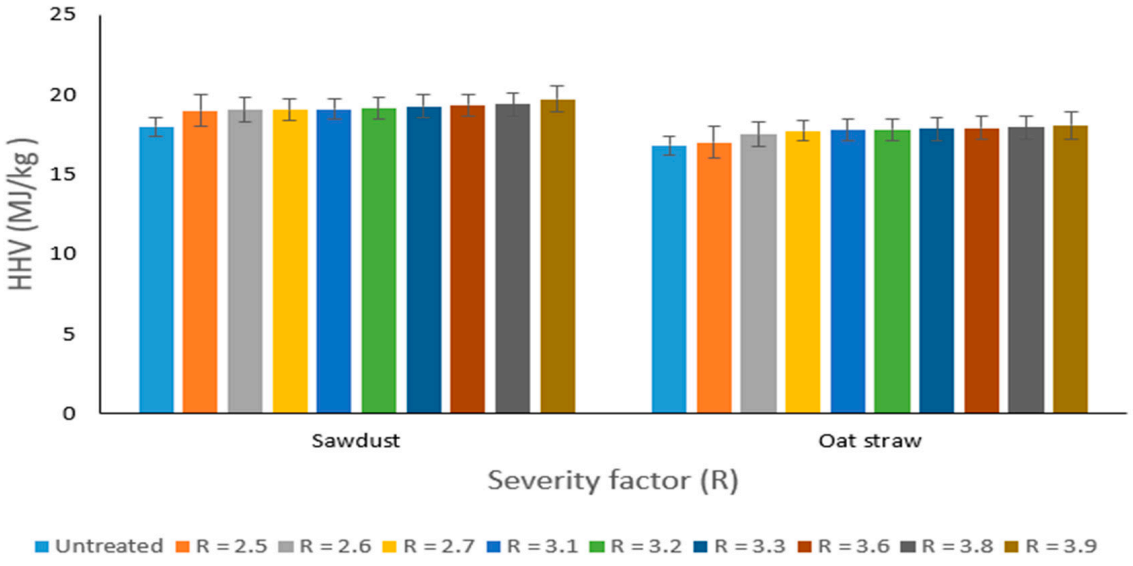

Figure 3. Higher heating value of steam-exploded and untreated biomass under different severities.

3.6. Moisture Absorption

Generally, the absorption of moisture by raw biomass samples occurs because of the presence of the OH group. During the steam explosion pretreatment, the lignocellulosic structure of the straw and sawdust are disintegrated by the sudden pressure change (from a very high value to 0 kPa); the lignin and hemicellulose content are hydrolyzed by this process, a portion of which are washed and drained with wastewater. This process tends to destroy the OH group, thereby causing the biomass sample to lose the ability to form

hydrogen bonds [60,61]. Several research groups optimized the steam explosion condition and concluded that less severe conditions resulted in the recovery of hemicelluloses. Wu et al. [62] used optimization of steam explosion to enhance hemicellulose recovery and enzymatic hydrolysis of cellulose in softwoods. In addition, Qing et al. [48] stated that mild conditions of steam explosion caused less sugar degradation when corn stover was pretreated. Figure 4 represents the graph of moisture content against time when the pellets were kept in the humidity chamber. After 1 h of subjecting the pellets to the humidifier condition, the untreated samples exhibited the least hydrophobic behavior, with a moisture uptake of 6.24% and 5.60% for oat straw and sawdust, respectively. The highest hydrophobic characteristic was observed in sawdust pellet samples steam exploded at 200 °C with moisture uptake of 2.89%. This could be explained by the fact that hemicelluloses were responsible for moisture absorption and biological degradation, therefore a decreased hemicellulose content reduced the total moisture absorption of the biomass. Lam [35] found similar results when Douglas fir (*Pseudotsuga taxifolia*) wood chips were steam exploded. Hence, steam-exploded pellets are less vulnerable to biological deterioration and distortion after moisture absorption. This study shows that steam-exploded pellet samples became more hydrophobic as the treatment severity condition increased.

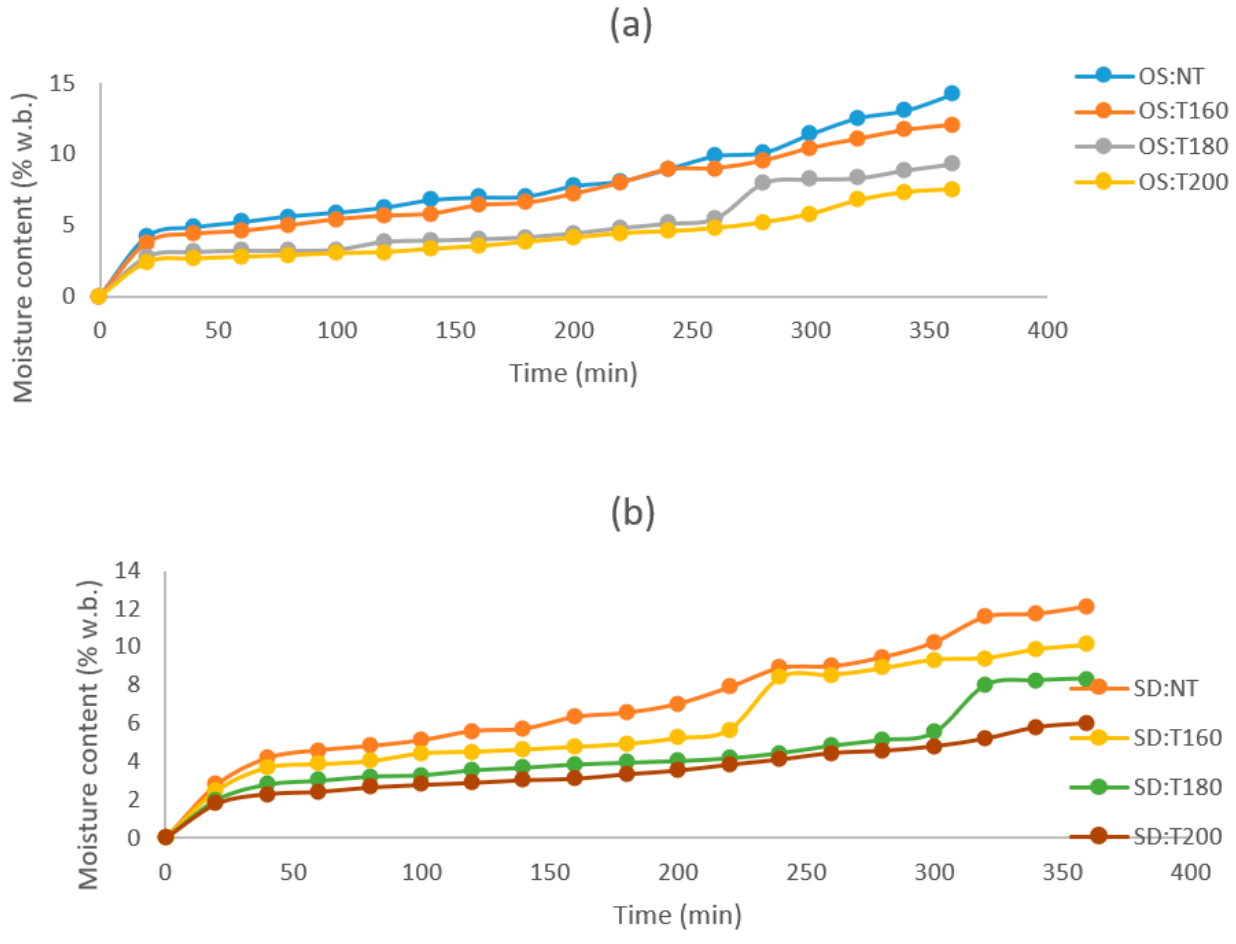

Figure 4. Moisture absorption of steam-treated and non-treated oat straw (**a**) and sawdust (**b**) pellets. OS: NT (oat straw: untreated); OS: T160 (oat straw: steam exploded at 160 °C); OS: T180 (oat straw: steam exploded at 180 °C); OS: T200 (oat straw: steam exploded at 200 °C); SD: NT (sawdust: untreated); SD: T160 (sawdust: steam exploded at 160 °C); SD: T180 (sawdust: steam exploded at 180 °C); SD: T200 (sawdust: steam exploded at 200 °C).

3.7. Microstructural Analysis

The microscale impact of the steam explosion pretreatment of sawdust and oat straw grinds can be explained further by microstructural analysis, giving insight on the morphology of the pellet samples. Figure 5a–h show the SEM micrographs of the longitudinal cross-section of the untreated and steam-exploded biomass pellets. Figure 5a,e show the pellet sample from the untreated oat straw and sawdust, respectively, with pore spaces, and the rough surface with more bonded particles that are loosely compacted are evident. The SEM images of the pellets from steam-exploded oat straw and sawdust (Figure 5b–d,f–h) reveal more tightly bonded particles and a cemented surface with fewer pores when compared with the untreated oat straw and sawdust. A lengthy defibrillation of fibers was noticed after the steam explosion pretreatment, mainly because of the mechanical effect from adiabatic expansion of absorbed water during the process. This shows that the biodeterioration of the lignocellulosic components of the biomass during steam explosion liberated more of the essential binders in the sample, resulting in high pellet tensile strength observed in the steam-exploded oat straw and sawdust pellets relative to the untreated oat straw and sawdust pellets. Similar observations were reported by some researchers [63,64].

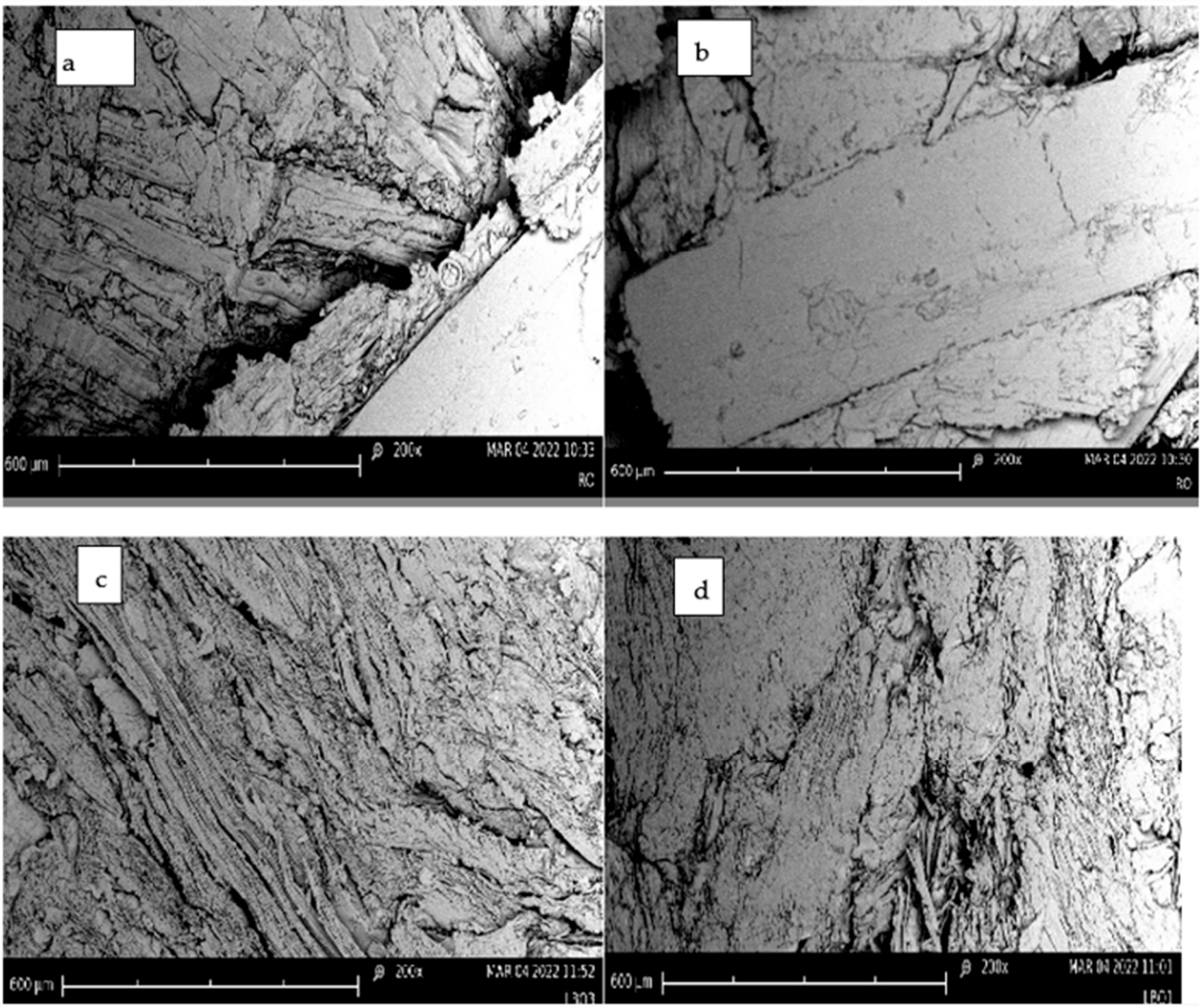

Figure 5. *Cont.*

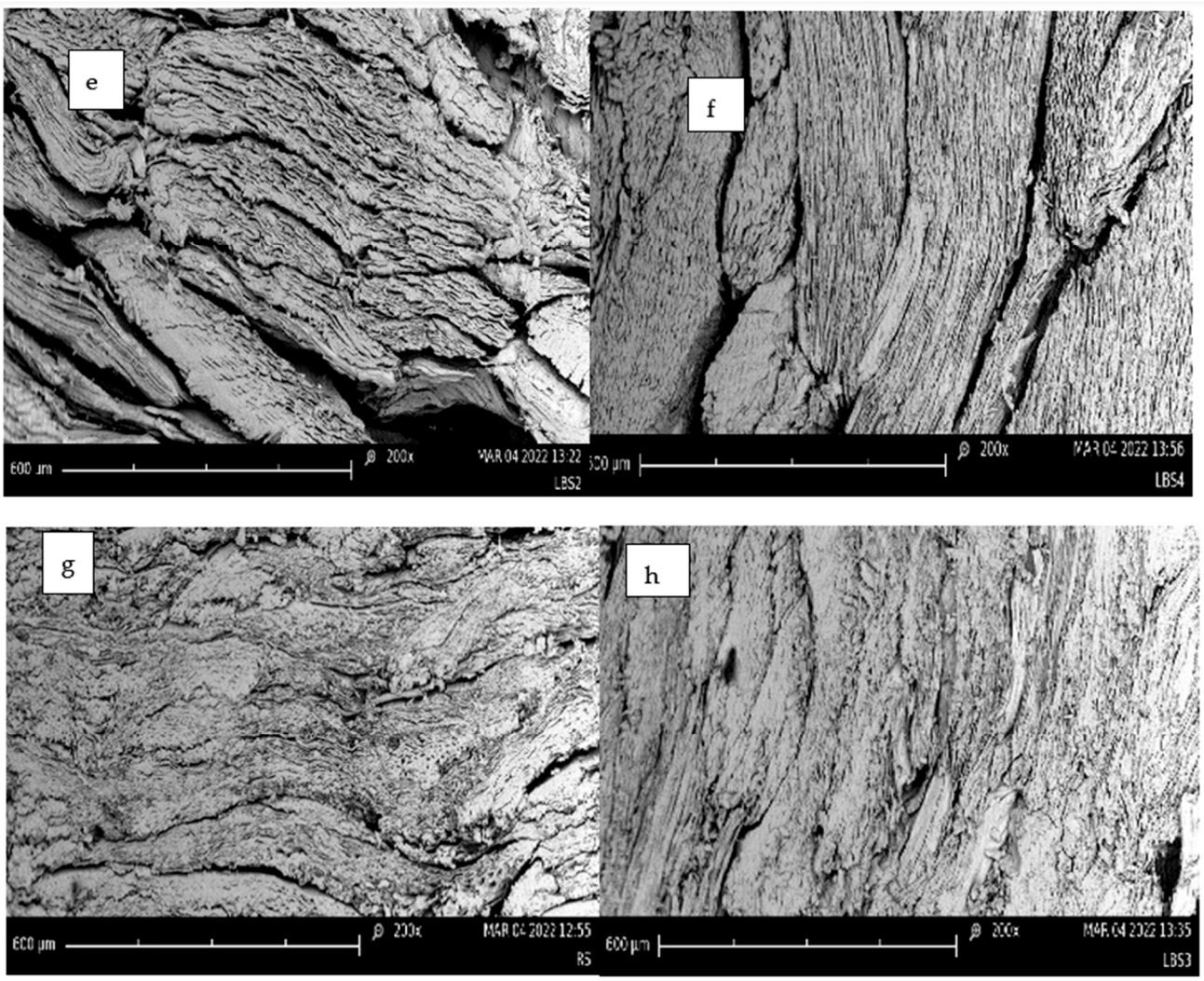

Figure 5. Scanning electron microscope images of the longitudinal cross-section of the untreated and steam-treated oat straw and sawdust pellets: (**a**) OS: NT (oat straw: untreated); (**b**) OS: T160 (oat straw: steam exploded at 160 °C); (**c**) OS: T180 (oat straw: steam exploded at 180 °C); (**d**) OS: T200 (oat straw: steam exploded at 200 °C); (**e**) SD: NT (sawdust: untreated); (**f**) SD: T160 (sawdust: steam exploded at 160 °C); (**g**) SD: T180 (sawdust: steam exploded at 180 °C); (**h**) SD: T200 (sawdust: steam exploded at 200 °C).

3.8. Effects of Independent Variables and Their Interactions on Pellet Quality

The effect of the steam explosion temperature, time, and moisture content after factorial regression are presented in Table 3. The analysis of variance (ANOVA) results showed that the *p*-values for all the response variables (dimensional stability, pellet tensile strength, and pellet density) studied showed a significant regression value ($p < 0.05$), excluding the pellet density of oat straw. The relationship among the three factors temperature, time, and moisture content of sample (*T*, *t*, *MC*) did not show significant effect on the response variables of oat straw pellets. Sample moisture content also affected the pellet density sawdust significantly. Generally, as sample moisture content increased, pellet density decreased. The compaction of hay across a wide range of moisture contents (28–44% w.b.) was studied by Gustafson and Kjelgaard [65], where they reported a decrease in density of the pellets as sample moisture contents increased. Furthermore, Rehkugler and Buchele [66] noticed that the relaxed density of pellets decreased with respect to sample moisture content ranging between 6% and 25% (w.b.). The results also indicate a statistically insignificant lack of fit for all the response variables, confirming the validity of the developed models. For

both biomass samples, steam explosion temperature and retention time had a significant effect ($p < 0.05$) on all the response variables investigated. Among the independent variables, the sample moisture content indicated no significant effect on the response variables of the oat straw pellets. This may be because of the fibrous nature of oat straw.

Table 3. Analysis of variance (ANOVA) table for factors affecting dimensional stability, pellet density, and tensile strength of biomass pellets.

	Sawdust					Oat Straw				
Source	**DF**	**SS**	**MS**	**F-Value**	***p*-Value**	**DF**	**SS**	**MS**	**F-Value**	***p*-Value**
Dimensional stability										
Temperature (*T*)	1	98.80	98.80	708.55	0.00	1	132.74	132.74	584.37	0.00
Time (t)	1	7.46	7.46	53.52	0.00	1	14.15	14.15	62.30	0.00
Moisture Content (*MC*)	1	0.55	0.55	3.93	0.06	1	1.81	1.81	7.95	0.01
$T \times t$	1	0.87	0.87	6.24	0.02	1	0.61	0.61	2.67	0.12
$T \times MC$	1	0.05	0.05	0.37	0.55	1	0.03	0.03	0.14	0.72
$t \times MC$	1	0.03	0.03	0.20	0.66	1	0.02	0.02	0.09	0.77
$T \times t \times MC$	1	0.07	0.07	0.49	0.49	1	0.06	0.06	0.26	0.62
Error	19	2.65	0.14			19	4.32	0.23		
Total	26	110.47				26	153.73			
Pellet Density										
Temperature (*T*)	1	16,138.90	16,138.90	25.24	0.00	1	28,251.40	28,251.40	36.51	0.00
Time (t)	1	3943.30	3943.30	6.17	0.02	1	2969.10	2969.10	3.84	0.07
Moisture Content (*MC*)	1	250.00	250.00	0.39	0.44	1	189.90	189.90	0.25	0.63
$T \times t$	1	6.60	6.60	0.01	0.92	1	141.00	141.00	0.18	0.67
$T \times MC$	1	65.20	65.20	0.1	0.75	1	39.90	39.90	0.05	0.82
$t \times MC$	1	543.20	543.20	0.85	0.37	1	2.60	2.60	0.00	0.95
$T \times t \times MC$	1	482.50	482.50	0.75	0.40	1	4.10	4.10	0.01	0.94
Error	19	12,150.60	639.50			19	14,701.40	773.80		
Total	26	33,580.30				26	46,299.40			
Tensile strength										
Temperature (*T*)	1	18.34	18.34	228.95	0.00	1	9.25	9.25	396.47	0.00
Time (t)	1	2.58	2.58	32.16	0.00	1	1.84	1.84	78.77	0.00
Moisture Content (*MC*)	1	0.03	0.03	0.39	0.44	1	0.03	0.03	1.24	0.28
$T \times t$	1	0.14	0.14	1.76	0.20	1	0.15	0.15	6.61	0.02
$T \times MC$	1	0.07	0.07	0.86	0.37	1	0.01	0.01	0.26	0.62
$t \times MC$	1	0.13	0.13	1.60	0.22	1	0.00	0.00	0.02	0.90
$T \times t \times MC$	1	0.01	0.01	0.14	0.71	1	0.00	0.00	0.02	0.89
Error	19	1.52	0.08			19	0.44	0.02		
Total	26	22.82				26	11.71			

4. Conclusions

This work investigated the effects of temperature, time, and sample moisture content on the mechanical properties of biomass (oat straw and spruce sawdust) pellets. These variables showed a significant effect on all the response variables studied for sawdust but not for oat straw. This study indicated that the most significant ($p < 0.05$) factors in the steam explosion of the biomass for pellet production were temperature and time. The p-values associated with the regression models for the dimensional stability, pellet unit density, and tensile strength of the steam exploded pellets were significant ($p < 0.05$). Investigation of the steam explosion technology shows that some factors influence the productivity of the process, including retention time, initial sample moisture content, and the pressure. Steam explosion breaks up the lignocellulosic structure of biomass, leading to lower bulk densities of pellets. After steam explosion pretreatment, the carbon and the nitrogen content of the steam-exploded sample increased significantly ($p < 0.01$), concomitant with temperature and retention time, while biomass hydrogen and oxygen content decreased. Hydroxyl groups are linked to biomass structural components, and their volatilization during steam explosion leads to an increase in the energy density of the residual carbon-rich solids. In addition, the carbonization and autohydrolysis process in the steam explosion removes the extractives with low volatility, resulting in increased HHV. Higher moisture content negatively affects the HHV of the solid pellet, while increased steam explosion temperature

positively affects the HHV. During the steam explosion pretreatment, the lignocellulosic structure of the straw and sawdust are disintegrated by the sudden pressure change and the lignin and hemicellulose are hydrolyzed by this process, a portion of which are washed and drained with wastewater. This process tends to destroy the OH group, thereby causing the biomass sample to lose the ability to form a hydrogen bond and increasing the hydrophobic characteristic of the sample. This study showed that steam-exploded pellet samples became more hydrophobic as the treatment severity condition increased. Extensive defibrillation of fibers was observed after the steam explosion pretreatment, mainly because of the mechanical effect from the adiabatic expansion of absorbed water during the process. SEM images of the pellets from steam-exploded pellets reveal more tightly bonded particles and cemented surfaces with fewer pores when compared with the untreated pellets. In this study, steam explosion pretreatment resulted in positive effects on the quality of pellets.

Supplementary Materials: The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/en15197168/s1, Table S1: Severity factor of the steam explosion pretreatment used in the experiment; Figure S1: Photograph of the steam explosion system used in the experiments.

Author Contributions: Conceptualization, C.O., L.G.T. and T.D.; methodology, C.O. and L.G.T.; software, C.O.; validation, L.G.T. and T.D.; formal analysis, C.O.; investigation, C.O.; resources, L.G.T., T.D., E.M., D.C., P.A. and C.K.; data curation, C.O.; writing—original draft preparation, C.O.; writing—review and editing C.O., L.G.T., T.D., E.M., D.C., P.A. and C.K.; visualization, C.O.; supervision, L.G.T. and T.D.; project administration, L.G.T., T.D., E.M. and D.C.; funding acquisition, L.G.T., T.D., E.M. and D.C. All authors have read and agreed to the published version of the manuscript.

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References

1. Tumuluru, S.J.; Sokhansanj, S.; Wright, C.T.; Boardman, R.D. REVIEW: A Review on Biomass Torrefaction Process and Product Properties for Energy Applications. *Ind. Biotechnol.* **2011**, *7*, 384–401. [CrossRef]
2. Natural Resources Canada (NRC). Forest Bioenergy Report. 2016. Available online: https://www.nrcan.gc.ca/forests/industry/bioproducts/13325 (accessed on 31 May 2019).
3. Sarkar, N.; Ghosh, S.K.; Bannerjee, S.; Kaustav, A. Bioethanol Production from Agricultural Wastes: An Overview. *Renew. Energy* **2012**, *37*, 19–27. [CrossRef]
4. Rabemanolontsoa, H.; Saka, S. Various Pretreatments of Lignocellulosics. *Bioresour. Technol.* **2016**, *199*, 83–91. [CrossRef] [PubMed]
5. Adapa, P.K.; Schonenau, L.G.; Canam, T.; Dumonceaux, T. Quantitative Analysis of Lignocellulosic Components of Non-treated and Steam Exploded Barley, Canola, Oat and Wheat Straw Using Fourier Transform Infrared Spectroscopy. *J. Agric. Sci. Technol.* **2011**, *1*, 177–188.
6. Saskatchewan Agriculture, Natural Resources, and Industry. Oat Production and Market Factsheet. 2017. Available online: http://publications.gov.sk.ca/documents/20/84129-oat%20production%20and%20markets.pdf (accessed on 6 April 2021).
7. Tavakoli, H.; Mohtasebi, S.S.; Jafari, A.; Mahdavinejad, D. Power Requirements for Particle Size Reduction of Wheat Straw as a Function of Straw Threshing unit Parameter. *Aust. J. Crop Sci.* **2009**, *3*, 231–236.
8. Canadian Council of Forest Ministry (CCFM). Fact Sheet Report on Bioenergy from Canadian Forest. 2018. Available online: www.sfmcanada.org (accessed on 22 December 2018).

9. Saskatchewan Forest Products Buyers' Guide. 2019. Available online: http://reaerch.yahoo.com/fpinnovations.ca (accessed on 6 April 2021).
10. Kim, S.; Dale, B.E. Global Potential Bioethanol Production from Wasted Crops and Crop Residues. *Biomass Bioenergy* **2004**, *26*, 361–375. [CrossRef]
11. Azargohar, R.; Nanda, S.; Kang, K.; Bond, T.; Karunakaran, C.; Dalai, A.K.; Kozinski, J.A. Effects of Bio-Additives on the Physicochemical Properties and Mechanical Behavior of Canola Hull Fuel Pellets. *Renew. Energy* **2019**, *132*, 296–307. [CrossRef]
12. Medic, D.; Darr, M.; Shah, A.; Potter, B.; Zimmerman, J. Effects of Torrefaction Process Parameters on Biomass Feedstock Upgrading. *Fuel* **2012**, *91*, 147–154. [CrossRef]
13. Haykiri-Acma, H.; Yaman, S.; Kucukbayrak, S. Combustion Characteristics of Torrefied Biomass Materials to Generate Power. In Proceedings of the 4th IEEE International Conference on Smart Energy Grid Engineering, SEGE 2016, Oshawa, ON, Canada, 21–24 August 2016; Institute of Electrical and Electronics Engineers Inc.: Piscataway, NJ, USA, 2016; pp. 226–230.
14. Ade-Omowaye, B.I.O.; Angersbach, A.; Taiwoy, K.A.; Knorrm, D. Use of Pulsed Electric Field Pre-treatment to Improve Dehydration Characteristics of Plant-Based Foods. *Trends Food Sci. Technol.* **2001**, *12*, 285–295. [CrossRef]
15. Bagby, M.O. *International Symposium on Ethanol from Biomass, Winnipeg*; Duckworth, H.E., Thompson, E.A., Eds.; The Royal Society of Canada: Ottawa, MB, Canada, 1982; pp. 561–569.
16. Bazhal, M.I.; Ngadi, M.O.; Raghavan, V.G.S. Influence of Pulsed Electroplasmolysis on the Porous Structure of Apple Tissue. *Biosyst. Eng.* **2003**, *86*, 51–57. [CrossRef]
17. Focher, B.; Marzetti, A.; Marsano, E.; Conio, G.; Tealdi, A.; Cosani, A.; Terbojevich, M. Regenerated and Graft Copolymer Fibres from Steam-Exploded Wheat straw: Characterization and Properties. *J. Appl. Polym. Sci.* **1998**, *67*, 961–974. [CrossRef]
18. Jacquet, N.; Maniet, G.; Vanderghem, C.; Delvigne, F.; Richel, A. Application of Steam Explosion as Pretreatment on Lignocellulosic Material: A Review. *Ind. Eng. Chem. Res.* **2015**, *54*, 2593–2598. [CrossRef]
19. Onu Olughu, O.; Tabil, L.G.; Dumonceaux, T.; Mupondwa, E.; Cree, D. Comparative Study on Quality of Fuel Pellets from Switchgrass Treated with Different White-Rot Fungi. *Energies* **2021**, *14*, 7670. [CrossRef]
20. Sokhansanj, S.; Mani, S.; Bi, X.; Zaini, P.; Tabil, L.G. *Binderless Pelletization of Biomass. Paper No. 056061*; ASABE: Portland, OR, USA; St. Joseph, MI, USA, 2005.
21. Mupondwa, E.; Li, X.; Tabil, L.; Sokhansanj, S.; Adapa, P. Status of Canada's Lignocellulosic Ethanol: Part I: Pretreatment Technologies. Renew. *Sustain. Energy Rev.* **2017**, *72*, 178–190. [CrossRef]
22. Jiang, L.; Yuan, X.; Xiao, Z.; Liang, J.; Li, H.; Cao, L.; Wang, H.; Chen, X.; Zeng, G. A Comparative Study of Biomass Pellet and Biomass-Sludge Mixed Pellet: Energy Input and Pellet Properties. *Energy Convers. Manag.* **2016**, *126*, 509–515. [CrossRef]
23. Lam, P.S.; Sokhansanj, S.; Bi, X.T.; Lim, C.J.; Larsson, S.H. Drying Characteristics and Equilibrium Moisture Content of Steam-Treated Douglas Fir (*Pseudotsuga menziesii* L.). *Bioresour. Technol.* **2012**, *116*, 396–402. [CrossRef]
24. Sun, X.F.; Xu, F.; Sun, R.C.; Fowler, P.; Baird, M.S. Characteristics of Degraded Cellulose Obtained from Steam-Exploded Wheat Straw. *Carbohydr. Res.* **2005**, *340*, 97–106. [CrossRef]
25. Kashaninejad, M.; Tabil, L.G. Effect of Microwave-Chemical Pre-Treatment on Compression Characteristics of Biomass Grinds. *Biosyst. Eng.* **2011**, *108*, 36–45. [CrossRef]
26. Li, X.; Yan, J.; Yang, J.; Peng, T.; Yang, Q. Study on Processing Technology for Microwave Pyrolysis of Municipal Solid Waste. In Proceedings of the 2011 International Conference on Materials for Renewable Energy Environment, Shanghai, China, 20–22 May 2011; pp. 336–340.
27. Azargohar, R.; Soleimani, M.; Nosran, S.; Bond, T.; Karunakaran, C.; Dalai, A.K.; Tabil, L.G. Thermo-Physical Characterization of Torrefied Fuel Pellet from Co-Pelletization of Canola Hulls and Meal. *Ind. Crops Prod.* **2019**, *128*, 424–435. [CrossRef]
28. American Society of Agricultural Engineers. *Standard Test for Moisture Content Measurement of Forages*; ANSI/ASAE S358.3; American Society of Agricultural Engineers: St. Joseph, MI, USA, 2012.
29. AACC Standard 44-15A. *Determination of Moisture Content by the Air-Oven Method*; The American Association of Cereal Chemists (AACC International): St. Paul, MN, USA, 2005.
30. Iroba, K.L.; Baik, O.-D.; Tabil, L.G. Torrefaction of Biomass from Municipal Solid Waste Fractions II: Grindability Characteristics, Higher Heating Value, Pelletability and Moisture Adsorption. *Biomass Bioenergy* **2017**, *106*, 8–20. [CrossRef]
31. Satpathy, S.K.; Tabil, L.G.; Meda, V.; Narayana, S.; Rajendra, S. Torrefaction and Grinding Performance of Wheat and Barley Straw after Microwave Heating. In Proceedings of the CSBE/SCGAB 2013 Annual Conference, Saskatoon, SK, Canada, 7–10 July 2013; Available online: http://www.csbescgab.ca/docs/meetings/2013/CSBE13065 (accessed on 15 May 2022).
32. *ASABE Standards EP496.3 49th ed.*; Agricultural Machinery Management. ASABE: St. Joseph, MI, USA, 2006; pp. 385–390.
33. Adapa, P.K. Densification of Selected Agricultural Crop Residues as Feedstock for the Biofuel Industry. Unpublished Ph.D. Thesis, Department of Chemical and Biological Engineering, University of Saskatchewan, Saskatoon, SK, Canada, 2011.
34. Cullis, I.F.; Saddler, J.N.; Mansfield, S.D. Effect of Initial Moisture Content and Chip Size on the Bioconversion Efficiency of Softwood Lignocellulosics. *Biotechnol. Bioeng.* **2004**, *85*, 413–421. [CrossRef] [PubMed]
35. Overend, R.P.; Chornet, E. Fractionation of Lignocellulosics by Steam Aqueous Pretreatments. *Phil. Trans. R. Soc. Lond. A* **1987**, *321*, 523–536.
36. Lam, P.S. Steam Explosion of Biomass to Produce Durable Wood Pellets. Unpublished Ph.D. Thesis, Department of Chemical and Biological Engineering, University of British Columbia, Vancouver, BC, Canada, 2011.

37. Kobayashi, N.; Okada, N.; Hirakawa, A.; Sato, T.; Kobayashi, J.; Hatano, S.; Itaya, Y.; Mori, S. Characteristics of Solid Residues Obtained from Hot-Compressed Water Treatment of Woody Biomass. *Ind. Eng. Chem. Res.* **2009**, *48*, 373–379. [CrossRef]
38. Ibrahim, M.; Glasser, W.G. Steam Assisted Biomass Fractionation. Part III: A Quantitative Evaluation of the Clean Fractionation Concept. *Bioresour. Technol.* **1999**, *70*, 181–192. [CrossRef]
39. Emmel, A.; Mathias, A.L.; Wypych, F.; Ramos, L.P. Fractionation of Eucalyptus Grandis Chips by Dilute Acid Catalysed Steam Explosion. *Bioresour. Technol.* **2003**, *86*, 105–115. [CrossRef]
40. Mani, S.; Tabil, L.G.; Sokhansanj, S. Grinding Performance and Physical Properties of Wheat and Barley Straws, Corn Stover, and Switchgrass. *Biomass Bioenergy* **2004**, *27*, 339–352. [CrossRef]
41. Tabil, L.G. Binding and Pelleting Characteristics of Alfalfa. Ph.D. Thesis, Department of Agricultural and Bioresource Engineering, University of Saskatchewan, Saskatoon, SK, Canada, 1996.
42. Shaw, M.D.; Karunakaran, C.; Tabil, L.G. Physicochemical Characteristics of Densified Untreated and Steam Exploded Poplar Wood and Wheat Straw Grinds. *Biosyst. Eng.* **2009**, *103*, 198–207. [CrossRef]
43. Ferro, T.; Torres, A.; Soler, P.B.; Zanzi, R.D. Biomass Torrefaction. In *ETA-Florence, and WIP-Munich, Proceedings of the 2nd World Conference on Biomass for Energy, Industry and Climate Protection, Rome, Italy, 10–14 May 2004*; Fjällström, V.S., Grassi, H., Eds.; Palazzo dei Congressi: Rome, Italy, 2004; pp. 859–862.
44. Iroba, K.L. Effect of Pretreatment on the Breakdown of Lignocellulosic Matrix in Barley Straw as Feedstock for Biofuel Production. Unpublished Ph.D. Thesis, Department of Chemical and Biological Engineering, University of Saskatchewan, Saskatoon, SK, Canada, 2014.
45. Yan, W.; Acharjee, T.C.; Coronella, C.J.; Vasquez, V.R. Thermal Pretreatment of Lignocellulosic Biomass. *Environ. Prog. Sustain. Energy* **2009**, *28*, 435–440. [CrossRef]
46. Angles, M.N.; Reguant, J.; Garcia-Valls, R.; Salvado, J. Characteristics of Lignin Obtained from Steam-Exploded Softwood with Soda/Anthraquinone Pulping. *Wood Sci. Technol.* **2003**, *37*, 309–320. [CrossRef]
47. Glasser, W.G.; Wright, R.S. Steam Assisted Biomass Fractionation II. Fractionation Behavior of Various Biomass Resources. *Biomass Bioenergy* **1998**, *14*, 219–235. [CrossRef]
48. Qing, Q.; Yang, B.; Wyman, C.E. Xylooligomers are Strong Inhibitors of Cellulose Hydrolysis by Enzymes. *Bioresour. Technol.* **2010**, *101*, 131–146. [CrossRef] [PubMed]
49. Selig, M.J.; Knoshaug, E.P.; Adney, W.S.; Himmel, M.E.; Decker, S.R. Synergistic Enhancement of Cellubiohydrolase Performance on Pretreated Corn Stover by Addition of Xylanase and Esterase Activities. *Bioresour. Technol.* **2008**, *99*, 4997–5005. [CrossRef] [PubMed]
50. Lam, P.S.; Sokhansanj, S.; Bi, X.T.; Lim, C.J.; Melin, S. Energy Input and Quality of Pellets Made from Steam-Exploded Douglas Fir (Psedotsugamenziesii). *Energy Fuels* **2011**, *25*, 1521–1528. [CrossRef]
51. Tooyserkani, Z.; Sokhansanj, S.; Bi, X.T.; Lim, C.J.; Lau, A.; Saddler, J.; Kumar, L.; Lam, P.S.; Melin, S. Steam Treatment of Softwood Particles to Produce Torrefied Material. *Appl. Energy* **2013**, *103*, 514–521. [CrossRef]
52. He, X.; Wang, L.; Lau, A. Investigation of Steam Treatment on the Sorption Behavior of Rice Straw Pellets. *Energies* **2020**, *13*, 5401. [CrossRef]
53. Iroba, K.L.; Tabil, L.G.; Sokhansanj, S.; Dumonceaux, T.J. Pretreatment and Fractionation of Barley Straw using Steam Explosion at low Severity Factor. *Biomass Bioenergy* **2014**, *66*, 286–300. [CrossRef]
54. Han, G.; Deng, J.; Zhang, S.; Bicho, P.; Wu, Q. Effect of Steam Explosion Treatment on Characteristics of Wheat Straw. *Ind. Crop. Prod.* **2010**, *31*, 28–33. [CrossRef]
55. Bhattacharya, S.C.; Albina, D.O.; Khaing, A.M. Effects of Selected Parameters on Performance and Emission of Biomass-Fired Cookstoves. *Biomass Bioenergy* **2002**, *23*, 387–395. [CrossRef]
56. Yibo, H.; Haixi, L.; Xiaofu, C.; Chunyu, X.; Chang, C.; Guangqing, L. Effects of Moisture Content in Fuel on Thermal Performance and Emission of Biomass Semi-Gasified Cookstove. *Energy Sustain. Dev.* **2014**, *21*, 60–65.
57. Kumar, P.; Barrett, D.M.; Delwiche, M.J.; Stroeve, P. Methods for Pretreatment of Lignocellulosic Biomass for Efficient Hydrolysis and Biofuel Production. *Ind. Eng. Chem. Res.* **2010**, *48*, 3713–3729. [CrossRef]
58. Lam, P.S.; Lam, P.Y.; Sokhansanj, S.; Lim, C.J.; Bi, X.T.; Stephen, J.D. Steam Explosion of Oil Palm Residues for the Production of Durable Pellets. *Appl. Energy* **2015**, *141*, 160–166. [CrossRef]
59. Tang, Y.; Chandra, R.P.; Sokhansanj, S.; Saddler, J.N. Influence of steam explosion processes on the durability and enzymatic digestibility of wood pellets. *Fuel* **2018**, *211*, 87–94. [CrossRef]
60. Jin, S.; Chen, H. Superfine Grinding of Steam Exploded Rice Straw and its Enzymatic Hydrolysis. *Biochem. Eng. J.* **2006**, *30*, 225–230. [CrossRef]
61. Adapa, P.; Tabil, L.; Schoenau, G.; Opoku, A. Pelleting characteristics of selected biomass with and without steam explosion pretreatment. *Int. J. Agric Biol. Eng.* **2010**, *3*, 62–79.
62. Wu, W.W.; Chang, K.; Gregg, D.J.; Boussaid, A.; Beatson, R.P.; Saddler, J.N. Optiimization of Steam Explosion to Enhance Hemicelluloses Recovery and Enzymatic Hydrolysis of Cellulose in Softwoods. *Appl. Biochem. Biotechnol.* **1999**, *77*, 47–79. [CrossRef]
63. Gastaldi, G.; Capretti, G.; Focher, B.; Cosentino, C. Characterization and Properties of Cellulose Isolated from the *Crambe Abyssinica* Hull. *Ind. Crops Prod.* **1998**, *8*, 208–218. [CrossRef]

64. Kristensen, J.B.; Thygesen, L.G.; Felby, C.; Jorgensen, H.; Elder, T. Cell-Wall Structural Changes in Wheat Straw Pretreated for Bioethanol Production. *Biotechnol. Biofuels* **2008**, *1*, 5. [CrossRef]
65. Gustafson, A.S.; Kjelgaard, W.L. Hay Pellet Geometry and Stability. *Agric. Eng.* **1963**, *44*, 442–445.
66. Rehkugler, G.E.; Buchele, W.F. Bio-mechanics of Forage Wafering. *Trans. Am. Soc. Agric. Eng.* **1969**, *12*, 1–8.