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# Influence of Raw Material Drying Temperature on the Scots Pine (*Pinus sylvestris* L.) Biomass Agglomeration Process—A Preliminary Study

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**Abstract:** For biomass compaction, it is important to determine all aspects of the process that will affect the quality of pellets and briquettes. The low bulk density of biomass leads to many problems in transportation and storage, necessitating the use of a compaction process to ensure a solid density of at least  $1000 \text{ kg}\cdot\text{m}^{-3}$  and bulk density of at least  $600 \text{ kg}\cdot\text{m}^{-3}$ . These parameters should be achieved at a relatively low compaction pressure that can be achieved through the proper preparation of the raw material. As the compaction process includes a drying stage, the aim of this work is to determine the influence of the drying temperature of pine biomass in the range of  $60\text{--}140 \text{ }^\circ\text{C}$  on the compaction process. To determine whether this effect is compensated by the moisture, compaction was carried out on the material in a dry state and on the materials with moisture contents of 5% and 10% and for compacting pressures in the  $130.8\text{--}457.8 \text{ MPa}$  range. It was shown that drying temperature affects the specific density and mechanical durability of the pellets obtained from the raw material in the dry state, while an increase in the moisture content of the raw material neutralizes this effect.

**Keywords:** drying; compaction; biomass; pellets; mechanical durability; specific density; Scots pine

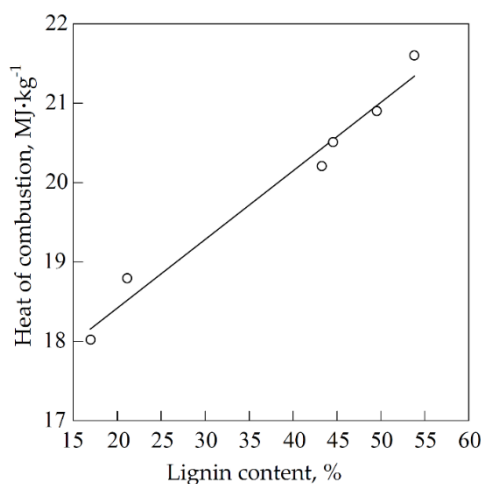
## 1. Introduction

Cell walls are the main structural elements of plant biomass. The composition and structure of cell walls vary depending on the plant species [1], cell function and response to environmental conditions [2]. However, from a chemical point of view, the cell walls of most plants consist of three main components, namely cellulose, hemicellulose and lignin. The biomass of this composition is called lignocellulose biomass [3–5]. Additionally, this natural composite may also contain extractives and a mineral fraction. The most common extractives are simple sugars, fats, waxes, proteins, phenols, gums, terpenes, saponins, pectin, resins, fatty acids and essential oils that mainly fulfill a protective function for the plants [6].

Chemically, lignocellulose biomass contains mainly about 50% of carbon and hydrogen—on average 8% [7,8]. This is why it is mainly used as a biofuel. Besides this, there are investigations into the use of biomass as a raw material for hydrogen production from biomass gasification [9–12].

From the point of view of energy use of biomass, most of the energy accumulated in a given biomass is found in cellulose, hemicellulose and lignin. An elementary analysis of several types of biomass has shown [7] that the differences between cellulose and hemicellulose are insignificant, so that their energy parameters are similar. The cellulosic heat of combustion is  $17.6 \text{ MJ}\cdot\text{kg}^{-1}$  and that for hemicellulose is  $17.9 \text{ MJ}\cdot\text{kg}^{-1}$ , whereas lignin has higher energy parameter values. The lignin heat of combustion depends on the type of biomass from which it was extracted. For example, the heats of

combustion for lignins from hazelnut shells, olive pomace, walnut shells, spruce and beech wood are in the 27–28 MJ·kg<sup>-1</sup> range [13,14]. Additionally, much higher heats of combustion of 35 MJ·kg<sup>-1</sup> are obtained for the extractives [6]. Thus, the biomass heat of combustion depends mainly on the relative contents of cellulose and hemicellulose on the one hand and lignin on the other hand. While the highest heat of combustion is found for extractives, particularly those containing large amounts of resins and terpenes, their small content in biomass (not exceeding 9.5%) [15–17] does not give rise to a significant increase in the heat of the combustion value of the raw material. The highest concentration of the extractives is found in the wood of coniferous plants, making the heat of the combustion values of these materials slightly higher compared to the wood of deciduous plants or herbaceous biomass [15,18,19]. Nevertheless, the main factor determining the high value of heat of combustion is the proportion of lignin in relation to holocellulose. It has been shown that, for certain biomass groups, this relationship is linear [7,17] (Figure 1). Therefore, despite the different levels of lignin, cellulose and hemicellulose in different types of biomass [15,20–23], their heat of combustion values lie within a narrow range of 18–22 MJ·kg<sup>-1</sup> [24–27].



**Figure 1.** Heat of combustion of dry, ash-free and extractive-free biomass of different plant species seed coatings as a function of the lignin content [17].

This relatively energy-homogeneous raw material has a wide range of sources such as coniferous and deciduous wood, grasses, dicotyledonous perennials, shells, and stones, and forms such as log wood, saw dust, chips, straw, and pomace. Therefore, biomass materials exhibit a wide range of specific and bulk density values. Most typical biomass raw materials used for pellet production have low specific densities in the approximately 200–450 kg·m<sup>-3</sup> range for the herbaceous type of biomass materials, and in the 350–720 kg·m<sup>-3</sup> range for the woody type of biomass materials [3,28,29]. According to EN ISO 17225-1:2014 standard [30], the main types of biomass for solid biofuel production are woody, herbaceous and fruity biomass (less common is so-called water biomass). This means that a very wide range of species are already used for this purpose and the range of the different plant species is being continuously extended as a result of research conducted at many scientific institutions worldwide [27,31–39].

This variability of biomass materials gives rise to many inconveniences related to the logistics and combustion of biomass. To fully exploit the energy potential of biomass and avoid these problems, biomass materials are densified, obtaining a higher energy density, and lower transportation and storage costs. Standardization of solid biofuels (pellets and briquettes) allows the automatization of feeding and burning processes in domestic and industrial-size boilers that is impossible for low-processed biomass. For pellets that represent the most compacted biofuels, quality standards EN ISO 17225-2:2014 standard [40], require the specific density (*DE*) to be equal to or greater than 1000 kg·m<sup>-3</sup> and mechanical durability (*DU*) to be equal to or greater than 97.5% (A1 quality class).

To obtain these parameters, it is necessary to properly prepare the raw material and properly set the parameters of the pressure compaction process to the requirements of the processed material. Many factors influence the compaction process. Pressure and compaction temperature, type, degree of fragmentation, friction coefficient and moisture content of the raw material are the most important factors [41–47].

The moisture content of the raw material is one of the most important factors that affect the compaction process, and is in the range of 6%–17% for most biomass materials [43]. In most cases, the biomass raw material has a moisture content higher than this level, so that a drying stage is almost always used in the granulation process. This process is carried out in a wide temperature range 40–200 °C or more [48–53]. According to the literature, the main components of the biomass such as lignin, cellulose, and hemicellulose can be transformed as a result of the temperature effect [49,54,55], which may affect the compaction process. During the drying of coniferous tree biomass, terpenic compounds that act as binders and energy carriers are also released [51]. It is known that the high temperature in the 150–300 °C range used in the torrefaction process also affects the compaction. This leads to an improvement in the raw material grindability but complicates the compactions process, mainly due to the decomposition of lignin, which acts as the binder during the densification [56–62].

Lignin plasticizes at temperatures of about 150 °C [15,63]; plasticization can occur at lower temperatures of 75–90 °C with the presence of moisture in the material [42,64]. A promising way of activating the properties of lignin as a binder is steam explosion [65,66]. It causes structural changes in biomass fiber and, under some pretreatment conditions, lignin in biomass cells comes out of the fiber. During the agglomeration process, lignin melts both on the particle surface and between particles. This results in the creation of a cover layer around pellets and bonds inside them [67]. The durability of such pellets is significantly higher than pellets from conventional materials [68].

The aim of this study was to investigate the effect of drying temperature on the process of Scots pine biomass compaction. Scots pine was chosen in this study because it is a typical biomass material that is widely used for the production of fuel pellets.

## 2. Materials and Methods

The research in this work was carried out according to the procedure shown in Figure 2.

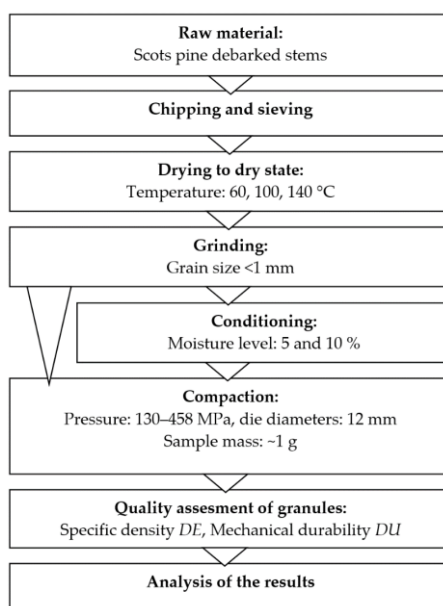


Figure 2. Flowchart of the procedure used for the investigations in this work.

## 2.1. Materials

Because it is the most popular raw material for fuel pellet production, Scots pine (*Pinus sylvestris* L.) biomass was used for the present study. According to the biomass classification [30], this material represents woody biomass. After the growth season, Scots pine stems were collected, debarked and chipped.

A set of analytical sieves with a diameter of 400 mm and hole sizes of 16 and 8 mm (Morek Multiserw, Marcyporęba, Poland) and a sieve shaker (LPzE-4e, Morek Multiserw, Marcyporęba, Poland) were used to separate the particle size fraction that passes through the sieve with the hole size of 16 mm and remains on the sieve with the hole size of 8 mm in order to ensure that the material is geometrically uniform prior to the drying stage. Pine chips were divided into three samples and were dried in a laboratory dryer (SLW 115, Pol-Eko, Wodzisław Śląski, Poland) to the dry state at the temperatures of 60, 100 and 140 °C.

After drying, all three samples were ground to obtain particles with a size of <1 mm using a hammer mill (PX-MFC 90D Polymix, Kinematika, Luzern, Switzerland). According to the literature [66,69–73], grain size and grain size distribution has a significant impact on the biomass agglomeration process. Therefore, in order to exclude this effect, the grain size distribution of all samples was standardized to the grain size distribution of the biomass sample dried in 60 °C. Samples were divided into four dimensional fractions, (sieve classes): C<sub>1</sub>: 0.1—grain diameter  $d \leq 0.1$  mm, C<sub>2</sub>: 0.25—grain diameter between  $0.1 < d \leq 0.25$  mm, C<sub>3</sub>: 0.5—0.25 <  $d \leq 0.5$  mm and C<sub>4</sub>: 1—0.5 <  $d \leq 1$  mm. The standardized grain size distribution of all samples is contained in Table 1.

**Table 1.** Standardized particle size distribution of samples.

Sieve Classes (mm)	C <sub>1</sub> : 0.1	C <sub>2</sub> : 0.25	C <sub>3</sub> : 0.5	C <sub>4</sub> : 1
Share [%]	8.2	16.1	38.2	37.5

The sample dried at 60 °C was also analyzed in terms of energy properties. The values of determined parameters are shown in Table 2.

**Table 2.** Characterization of raw material—all values refer to the dry state of sample.

Parameter	Unit	Value	Standard
Heat of combustion	MJ/g	20.9	EN ISO 18125 [74]
Ash content	%	0.2	EN ISO 18122 [75]
Volatile matter	%	79.8	EN ISO 18123 [76]

Each sample of dry, ground and size standardized material was divided into three subsamples, giving a final total of nine test samples. Three of the samples (dried at the examined temperatures) were left in the dry state, and from the remaining six samples, three samples were moisturized to 5% and three samples were moisturized to 10%. Based on the literature, these moisturization values were considered to be most suitable for investigating the effects of the moisture content on the pine pellet quality parameters [46,47,77]. The samples in the dry state were treated as controls (Figure 3). The materials were moisturized and conditioned in a climate chamber with controlled temperature and humidity (KBF-S 115, Binder, Tuttlingen, Germany). This allowed us to maintain a stable moisture content during the experiments, which was highly important for the accuracy of the investigations.



**Figure 3.** Samples ready for densification: from left to right, the samples of the raw material dried at 60, 100, and 140 °C, respectively, in the dry state are shown.

## 2.2. Samples Densification

The biomass compacts (test pellets) were produced using a compaction stand (hydraulic press P400, Sirio, Meldola, Italy) with a special appliance. The components of this stand are shown in Figure 4. The main parts of the stand are the compaction unit that consisted of hardened steel and a cylindrical die (with an internal die channel diameter of 12 mm and length of 110 mm). The unit also contains a piston and bottom (diameter of both was 11.9 mm). The compaction unit is placed between the press plate and pressure is applied by the pistons.



**Figure 4.** Lab-scale compaction stand for test pellets production: (a) hydraulic press, (b) samples of compacts, and (c) die, piston and bottom.

Using the compaction stand, we obtained precise information regarding the pressure used for sample densification. Based on the previous studies reported in the literature [3,78,79], six pressure values were selected, namely 130.8, 196.2, 216.6, 327, 392.4, and 457.8 MPa (14.5, 21.7, 29, 36.2, 43.5, 50.7 kN, respectively),

The mass of the sample (approximately 1 g) allowed us to obtain compacts with heights that were smaller than their diameters (approximately 8–10 mm). A total of 54 combinations of the different dry temperature, moisture content and pressure values were investigated in this work. To ensure the quality of the obtained results, for each set of conditions we examined three test pellets, even though the standards for *DU* and *DE* require the use of only two samples [80,81]. Thus, a total of 162 test pellets

were produced. After compaction, the samples were placed into individual labeled compartments to prevent moisture change and human error during further measurements.

### 2.3. Pellets Quality Parameters Analysis

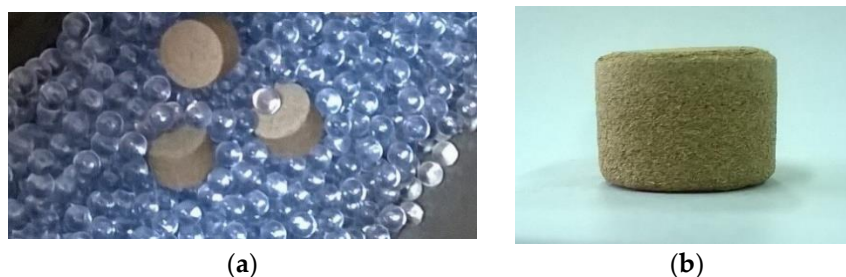
After waiting for 24 h after the compaction, the height, diameter and mass of the test pellets were measured using a caliper with an accuracy of 0.01 mm and a scale (AS 160.R2, Radwag, Radom, Poland) with an accuracy of 0.1 mg. The  $DE$  of each pellet was calculated according to the values of the specific densities, as shown in Equation (1)

$$DE = \frac{m_{24}}{V_{24}} \quad (1)$$

where  $DE$  is the specific density,  $m_{24}$  is the mass of the sample 24 h after compaction and  $V_{24}$  is the volume of the sample 24 h after compaction.

The mechanical durability ( $DU$ ) was also determined for these samples. Hardness and durability are very important parameters for logistics. While many standards refer to  $DU$  [57], the requirements for energetic pellets are described in the EN ISO 17831-1:2015 standard [81] dedicated standard. In this case, where only a few granules were produced, some modification of this method was necessary. This is because the standard procedure requires the use of approximately 500 g of pellets per test. In this research, only three granules were made, and the equipment described in the EN ISO 17831-1:2016-02 standard was used to simulate the damages incurred by the particles during logistics operations. The main experimental apparatus is a cuboidal chamber with two baffles inside that rotates at a rate of 50 rpm for 10 min.

The requirement for the 500 g mass of the sample was met by using ballast material in which three tested pellets were placed (Figure 5). For the volume of the ballast sample to be comparable to that of the classic sample, the ballast material should have a specific density similar to that of a typical pellet (approximately  $1000 \text{ kg}\cdot\text{m}^{-3}$ ). Therefore, polystyrene with a specific density of  $1070 \text{ kg}\cdot\text{m}^{-3}$  was used. The grains forming the ballast material had a sphere-like shape with a diameter of 5 mm (Figure 5). Some of the samples were made from the biomass in the dry state, which means that the strength of some compacts was low and, therefore, in order not to cause their complete disintegration during the test, it was decided to reduce the time of rotation. Based on previous research, the time of rotations was reduced to 5 min [3].



**Figure 5.** Test pellet sample: (a) the ballast material placed in the testing chamber, (b) after test with the visible crumbling zones.

After the test, the compacts were removed from the box, and weighed (scale AS 160.R2, Radwag, Radom, Poland with accuracy of 0.1 mg), and using Formula 2, the  $DU$  values of each sample were obtained. The obtained values are only compared for the materials used in this research, but further research on this method is in progress in order to compare the results obtained using this method for a variety of materials.

$$DU = \frac{m_A}{m_E} \cdot 100 \quad (2)$$

where  $DU$  is the mechanical durability,  $m_A$  is the mass of the sample after the test and  $m_E$  is the mass before the test.

The last modification to the basic procedure was the mode used for the calculation of the compact material's mechanical durability ( $DU$ ). In the standard, the  $DU$  result is related to the mass of the sample as a whole, while in the case of the conducted tests, the durability of each individual granule was determined, so that the result had to be related to its mass. The cylindrical granule was crushed only along the circumference of its base, while the central part of the cylinder side remained undamaged (Figure 5b). Thus, although the mass of the fines for two granules with the same diameter but different heights is comparable, when using the classic formula for  $DU$ , different calculated durability values are obtained for these two granules because the mass of the longer granule is greater than that of the shorter granule. To make the calculation of  $DU$  independent of the granule height, the mechanical durability of the equivalent  $DU_{10}$  granules with a constant height of 10 mm  $h_{10}$  was calculated as

$$DU_{10} = \frac{m_{A10}}{m_{E10}} \cdot 100 \quad (3)$$

where  $DU_{10}$  is the mechanical durability of the substitute compact,  $m_{A10}$  is the mass of the substitute compact after the test and  $m_{E10}$  is the mass of the substitute compact prior to the test.

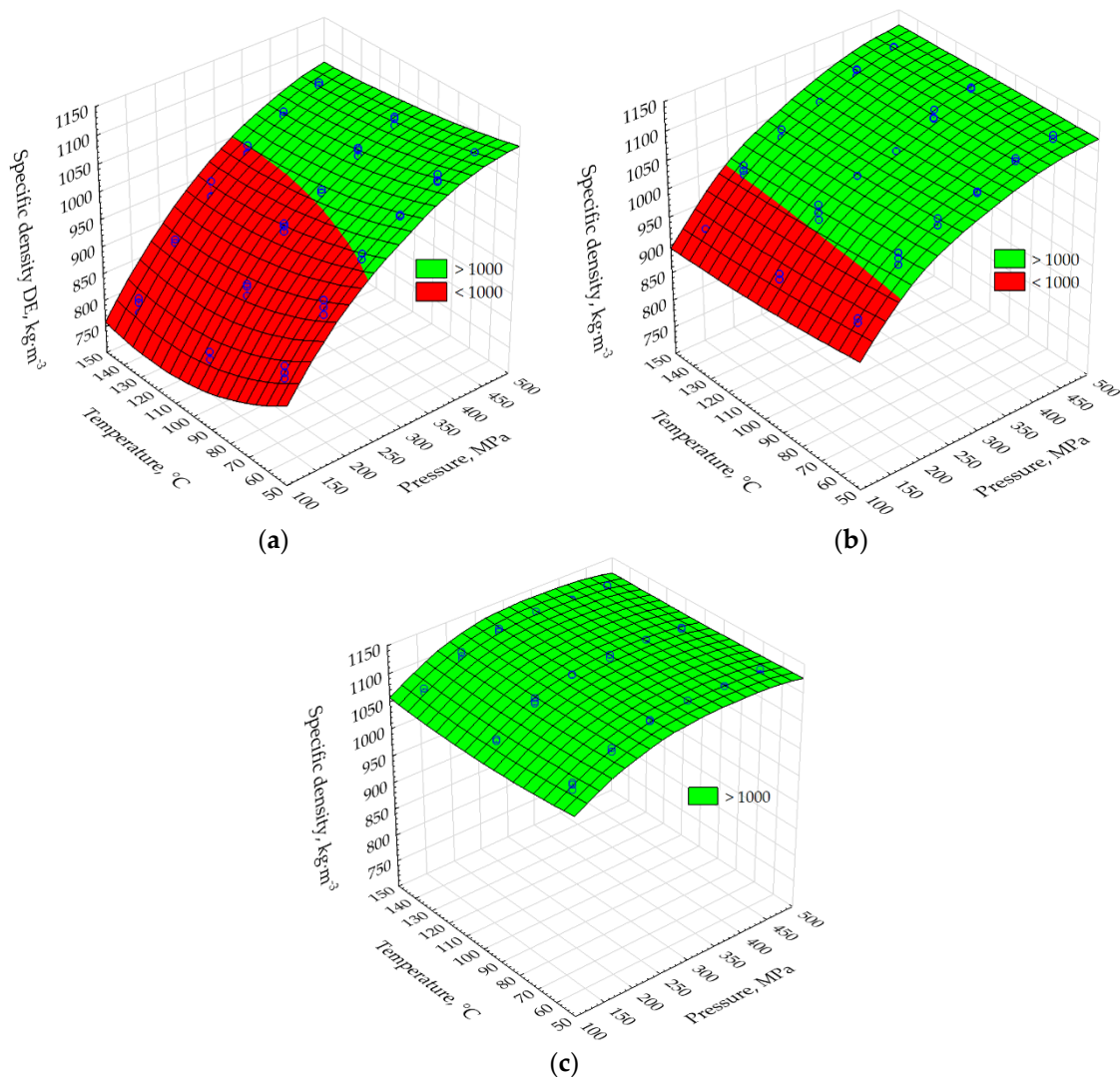
Due to the use of equivalent granules, the mechanical durability value does not depend on the height of the granules, enabling comparison of the  $DU_{10}$  results regardless of the height of the granules. The height of the granules was between 8 and 10 mm, and a more detailed description of the procedure use in this work can be found in a previous report [3].

### 3. Results

Figure 6 shows the changes in the pellets' specific density ( $DE$ ) due to the effects of the raw material drying temperature and the increase in the densification pressure. Figure 6a shows the changes for the material in the dry state and Figure 6b,c show the changes for the materials with moisture contents of 5% and 10%, respectively. For the dry materials, the applied pressure results in the pellets with  $DE$  values in the 794–1100 kg·m<sup>-3</sup> range. The threshold value of 1000 kg·m<sup>-3</sup> (the dividing line between the red and green part of the density variation area) was reached most rapidly for the material that was dried at 60 °C. This was achieved at the pressure of 261 MPa, whereas for the materials dried at 100 and 140 °C, the threshold values were achieved at pressures of 319 and 333 MPa, respectively.

For a test material moisturized to 5%, the area of the specific density variation over the entire range of the test pressures was shifted up and the  $DE$  values of the resulting pellets were in the 930–1120 kg·m<sup>-3</sup> range. The density threshold value was obtained in this case at 169 MPa, showing a lower value than that of the material in the dry state. As the drying temperature increases, this value increases slightly to 187 MPa, suggesting that the drying temperature of the raw material is less important in this case, while the increase in humidity has resulted in an increase in the pellet  $DE$ . The increase in the moisture content to 10% caused the whole pressure range to reach specific density values above the threshold value for all pellets. Similar evolutions of the density values were observed for all drying temperatures, showing that the density changes do not depend strongly on the raw material drying temperature. At first pressure level (130.8 MPa), the specific density was 1070 kg·m<sup>-3</sup>. This values increased with pressure, and was 1110 kg·m<sup>-3</sup> for 196.2 MPa and 1130 kg·m<sup>-3</sup> for 261.6 MPa. A further increase in the compaction pressure did not lead to an increase in the pellet specific density.

Changes in mechanical durability ( $DU$ ) for the same pellets are shown in Figure 7. The  $DU$  threshold value was set at 97.5% (this value is required for wood pellets of class A1 according to standard EN ISO 17225-2:2014 [40]). This is a theoretical value because the  $DU$  of the tested compacts was determined by the modified method. For the pellets obtained from raw material in the dry state (Figure 7a), the  $DU$  threshold level of the obtained pellets was obtained at a pressure of 392.4 MPa, and compacts fabricated at 130.8 MPa had a  $DU$  of only 65. No effect of the drying temperature was observed.



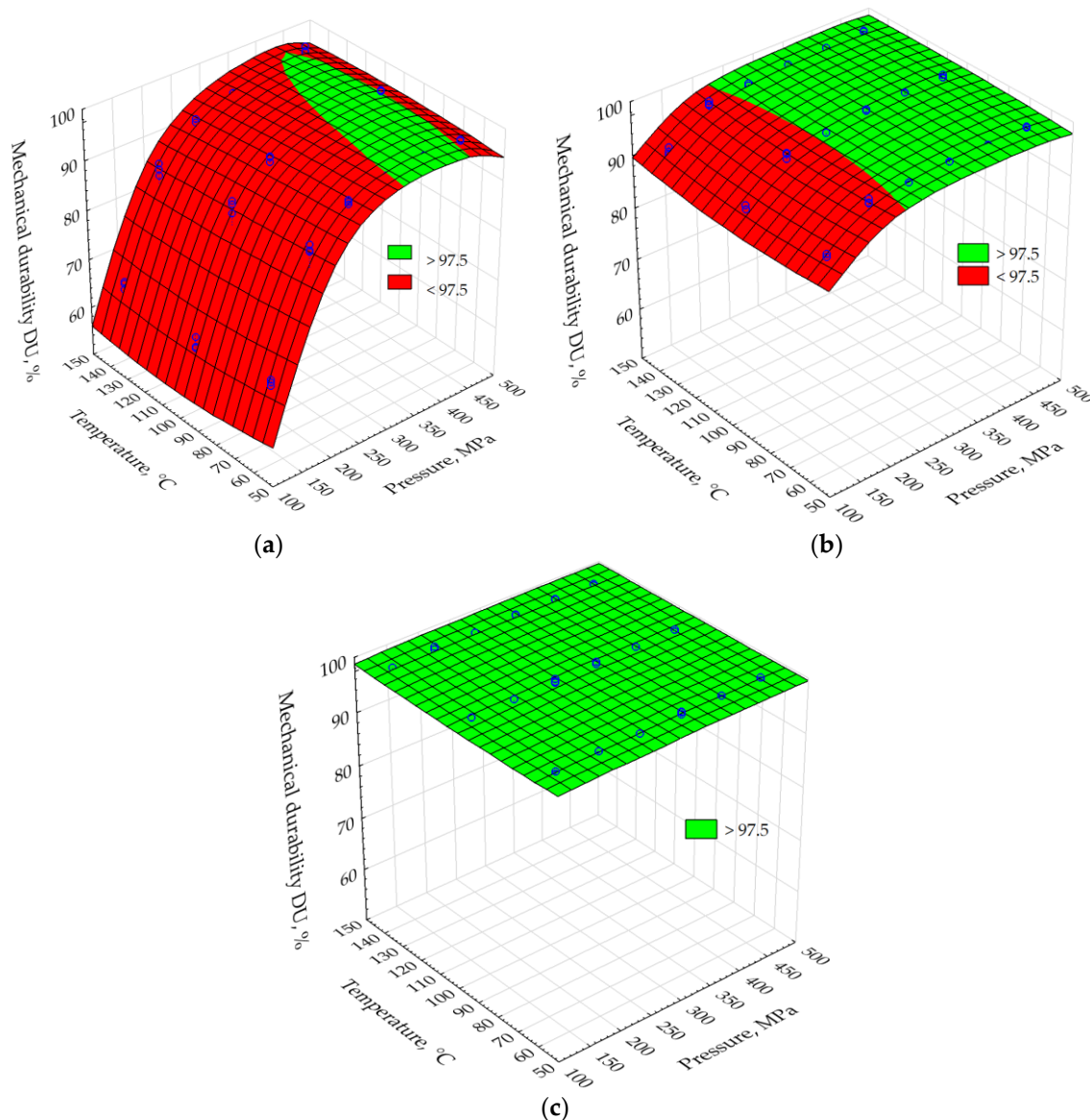
**Figure 6.** Pellet specific density ( $DE$ ) changes as a function of the raw material drying temperature and densification pressure: (a) raw material in the dry state, (b) with 5% moisture content, and (c) with 10% moisture content.

An increase in the raw material moisture content up to 5% causes the  $DU$  threshold value to be reached for pressures greater than 196.2 MPa, regardless of the raw material drying temperature (Figure 7b). At the lowest pressure, the  $DU$  of pellets was approximately 91%, which is much higher than that of the dry pellets. A further increase in the moisture content of the raw material to 10% led to an increase in the mechanical durability of the compacts above the threshold value, regardless of the applied pressure. (Figure 7c).

To confirm the significance of the discussed evolution of  $DE$  and  $DU$ , a statistical analysis was carried out for the obtained results. A detailed statistical analysis was performed for two hypotheses:

- The drying temperature of the raw material significantly affects the  $DE$  and  $DU$  values of the pellets obtained at the tested levels of pressure and raw material moisture content;
- The increase in the raw material moisture content compensated the influence of the drying temperature on the quality parameters ( $DE$  and  $DU$ ) for the tested pressure levels.





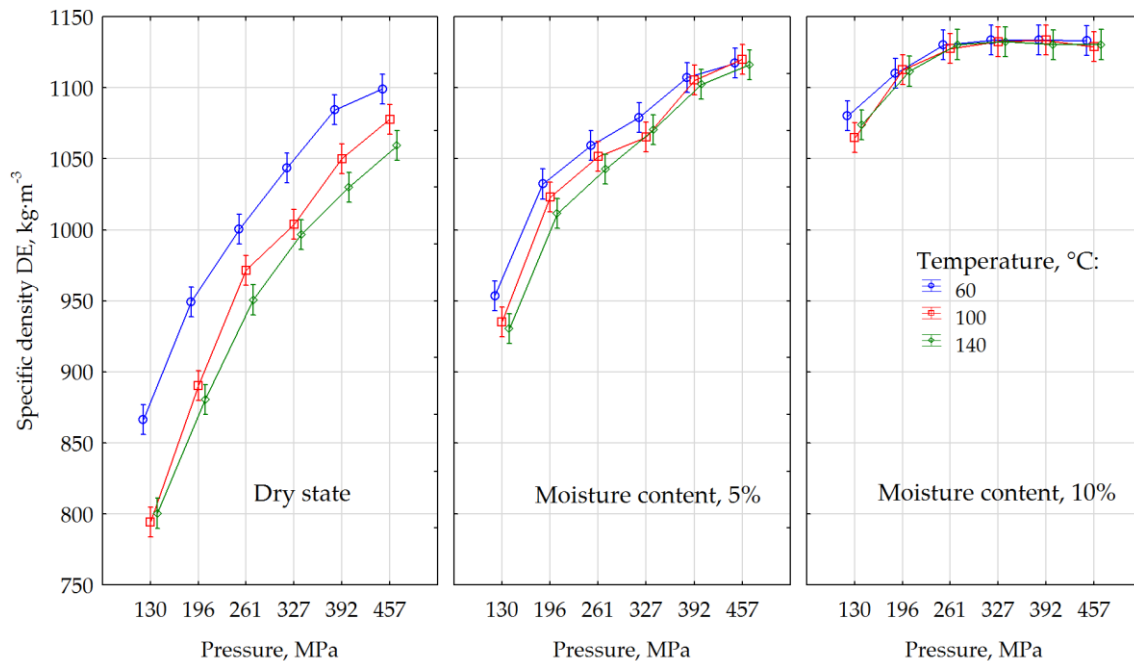
**Figure 7.** Pellet mechanical durability (*DU*) changes as a function of the raw material drying temperature and densification pressure: (a) raw material in dry state, (b) 5% moisture content, (c) 10% moisture content.

In this work, ANOVA analysis was carried out, and the normality of the decomposition was checked using the Shapiro–Wilk test. It was found that the distribution was normal for all cases. Then, the assumption of the equality of variance was also examined using the Brown–Forsythe test. For all cases, this equality was met. Then, ANOVA and post-hoc analysis (Scheffé’s test) were carried out to determine whether there were statistically significant differences between the groups.

Figure 8 shows the results of the three-factor ANOVA examining the effects of the drying temperature, moisture content and pressure on the specific density. The graphs show the compaction profiles derived from the mean values together with the standard deviations.

Post-hoc analyses have shown that for the dry material, there are significant differences between the specific density values of the pellets produced from the material dried at 60 °C and those produced from the material dried at 100 and 140 °C. These differences exist for each of the examined pressures with the exception of 261.6 MPa and 457.8 MPa, for which the change in the drying temperature from 60 and 100 °C does not have a significant effect on the specific density of compacts. For each tested pressure, the differences in the specific density values between the pellets obtained from the materials

dried at 100 and 140 °C were insignificant. As mentioned above, the specific densities of the pellets decreased as the drying temperature increased from 60 to 100 °C. This means that the drying of raw material at 100 and 140 °C leads to a decrease in the densification ability of the raw material.

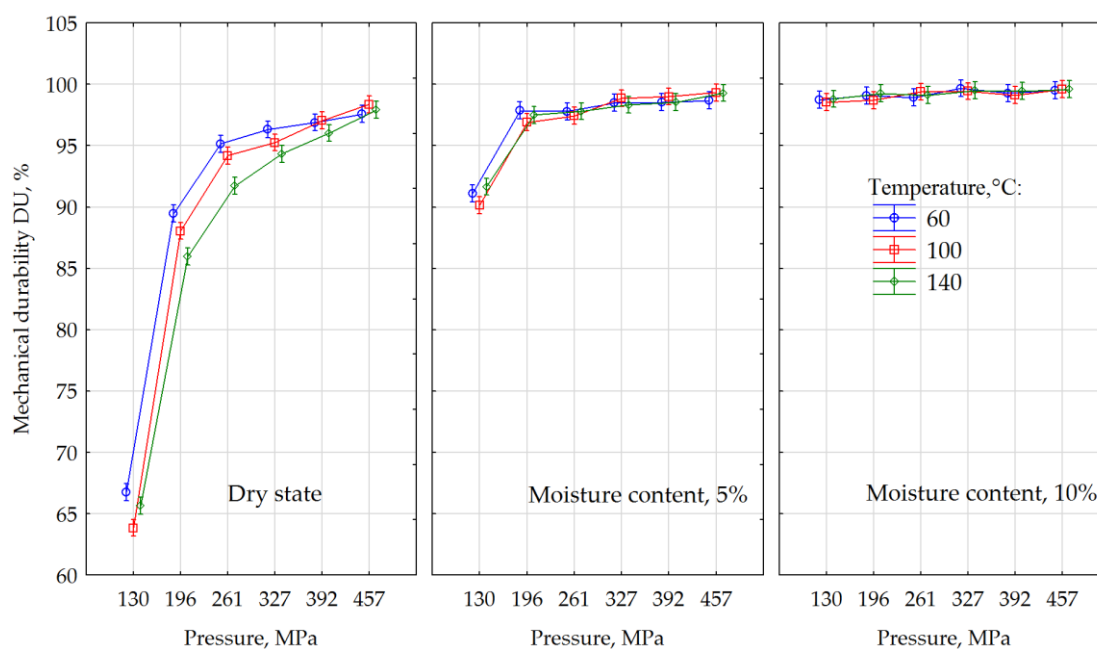


**Figure 8.** Changes in the specific density ( $DE$ ) of the pellets as a function of the compaction process parameters.

For the materials moistened to 5% and 10% moisture content, the effect of the drying temperature on the obtained  $DE$  values was insignificant in the entire examined pressure range. This means that moistening the material even to the level of only 5% eliminates the influence of the drying temperature and at the same time allows us to obtain pellets with higher  $DE$  compared to those obtained from the material in the dry state.

A similar analysis was performed for  $DU$ . Figure 9 shows the result of the three-factor ANOVA examining the effects of the drying temperature, moisture content and pressure on the  $DU$  values. The graphs show the compactibility profiles derived from the average values together with the standard deviations.

Post-hoc analyses showed that, for the dry material, there are significant differences between the  $DU$  values of the pellets produced from the material dried at 60 °C and those produced from the material dried at 100 and 140 °C. These differences are observed only for pressures lower than 261.6 MPa, while for higher pressures, the differences are insignificant. At a pressure of 130.8 MPa, the significant difference occurs between the drying temperature of 60 and 100 °C, at 196.2 and 261.6 MPa a significant difference is observed between the samples obtained using drying temperatures of 60 and 140 °C. The differences in mechanical durability between the pellets made of the materials dried at 100 and 140 °C were insignificant in the entire pressure range. Similar to the specific density results,  $DU$  decreased with increasing drying temperature for the dry materials, while for the materials moistened to the 5% and 10% moisture content, the influence of the drying temperature on the obtained mechanical durability values was insignificant in the entire range of tested pressures.



**Figure 9.** Changes in the mechanical durability (*DU*) of the pellets for different compaction process parameters.

Thus, the conducted tests show that the drying temperature significantly influences the obtained values of the specific density of the pellets produced from the material in a dry state in the whole pressure range, while for *DU*, the changes were significant only in the 130.8–261.6 MPa range. Significant changes occur between the drying temperature of 60 °C on the one hand, and the drying temperature of 100 and 140 °C on the other hand, while the differences between 100 and 140 °C are not significant. An increase in the moisture content to 5% and 10% eliminates the influence of the drying temperature and leads to an increase in the values of the tested quality parameters. What is remarkable is that this range of moisture content is optimal for the pelletization process and meets the quality standard requirements [40,82]. In order to obtain high parameters of *DE* and *DU*, it is not necessary use the maximum tested pressure values and that these pressure are lower than the pressure occurring in the real process of pelletization up to 750 MPa [83].

#### 4. Discussion

Higher values of the quality parameters obtained for the pellets produced from the material dried at 60 °C compared to those produced from the materials dried at 100 and 140 °C (compacted in dry state) may be explained by the fact that, at this temperature, only water is removed from the material and other components such as terpenes, resins, and essential oils (generally called volatile substances) remain in the material, while when the drying temperature exceeds 100 °C, these substances are also gradually released. This phenomenon is confirmed by studies performed by Stahl et al. [84] comparing the drying process on industrial systems. They have shown that the highest terpene emissions occur in dryers with high drying temperatures (above 100 °C) and long material residence times in the dryer. The type of dryer also affects the emission of terpenes – steam dryers cause less emission compared to rotary drum dryers. Moreover, the Englund and Nussbaum research [85] confirms that the emission of terpenes at a drying temperature of 60 °C is lower compared to emission at 110 °C.

Generally, these substances act as binders, and also play the role of binder in the material dried at 60 °C. For the pellets produced from the biomass dried at 100 and 140 °C, the removal of the binder substances from the material results in a decrease in the *DE* and *DU* values. For the material with 5% and 10% moisture content, the water contained in the material plays the role of a binder and its higher agglomeration capacity compared to the volatile substances leads to the better connection between

the material particles, resulting in an increase in the *DE* and *DU* values. At the same time, water eliminates the weaker impact of volatile substances, as evident from the absence of the significant effect of the drying temperature on the obtained values of *DE* and *DU* at this raw material moisture content. The increase in the water content in the raw material to the level of 10% results in an increase in the number of bonds between the particles (so-called interparticle bridges) [3,86], which further increases the *DE* and *DU* values. This is probably caused by the increase in the moisture content of the raw material. This has a significant effect on the plasticization of lignin. This is probably the main reason for the mentioned increase in the number of bonds between the particles.

A different course of *DE* and *DU* changes was observed by Filbak et al. [83] during a study on the influence of drying methods for Scots pine raw material on pellets' *DE* and *DU*. Pellets made of raw material dried at 75 °C had a higher *DU* value compared to those made of material dried at 450 °C. An opposite relationship was observed for *DE*. In these studies, the pellets were made on an industrial line where, apart from moisture content, the main factor influencing the agglomeration process is temperature changes occurring during the process [19].

Similar to most lignocellulose materials, pine wood has a spatial porous structure. Therefore, the specific density of dry pine wood is approximately 490 kg·m<sup>-3</sup>, while its absolute density is approximately 1470 kg·m<sup>-3</sup> (lignocellulose material without any pores) and its bulk density is approximately 180 kg·m<sup>-3</sup> [3]. During compaction, the applied pressure is responsible for reducing the external (between material particles) and internal (inside material particles) pores. Drying at 60 °C removes the water from the inner pores of the material, while an increase in the temperature to 100 and 140 °C also leads to the removal of the volatile components, increasing the volume of the pores. The pressure reduces the volume of the pores (both external and internal), and the degree of pore reduction is greater for the material dried at 60 °C. Upon the moistening of the material to 5% water content, the water infiltrates the wood and the material becomes plasticized, making a lower pressure sufficient for compacting it. Then, a further moisturization leads to an increase in the plasticity and a further increase in the density at lower pressures. The maximum obtained specific density values oscillate at 1130 kg·m<sup>-3</sup>. A value of *DE* equal to the absolute density (1470 kg·m<sup>-3</sup>) cannot be achieved due to the expansion of the material when the pressure stops. The value of this parameter depends on the elasticity of the material and the amount and strength of the joints between the particles formed during the compaction. In this case, the best results were achieved by moisturizing the material to the 10% water content and compacting at the pressure 261.6 MPa, and a further increase in the pressure did not increase the achieved *DE*. For *DU*, 130.8 MPa and 10% moisture content is sufficient to obtain the maximum value. Although these studies have shown that the influence of the drying temperature on the compaction process is important for the material in a dry state, and while moistening to 5% and 10% water content eliminates this influence, it is an open question as to whether this dependence is also found for other materials used in solid biofuel production. While a classic material that can be easily agglomerated was tested in this work, materials that are difficult to compact, e.g., straw, miscanthus, and reed canary grass, may be more sensitive to the influence of the drying temperature. Our investigations showed that the drying temperature changes the properties of the raw material. This means that these differences can affect other stages of raw material processing, such as grinding. The fact that the moisture content reduces this effect during pelletization is important because it means that, for the compaction stage, it is possible to use raw material dried over a wide temperature range without affecting the main quality parameters. If further investigations confirm that the drying temperature affects the grinding process, then the drying temperature can be selected so that grinding can be carried out with minimum effort and without fear of losing the quality of the final product. The drying temperature may affect the energy parameters of the raw material. High temperature causes a loss of extractives, which may decrease the pellets' combustion heat and change the combustion process [51,85]. The study of these relationships will be taken into account in further research.

## 5. Conclusions

The aim of this study was to determine the effect of the drying temperature of the raw material on the compacting process of pine biomass. This effect was determined for three different material moisture contents and six different compaction pressures. Based on the presented data, the following key results were obtained:

- The drying temperature significantly affects the specific density of the compacts over the entire pressure range when the compacted material is in the dry state;
- For *DU*, the influence of the drying temperature is significant in the pressure range of 130.8–261.6 MPa for the materials in the dry state;
- An increase in the drying temperature leads to a decrease in the *DE* and *DU* values (dry material);
- Significant differences are observed between the material dried at 60 °C and the materials dried at 100 and 140 °C, while the differences between the materials treated at the drying temperatures of 100 and 140 °C are not significant;
- An increase in the moisture content of the raw material to 5% eliminates the influence of the drying temperature on the obtained values of *DE* and *DU* and increases the obtained *DE* and *DU* values relative to those of the dry material;
- An increase in the moisture content to 10% results in a further increase in *DE* and *DU*;
- Assumed threshold values of specific density and mechanical durability were obtained for each drying temperature and moisture content level. As the moisture content of the raw material increases, the pressure necessary to obtain the threshold *DE* and *DU* decreases.

These results reveal an effect that has not been described in previous reports in the literature. Further research on the effects of the drying temperature on the other biomass raw materials and the stages of the biomass compaction process should be carried out to improve the understanding of this topic.

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## References

1. Popper, Z.A.; Michel, G.; Hervé, C.; Domozych, D.S.; Willats, W.G.T.; Tuohy, M.G.; Kloareg, B.; Stengel, D.B. Evolution and Diversity of Plant Cell Walls: From Algae to Flowering Plants. *Annu. Rev. Plant Biol.* **2011**, *62*, 567–590. [[CrossRef](#)] [[PubMed](#)]
2. Knox, J.P. Revealing the structural and functional diversity of plant cell walls. *Curr. Opin. Plant Biol.* **2008**, *11*, 308–313. [[CrossRef](#)] [[PubMed](#)]
3. Wróbel, M. *Zagęszczalność i Kompaktowalność Biomasy Lignocelulozowej*; PTIR: Kraków, Poland, 2019; ISBN 978-83-64377-35-8.
4. Welker, C.; Balasubramanian, V.; Petti, C.; Rai, K.; DeBolt, S.; Mendu, V.; Welker, C.M.; Balasubramanian, V.K.; Petti, C.; Rai, K.M.; et al. Engineering Plant Biomass Lignin Content and Composition for Biofuels and Bioproducts. *Energies* **2015**, *8*, 7654–7676. [[CrossRef](#)]
5. 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.* **2009**, *48*, 3713–3729. [[CrossRef](#)]
6. Zethræus, B. The Bioenergy System Planners Handbook—BISYPLAN Chapter 04. Available online: <http://bisypplan.bioenarea.eu/html-files-en/02-02.html> (accessed on 12 December 2018).
7. Demirbaş, A. Estimating of structural composition of wood and non-wood biomass samples. *Energy Sour.* **2005**, *27*, 761–767. [[CrossRef](#)]

8. Lamloom, S.H.; Savidge, R.A. A reassessment of carbon content in wood: Variation within and between 41 North American species. *Biomass Bioenergy* **2003**, *25*, 381–388. [[CrossRef](#)]
9. Fajín, J.L.C.; Cordeiro, M.N.D.S. Probing the efficiency of platinum nanotubes for the H<sub>2</sub> production by water gas shift reaction: A DFT study. *Appl. Catal. B Environ.* **2020**, *263*, 118301.
10. García-Moncada, N.; González-Castaño, M.; Ivanova, S.; Centeno, M.Á.; Romero-Sarria, F.; Odriozola, J.A. New concept for old reaction: Novel WGS catalyst design. *Appl. Catal. B Environ.* **2018**, *238*, 1–5. [[CrossRef](#)]
11. Chianese, S.; Fail, S.; Binder, M.; Rauch, R.; Hofbauer, H.; Molino, A.; Blasi, A.; Musmarra, D. Experimental investigations of hydrogen production from CO catalytic conversion of tar rich syngas by biomass gasification. *Catal. Today* **2016**, *277*, 182–191. [[CrossRef](#)]
12. Li, W.; Li, Q.; Chen, R.; Wu, Y.; Zhang, Y. Investigation of hydrogen production using wood pellets gasification with steam at high temperature over 800 °C to 1435 °C. *Int. J. Hydrogen Energy* **2014**, *39*, 5580–5588. [[CrossRef](#)]
13. Demirbas, A. Higher heating values of lignin types from wood and non-wood lignocellulosic biomasses. *Energy Sources Part A Recover. Util. Environ. Eff.* **2017**, *39*, 592–598. [[CrossRef](#)]
14. Dziurzyński, A. *Zależności Między Wybranymi Właściwościami Fizycznymi Drewna Sosny i Buki a Zawartością jego Strukturalnych Składników Chemicznych*; Wydawnictwo Akademii Rolniczej im. Augusta Cieszkowskiego: Poznań, Poland, 2003.
15. Kaliyan, N.; Vance Morey, R. Factors affecting strength and durability of densified biomass products. *Biomass Bioenergy* **2009**, *33*, 337–359. [[CrossRef](#)]
16. Li, W.; Amos, K.; Li, M.; Pu, Y.; Debolt, S.; Ragauskas, A.J.; Shi, J. Fractionation and characterization of lignin streams from unique high-lignin content endocarp feedstocks. *Biotechnol. Biofuels* **2018**, *11*, 304. [[CrossRef](#)] [[PubMed](#)]
17. Demirbaş, A. Relationships between heating value and lignin, fixed carbon, and volatile material contents of shells from biomass products. *Energy Sources* **2003**, *25*, 629–635. [[CrossRef](#)]
18. White, R.H. Effect of lignin content and extractives on the higher heating value of wood. *Wood Fiber Sci.* **1987**, *19*, 446–452.
19. Križan, P. *The Densification Process of Wood Waste*; De Gruyter Open Ltd.: Warsaw, Poland; Berlin, Germany, 2015; ISBN 978-3-11-044001-0.
20. Robbins, M.P.; Evans, G.; Valentine, J.; Donnison, I.S.; Allison, G.G. New opportunities for the exploitation of energy crops by thermochemical conversion in northern Europe and the UK. *Prog. Energy Combust. Sci.* **2012**, *38*, 138–155. [[CrossRef](#)]
21. Bajpai, P. *Pretreatment of Lignocellulosic Biomass for Biofuel Production*; Springer: Singapore, 2016; ISBN 978-981-10-0686-9.
22. Mendu, V.; Harman-Ware, A.E.; Crocker, M.; Jae, J.; Stork, J.; Morton, S.; Placido, A.; Huber, G.; Debolt, S. Identification and thermochemical analysis of high-lignin feedstocks for biofuel and biochemical production. *Biotechnol. Biofuels* **2011**, *4*, 43. [[CrossRef](#)]
23. van Dam, J.E.G.; van den Oever, M.J.A.; Teunissen, W.; Keijsers, E.R.P.; Peralta, A.G. Process for production of high density/high performance binderless boards from whole coconut husk: Part 1: Lignin as intrinsic thermosetting binder resin. *Ind. Crops Prod.* **2004**, *19*, 207–216. [[CrossRef](#)]
24. Niemczyk, M.; Kaliszewski, A.; Jewiarz, M.; Wróbel, M.; Mudryk, K. Productivity and biomass characteristics of selected poplar (*Populus* spp.) cultivars under the climatic conditions of northern Poland. *Biomass Bioenergy* **2018**, *111*, 46–51. [[CrossRef](#)]
25. Bryś, A.; Bryś, J.; Głowacki, S.; Tulej, W.; Zajkowski, P.; Sojak, M. *Analysis of Potential Related to Grass-Derived Biomass for Energetic Purposes*; Springer: Cham, Switzerland, 2018; pp. 443–449.
26. Knapczyk, A.; Francik, S.; Wójcik, A.; Bednarz, G. Influence of Storing *Miscanthus* × *Gigantopus* on Its Mechanical and Energetic Properties. In *Renewable Energy Sources: Engineering, Technology, Innovation*; Springer: Cham, Switzerland, 2018; pp. 651–660.
27. Wróbel, M.; Mudryk, K.; Jewiarz, M.; Głowacki, S.; Tulej, W. Characterization of Selected Plant Species in Terms of Energetic Use. In *Proceedings of the Renewable Energy Sources: Engineering, Technology, Innovation. Springer Proceedings in Energy ICORES 2017*; Mudryk, K., Werle, S., Eds.; Springer Proceedings in Energy: Cham, Switzerland, 2018; pp. 671–681.
28. Stelte, W.; Sanadi, A.R.; Shang, L.; Holm, J.K.; Ahrenfeldt, J.; Henriksen, U.B. Recent developments in biomass pelletization—A review. *BioResources* **2012**, *7*, 4451–4490.

29. Bilbao, R.; Lezaun, J.; Abanades, J.C. Fluidization velocities of sand/straw binary mixtures. *Powder Technol.* **1987**, *52*, 1–6. [[CrossRef](#)]
30. EN ISO 17225-1:2014. *Solid Biofuels—Fuel Specifications and Classes—Part 1: General Requirements*; International Organization for Standardization: Geneva, Switzerland, 2014.
31. Brzychczyk, B.; Hebda, T.; Giełżecki, J. Physical and Chemical Properties of Pellets Produced from the Stabilized Fraction of Municipal Sewage Sludge. In *Renewable Energy Sources: Engineering, Technology, Innovation*; Springer: Cham, Switzerland, 2018; pp. 613–622.
32. Brzychczyk, B.; Hebda, T.; Giełżecki, J. Energy Characteristics of Compacted Biofuel with Stabilized Fraction of Municipal Waste. In *Renewable Energy Sources: Engineering, Technology, Innovation*; Springer: Cham, Switzerland, 2018; pp. 451–462.
33. Hebda, T.; Brzychczyk, B.; Francik, S.; Pedryc, N. Evaluation of suitability of hazelnut shell energy for production of biofuels. In *Proceedings of the Engineering for Rural Development, Jelgava, Latvia, 23–25 May 2018*; Volume 17, pp. 1860–1865.
34. Knapczyk, A.; Francik, S.; Frączek, J.; Slipek, Z. Analysis of research trends in production of solid biofuels. In *Proceedings of the Engineering for Rural Development, Jelgava, Latvia, 22–24 May 2019*; Volume 18, pp. 1503–1509.
35. Karbowniczak, A.; Hamerska, J.; Wróbel, M.; Jewiarz, M.; Nęcka, K. Evaluation of Selected Species of Woody Plants in Terms of Suitability for Energy Production. In *Renewable Energy Sources: Engineering, Technology, Innovation. Springer Proceedings in Energy ICORES 2017*; Mudryk, K., Werle, S., Eds.; Springer Proceedings in Energy: Cham, Switzerland, 2018; pp. 735–742.
36. Telmo, C.; Lousada, J. Heating values of wood pellets from different species. *Biomass Bioenergy* **2011**, *35*, 2634–2639. [[CrossRef](#)]
37. Viana, H.; Vega-Nieva, D.J.; Ortiz Torres, L.; Lousada, J.; Aranha, J. Fuel characterization and biomass combustion properties of selected native woody shrub species from central Portugal and NW Spain. *Fuel* **2012**, *102*, 737–745. [[CrossRef](#)]
38. Luca, C.; Pilu, R.; Tambone, F.; Scaglia, B.; Adani, F. New energy crop giant cane (*Arundo donax* L.) can substitute traditional energy crops increasing biogas yield and reducing costs. *Bioresour. Technol.* **2015**, *191*, 197–204. [[PubMed](#)]
39. Zegada-Lizarazu, W.; Monti, A. Energy crops in rotation. A review. *Biomass Bioenergy* **2011**, *35*, 12–25. [[CrossRef](#)]
40. EN ISO 17225-2:2014. *Solid Biofuels—Fuel Specifications and Classes—Part 2: Graded Wood Pellets*; International Organization for Standardization: Geneva, Switzerland, 2014.
41. Sitzmann, W.; Buschhart, A. *Pelleting as Prerequisite for the Energetic Utilization of Byproducts of the Wood-Processing Industry*; Amandus Kahl GmbH & Co.: Oslo, Norway, 2009.
42. Kaliyan, N.; Morey, R.V. Natural binders and solid bridge type binding mechanisms in briquettes and pellets made from corn stover and switchgrass. *Bioresour. Technol.* **2010**, *101*, 1082–1090. [[CrossRef](#)]
43. Dyjakon, A.; Noszczyk, T. The Influence of Freezing Temperature Storage on the Mechanical Durability of Commercial Pellets from Biomass. *Energies* **2019**, *12*, 2627. [[CrossRef](#)]
44. Wójcik, A.; Frączek, J.; Wota, A.K. The methodical aspects of the friction modeling of plant granular materials. *Powder Technol.* **2019**, *344*, 504–513. [[CrossRef](#)]
45. Wójcik, A.; Frączek, J.; Niemczewska-Wójcik, M. The relationship between static and kinetic friction for plant granular materials. *Powder Technol.* **2019**, *361*, 739–747. [[CrossRef](#)]
46. Nielsen, N.P.K.; Gardner, D.J.; Poulsen, T.; Felby, C. Importance of temperature, moisture content, and species for the conversion process of wood residues into fuel pellets. *Wood Fiber Sci.* **2009**, *41*, 414–425.
47. Arshadi, M.; Gref, R.; Geladi, P.; Dahlqvist, S.A.; Lestander, T. The influence of raw material characteristics on the industrial pelletizing process and pellet quality. *Fuel Process. Technol.* **2008**, *89*, 1442–1447. [[CrossRef](#)]
48. Amos, W.A. *Report on Biomass Drying Technology*; National Renewable Energy Laboratory: Golden, CO, USA, 1999.
49. Fagernäs, L.; Brammer, J.; Wilén, C.; Lauer, M.; Verhoeff, F. Drying of biomass for second generation synfuel production. *Biomass Bioenergy* **2010**, *34*, 1267–1277. [[CrossRef](#)]
50. Gebreegziabher, T.; Oyedun, A.O.; Hui, C.W. Optimum biomass drying for combustion—A modeling approach. *Energy* **2013**, *53*, 67–73. [[CrossRef](#)]

51. Ståhl, M.; Granström, K.; Berghel, J.; Renström, R. Industrial processes for biomass drying and their effects on the quality properties of wood pellets. *Biomass Bioenergy* **2004**, *27*, 621–628. [[CrossRef](#)]
52. Francik, S.; Łapczyńska-Kordon, B.; Francik, R.; Wójcik, A. Modeling and Simulation of Biomass Drying Using Artificial Neural Networks. In *Renewable Energy Sources: Engineering, Technology, Innovation*; Springer: Cham, Switzerland, 2018; pp. 571–581.
53. Głowacki, S.; Tulej, W.; Jaros, M.; Sojak, M.; Bryś, A.; Kędziora, R. Kinetics of Drying Silver Birch (*Betula pendula* Roth) as an Alternative Source of Energy. In *Renewable Energy Sources: Engineering, Technology, Innovation*; Springer: Cham, Switzerland, 2018; pp. 433–442.
54. Kumar, A.; Jones, D.; Hanna, M. Thermochemical Biomass Gasification: A Review of the Current Status of the Technology. *Energies* **2009**, *2*, 556–581. [[CrossRef](#)]
55. Jahirul, M.I.; Rasul, M.G.; Chowdhury, A.A.; Ashwath, N. Biofuels production through biomass pyrolysis—A technological review. *Energies* **2012**, *5*, 4952–5001. [[CrossRef](#)]
56. Peng, J.H.; Bi, X.T.; Sokhansanj, S.; Lim, C.J. Torrefaction and densification of different species of softwood residues. *Fuel* **2013**, *111*, 411–421. [[CrossRef](#)]
57. Li, H.; Liu, X.; Legros, R.; Bi, X.T.; Jim Lim, C.; Sokhansanj, S. Pelletization of torrefied sawdust and properties of torrefied pellets. *Appl. Energy* **2012**, *93*, 680–685. [[CrossRef](#)]
58. Wróbel, M.; Hamerska, J.; Jewiarz, M.; Mudryk, K.; Niemczyk, M. Influence of Parameters of the Torrefaction Process on the Selected Parameters of Torrefied Woody Biomass. In *Renewable Energy Sources: Engineering, Technology, Innovation. Springer Proceedings in Energy ICORES 2017*; Mudryk, K., Werle, S., Eds.; Springer Proceedings in Energy: Cham, Switzerland, 2018; pp. 691–700.
59. Chandler, C.; Cheney, P.; Thomas, P.; Traband, L.D.W. *Fire in Forestry—Vol.I. Forest Fire Behavior and Effects*; John Wiley & Sons: New York, NY, USA, 1983.
60. Chen, D.; Gao, A.; Cen, K.; Zhang, J.; Cao, X.; Ma, Z. Investigation of biomass torrefaction based on three major components: Hemicellulose, cellulose, and lignin. *Energy Convers. Manag.* **2018**, *169*, 228–237. [[CrossRef](#)]
61. Chen, W.H.; Kuo, P.C. Torrefaction and co-torrefaction characterization of hemicellulose, cellulose and lignin as well as torrefaction of some basic constituents in biomass. *Energy* **2011**, *36*, 803–811. [[CrossRef](#)]
62. Mierzwa-Hersztek, M.; Gondek, K.; Jewiarz, M.; Dziedzic, K. Assessment of energy parameters of biomass and biochars, leachability of heavy metals and phytotoxicity of their ashes. *J. Mater. Cycles Waste Manag.* **2019**, *21*, 786–800. [[CrossRef](#)]
63. Jankowska, A.; Kozakiewicz, P. Determination of fibre saturation point of selected tropical wood species using different methods. *Drewno* **2016**, *59*, 89–97.
64. Gilbert, P.; Ryu, C.; Sharifi, V.; Swithenbank, J. Effect of process parameters on pelletisation of herbaceous crops. *Fuel* **2009**, *88*, 1491–1497. [[CrossRef](#)]
65. Lam, P.S.; Sokhansanj, S.; Bi, X.; Lim, C.J.; Melin, S. Energy input and quality of pellets made from steam-exploded douglas fir (*Pseudotsuga menziesii*). *Energy Fuels* **2011**, *25*, 1521–1528. [[CrossRef](#)]
66. 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](#)]
67. Anglès, M.N.; Ferrando, F.; Farriol, X.; Salvadó, J. Suitability of steam exploded residual softwood for the production of binderless panels. Effect of the pre-treatment severity and lignin addition. *Biomass Bioenergy* **2001**, *21*, 211–224. [[CrossRef](#)]
68. Biswas, A.K.; Yang, W.; Blasiak, W. Steam pretreatment of *Salix* to upgrade biomass fuel for wood pellet production. *Fuel Process. Technol.* **2011**, *92*, 1711–1717. [[CrossRef](#)]
69. Carone, M.T.; Pantaleo, A.; Pellerano, A. Influence of process parameters and biomass characteristics on the durability of pellets from the pruning residues of *Olea europaea* L. *Biomass Bioenergy* **2011**, *35*, 402–410. [[CrossRef](#)]
70. Kirsten, C.; Lenz, V.; Schröder, H.W.; Repke, J.U. Hay pellets—The influence of particle size reduction on their physical-mechanical quality and energy demand during production. *Fuel Process. Technol.* **2016**, *148*, 163–174. [[CrossRef](#)]
71. Tumuluru, J. Effect of Moisture Content and Hammer Mill Screen Size on the Briquetting Characteristics of Woody and Herbaceous Biomass. *KONA Powder Part.* **2019**. [[CrossRef](#)]
72. Payne, J.D. Troubleshooting the Pelletizing Process. In *Feed Technology Technical Report Series*; American Soybean Association: Singapore, 2006.



73. Muramatsu, K.; Massuquetto, A.; Dahlke, F.; Maiorka, A. Factors that Affect Pellet Quality: A Review. *J. Agric. Sci. Technol. A* **2015**, *5*, 717–722. [[CrossRef](#)]
74. EN ISO 18125:2017. *Solid Biofuels—Determination of Calorific Value*; International Organization for Standardization: Geneva, Switzerland, 2017.
75. EN ISO 18122:2015. *Solid Biofuels—Determination of Ash Content*; International Organization for Standardization: Geneva, Switzerland, 2015.
76. EN ISO 18123:2015. *Solid Biofuels—Determination of the Content of Volatile Matter*; International Organization for Standardization: Geneva, Switzerland, 2015.
77. Stelte, W.; Holm, J.K.; Sanadi, A.R.; Barsberg, S.; Ahrenfeldt, J.; Henriksen, U.B. A study of bonding and failure mechanisms in fuel pellets from different biomass resources. *Biomass Bioenergy* **2011**, *35*, 910–918. [[CrossRef](#)]
78. Stelte, W.; Holm, J.K.; Sanadi, A.R.; Barsberg, S.; Ahrenfeldt, J.; Henriksen, U.B. Fuel pellets from biomass: The importance of the pelletizing pressure and its dependency on the processing conditions. *Fuel* **2011**, *90*, 3285–3290. [[CrossRef](#)]
79. Wróbel, M. Assessment of Agglomeration Properties of Biomass—Preliminary Study. In *Renewable Energy Sources: Engineering, Technology, Innovation. Springer Proceedings in Energy ICORES 2018*; Wróbel, M., Jewiarz, M., Szłek, A., Eds.; Springer Proceedings in Energy: Cham, Switzerland, 2020; pp. 411–418.
80. EN ISO 18847:2016. *Solid Biofuels—Determination of Particle Density of Pellets and Briquettes*; International Organization for Standardization: Geneva, Switzerland, 2016.
81. EN ISO 17831-1:2015. *Solid Biofuels—Determination of Mechanical Durability of Pellets and Briquettes—Part 1: Pellets*; International Organization for Standardization: Geneva, Switzerland, 2015.
82. EN ISO 17225-6:2014. *Solid Biofuels—Fuel Specifications and Classes—Part 6: Graded Non-Woody Pellets*; International Organization for Standardization: Geneva, Switzerland.
83. Filbakk, T.; Skjevraak, G.; Høibø, O.; Dibdiakova, J.; Jirjis, R. The influence of storage and drying methods for Scots pine raw material on mechanical pellet properties and production parameters. *Fuel Process. Technol.* **2011**, *92*, 871–878. [[CrossRef](#)]
84. Ståhl, M.; Berghel, J. Energy efficient pilot-scale production of wood fuel pellets made from a raw material mix including sawdust and rapeseed cake. *Biomass Bioenergy* **2011**, *35*, 4849–4854. [[CrossRef](#)]
85. Englund, F.; Nussbaum, R.M. Monoterpenes in Scots pine and Norway spruce and their emission during kiln drying. *Holzforschung* **2000**, *54*, 449–456. [[CrossRef](#)]
86. Pietsch, W. *Agglomeration Processes. Phenomena, Technologies, Equipment*; Wiley-VCH Verlag GmbH.: Weinheim, Germany, 2002; ISBN 3-527-30369-3.



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