Evaluation of the Physical, Mechanical, and Calorific Properties of Briquettes with or without a Hollow Made of Wheat (Triticum aestivum L.) Straw Waste

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Abstract: Large amounts lignocellulosic biomass in the form of straw is leftover after wheat harvesting that could be utilized for beneficial purposes. The latter has led to the emergence of new technologies to make use of this resource. One such technology currently in use turns wheat straw into briquettes. In the present study, we have prepared and evaluated the physical, mechanical and calorific properties of two types of briquettes made of wheat straw. The two types of briquettes prepared were (i) hollow briquettes and (ii) solid briquettes. The densities of these briquettes obtained on a mechanical device with a crank mechanism were 1.169 kg/m$^3$, irrespective of whether the briquettes were hollow or solid. The briquette densities are consistent with European standards. The calorific value of wheat straw was 17.69 MJ/kg. Although the calorific value was somewhat lower than the beech wood briquettes (18.38 MJ/kg), it is adequate for their combustion in both stoves and thermal power plants. The ash content of wheat straw was 9.1% (~10-fold higher than that of beech wood). The briquettes showed a compressive mechanical strength of 1.15–2.17 N/mm$^2$ and splitting mechanical strength of 0.17–0.39 N/mm$^2$ suggesting that the straw briquettes were well compacted and can be stable during transport and/or other manipulations. In conclusion, wheat straw briquettes have similar physical, mechanical and calorific properties to those prepared from beech wood biomass and are a viable solution to replace beech wood briquettes with similar efficiency.

Keywords: briquette; wheat straw; lignocellulosic biomass; calorific value; ash content; compressive strength; splitting strength

1. Introduction

Plant biomass is a source of renewable energy, along with solar, wind, and geothermal energy. In agriculture, vegetable remains in the form of straw, corn stalks, sunflower, flax, castor and hemp, husks of sunflower seeds, rice seeds, the kernels of various fruits, etc., can become an additional energy source for households [1]. Among these cheap leftovers, straw represents one of the most important quantitative resources. Wheat straw usually contains about 12–18% moisture at the time of harvesting, so it does not require drying before briquetting, as the residual 12–15% water evaporates during burning. As with any lignocellulosic resource, straw dry matter consists of carbon (45.6%), hydrogen (5.8%), and oxygen (42.4%), as well as small amounts of oxides of nitrogen, sulfur, alkali silicon, chloride, and others (6.2%) that are usually found in the ash [2]. The ash content of straw can vary between 2 and 12%, with the average being 6–7%. Compared to this straw biomass, beech wood biomass (with bark) has 47.9% carbon, 6.2% hydrogen, 43.3% oxygen, and 2.6% ash [2]. Straw from crops that have been grown on sandy soils normally has the lowest ash content, while straw from lowland soils usually has the highest ash content. The calorific value of European beech biomass is usually 18.5 MJ/kg, and the ash content is lower than that of straw [2]. Globally, a huge amount of straw is produced [3]. The amount of heat obtained by burning straw as an alternative energy source [4,5] has a maximum of
22.2 MJ/m$^2$ of agricultural land optimal for the cultivation of straw [6]. The average grain wheat production in the European Union is 6.5 t/ha, which means that, if we take into account a grain/straw ratio of 1:0.8, straw production of 5.2 t/ha is obtained annually. Corn with 1:1.3, sunflower with 1:4.1, and rapeseed with 1:2.9 have better ratios between the agricultural product and lignocellulosic residues [7]. The calorific value of the remains from agricultural crops is different from one crop to another, with the best being wheat straw at 17.21 MJ/kg and energetic willow at 17.28 MJ/kg, compared to wood pellets at 18.11 MJ/kg. A bale of parallelepiped straw with dimensions of 1.2 $\times$ 1.2 $\times$ 2.5 m (with a mass of about 500 kg) will have a calorific value of 4.7 kWh/kg, which means a total amount of energy of 2 MWh and represents the equivalent of 200 l of oil or 300 kg of coal [7]. During the natural or artificial drying of straw in order to obtain briquettes with a moisture content of 8–10%, for every 10% decrease in the moisture content of the straw, an increase in calorific value of 2.16 MJ/kg (0.6 kWh/kg) can be obtained. The main advantages of using straw in combustion are the large quantities obtained per hectare, the year-to-year cyclic availability of renewable biomass [8,9], the easy ignition and rapid burning, as well as the fact that at harvest the moisture content is below 15% (it does not require additional drying).

Briquettes are combustible products obtained from compressed lignocellulosic material (CEN/TC 335:2004), with densities of over 850 kg/m$^3$. Typical values of 900–1000 kg/m$^3$ of briquette density can be found in the specialized literature [10], depending on the production process, with lower values for hydraulic briquetting machines and higher values for mechanical conveyor machines with a helical snack or connecting rod-crank mechanisms. The great advantage of these briquettes is that they have a high density beyond the density of the wood biomass from which they were obtained, and therefore, have a higher calorific density than the wood from which they came. Straw briquettes are among the cheapest because the raw material can be obtained at a low cost, following the wheat harvest. When making straw briquettes, high pressing forces must be used because a large amount of silicon in straw creates adhesion problems for the chips [3]. The Austrian standard ÖNORM M 7135:2000 [11] stipulates that the briquettes must have a minimum value of 850 kg/m$^3$, regardless of their nature, i.e., if they are made of wood or vegetable remains, and other authors [2] have specified a minimum value of 650 kg/m$^3$. The density of the briquettes differs depending on the type and power of the production machine, with values of about 800 kg/m$^3$ for hydraulic installations and 1100 kg/m$^3$ for presses with helical screws or connecting rod-crank mechanisms [12]. The briquettes can be obtained separately from straw or mixed with wood or inferior coal [13–16], with the lignocellulosic particles being native or torrefied [17–19]. Regarding the properties of briquettes, such as any lignocellulosic material, briquettes have physical, mechanical, chemical, and calorific properties. The physical–mechanical properties of briquettes are based on the dimensions of the briquettes, usually their diameter and length. The specifications of the standards in the field of briquettes and pellets [20] provide that the length should be about five times greater than the diameter. The moisture content of briquettes is limited to 10% by the British BioGen/UK standard [20] and 18% by ÖNORM M7135/Austria [11]. Unit density is limited above 525 kg/m$^3$ by the standard CTI-R04/5/Italy [21] and is a minimum of 1000 kg/m$^3$ in DIN 51731/Germany [22] and ÖNORM M7135/Austria [20]. The calorific value is limited to less than 16.7 MJ/kg by British BioGen/UK and to 18 MJ/kg by ÖNORM M7135/Austria. Ash content is limited above 1.5% by SS 18 71 20/Sweden, CTI-R 04/5, and DIN 51731, and at 6% by ÖNORM M7135/Austria [20]. Many authors [10,23–29] have analyzed the physical–mechanical and calorific properties of lignocellulose briquettes.

The main objective of this work was to compare briquettes with hollows and those without hollows, obtained from wheat straw. In this sense, the physical–mechanical and calorific properties of the briquettes, obtained with a connecting rod-crank mechanism with high densities, were analyzed. We sought to determine properties such as density, compression resistance, splitting resistance, moisture content, calorific value, energy density, and ash content. Additionally, a comparison was made of briquettes made of straw with the properties of briquettes obtained from the biomass of beech sawdust.
2. Materials and Methods

Three types of briquettes made of wheat straw, one type with hollows and two without hollows, were taken from a production company using briquetting installations with a connecting rod-crank mechanism.

All types of briquettes, with a length of 350 mm, were coated in groups of five, the ones without holes having two different average diameters of 59 and 73 mm, and the ones with holes with an average outer diameter of 71 mm. The samples without holes with a diameter of 59 mm were marked with T1, the samples without holes with a diameter of 73 mm were designated T2, and the samples with holes with an outer diameter of 71 mm and an inner diameter of 26 mm were marked as T3. After being removed from the foil, they were placed in a conditioning chamber for 48 h, until reaching an average moisture content of 10%. After that, the briquettes were cut with a circular saw with carbide teeth to a length of 60 mm. The samples were protected in polyethylene film in order to maintain the same moisture content during the laboratory tests.

2.1. Determination of Density

Each test piece, with a length of 60 mm and a moisture content of 10%, was weighed with an electronic balance type Kern, China. The density of the specimens obtained from whole specimens [30] was determined as the ratio between the mass and the volume of the specimens (EN 323:1993), using the following relationship (Equation (1)):

$$\rho_b = \frac{(4 \cdot m)}{(\pi \cdot d^2 \cdot l)} \cdot 10^6 \ [kg/m^3]$$

where $\rho_b$ is the density of the briquettes in kg/m$^3$; $m$ is the mass of the briquettes in g; $d$ is the diameter of the briquettes in mm and $l$ is the length of the samples in mm.

Twenty samples were used to determine the statistical parameters of the density values.

2.2. Determination of Resistance to Breaking by Compression

As a mechanical property, the briquettes’ resistance to breaking (crushing) by compression ensures the compaction of briquette stacks during their storage and transport [31–34]. Each specimen was inserted between two plates on a universal testing machine and submitted to the compression action with an advanced speed of 10 mm/min. This resistance was determined as the ratio between the maximum breaking force and the cross-sectional area of the briquette, with the following relationship for briquettes without hollows (Equation (2)):

$$\sigma_c = \frac{4 \cdot F_c}{(\pi \cdot D^2)} \ [N/mm^2]$$

where $\sigma_c$ is the compression resistance in N/mm$^2$; $F_c$ is the compression force at breaking of the specimen in N and D is the diameter of the briquette in mm.

In the case of briquettes with hollows, the different sections of the briquettes were taken into account by using the following relationship (Equation (3)):

$$\sigma_c = \frac{(4 \cdot F_c)}{(\pi \cdot (D_e^2 - d_i^2))} \ [N/mm^2]$$

where $D_e$ is the outer diameter in mm and $d_i$ is the inner diameter in mm.

Ten samples were tested in order to determine the statistical parameters of this resistance. The modeling used in the case of compression aimed to find the maximum height of the stack on a pallet (by the maximum number of briquettes), such that the briquettes at the base of the stack would not break, based on the effective resistance average obtained during the research. For this, a maximum triangular model of weight action was created on one of the briquettes disposed to the lower side, as can be seen in Figure 1.
Figure 1. The triangular model for determining the maximum height of the stack.

The arrangement of the briquettes in rows (Figure 1), with each upper row having one more briquette than the previous row, led to the use of the following mathematical calculation relationship:

\[ 1 + 2 + 3 + 4 + \ldots + n = \frac{n(n + 1)}{2} \]  

(4)

where \( n \) is the number of rows in the briquette stack.

Since the pressing force on the briquettes at the base of the stack must be lower than the average acceptable force, the following limiting relationship was found (Equation (5)):

\[ \frac{n(n + 1)}{2} \times m \times g \leq F_{\text{med}} \]

(5)

where \( F_{\text{med}} = \sigma_c \times A \); for briquettes without hollows \( A = \pi D^2/4 \); for briquettes with hollows \( A = \pi (D_e^2 - d_i^2)/4 \); \( m \) is the average mass of the samples extracted from the briquettes in g; \( g \) is the gravitational acceleration of 9.8 N/kg; \( \sigma_c \) is the average compressive strength in N/mm²; \( D \) is the average diameter of briquettes without hollows in mm; \( D_e \) is the outer diameter of the hollowed briquettes in mm; \( d_i \) is the inner diameter of the hollowed briquettes and \( l \) is the average length of the briquettes in mm.

From this relationship (5), the maximum admissible number of rows of briquettes on a stack was determined, extracting the positive value of the solution of the obtained equation of the second degree, such that the briquettes at the base of the stack would not break.

2.3. Determination of the Splitting Strength

For this purpose, a specific device was made in the form of a cutting knife with a blunt tip (rounding radius of 0.3 mm) with a thickness of 10 mm and a width of 12 cm, and a tip angle of 34 degrees. This was fixed in the universal tester and acted on the briquette until it was broken into two pieces. The splitting resistance was determined as the ratio between the maximum splitting force and the cross-sectional area of the briquette, with the following calculation relationship (Equation (6)):

\[ \sigma_s = \frac{4 \times F_s}{\pi D^2} \text{ [N/mm}^2\text{]} \]

(6)

where \( \sigma_s \) is the briquette’s resistance to splitting in N/mm²; \( F_s \) is the maximum splitting force in N; and \( D \) is the average diameter of the briquette, determined as the arithmetic mean of two perpendicular diameters made at the middle of the sample in mm.

Ten samples were tested to obtain the statistical parameters of this resistance.
2.4. Determination of Calorific Value

Calorific power is the main thermal property of briquettes, which gives information about the amount of heat obtained when burning them (EN ISO 17225-1:2014) [35]. To test the calorific power, an adiabatic calorimeter (XRY-1C oxygen bomb calorimeter, Shanghai Geological Ltd., Shanghai, China) was used, which determined the value by obtaining the temperature increase inside a calorimeter bomb between the moment of ignition of the sample and the end of its combustion, based on the calorimetric coefficient of the calorimetric installation. Eight samples were used [36,37], extracted from briquettes, with a mass of 0.8 ± 0.2 g, dried to 2–3% moisture content, and weighed with an accuracy of 3 decimals with an analytical balance. This degree of moisture was chosen because, during the test, 3 mL of distilled water was put inside the calorimetric bomb for the absorption of nitric acids [38], which increased the moisture content of the samples up to 4% until the moment of ignition (about 5–6 min), regardless of whether the samples were absolutely dry or had a moisture content of 2–3%. For the determination, the Renault–Phander program was used, by using the following general relationship (Equation (7)):

\[
HCV = \frac{(C_c(T_f - T_i) - Q_w - Q_c)}{m} \text{[MJ/kg]} \tag{7}
\]

where HCV is the higher calorific value in MJ/kg; \(C_c\) is the coefficient of the calorimeter in MJ/°C; \(T_f\) is the final temperature after complete combustion of the sample in °C; \(T_i\) is the initial temperature before igniting the sample in °C; \(Q_w\) is the amount of heat given off by the nickel wire in MJ; \(Q_c\) is the amount of heat given off by the cotton thread in MJ and \(m\) is the mass of the sample extracted from the straw briquette in kg.

The calorimeter software provided, at the end of the test, the high calorific value (HCV) and lower calorific value (LCV), as well as all the temperature values obtained during the tests and the total test duration for the samples of dry briquettes with a moisture content of 2–3%, which in the determination time became 4%.

To calculate the calorific value of the briquettes for absolutely dry briquettes, with 0% moisture content, the following relationship was used [8]:

\[
CV_0 = \frac{(100 \cdot CV_{Mc} - 2.44 \cdot Mc)}{100 - Mc} \text{[kJ/kg]} \tag{8}
\]

where \(CV_0\) is the calorific value for 0% moisture content in kJ/kg; \(CV_{Mc}\) is the calorific value for a certain moisture content in kJ/kg and \(Mc\) is the moisture content dry mass percentage.

In order to find the influence of moisture content on the calorific value, based on the previous relationship (8), a coordinate point (0; CV) was found, which was arranged on a graph with the moisture content of the briquettes on the horizontal axis and the calorific value on the vertical axis. On the same graph, the two previously determined points were arranged, respectively (4; LCV) and (4; HCV), thus obtaining two lines that intersected the horizontal axis of moisture content in two limiting points.

Caloric power per hectare took into account an average production of 5200 kg of straw/ha. The calorific value per hectare of wheat was calculated with the following formula (Equation (9)):

\[
CV_{ha} = CV \cdot m_{sha} \text{[MJ/ha]} \tag{9}
\]

where \(CV_{ha}\) is the calorific value related to the crop hectares in MJ/ha; \(CV\) is the calorific value of wheat straw in MJ/kg and \(m_{sha}\) is the mass of the resulting straw per hectare in kg/ha.

Modeling of the calorific power was undertaken depending on the carbon content of the biomass of the wheat straw mass [39].
2.5. Determination of Caloric Density

This determination gave the calorific value related to the volume unit of the briquettes by using the following calculation relationship (Equation (10)):

\[ Dc = CV \cdot \rho_b \text{ [MJ/m}^3\text{]} \]  

(10)

where \( Dc \) is the caloric density in MJ/m\(^3\); \( CV \) is the calorific value of straw briquettes in MJ/kg and \( \rho_b \) is the density of the briquettes for a moisture content of 10% in kg/m\(^3\).

2.6. Determination of Ash Content

The samples from the briquettes were crushed in a laboratory mill. The crushed material that passed through the 1 × 1 mm sieve (about 1–2 g, weighed with an analytical balance with self-calibration) was dried to an anhydrous state and arranged in a thin layer on a metal crucible made of nickel–chromium alloy, which is resistant at high temperatures. For drying, a Memmert oven (Schwabach, Germany) was used at a temperature of 105 °C for at least 1 h, until there were no more differences between two successive weighings. The actual determination test used an STS-type calcination furnace (Protherm, Ploiesti, Romania), heated to a temperature of 750 °C [40]. After calcination of the crushed material for 2 h, the ash crucibles were cooled in a desiccator and weighed again with a precision balance. The relationship for calculating the ash content was given by the ratio between the mass of the ash and the mass of the dry sample, taking into account the mass of the empty crucible, following (Equation (11)):

\[ Ac(\%) = \frac{(m_{c+ac} - m_c)}{(m_{c+s} - m_c)} \times 100 \% \]  

(11)

where \( Ac\% \) is the ash content in %; \( m_{c+ac} \) is the mass of the crucible plus the mass of calcined ash in g and \( m_{c+s} \) is the mass of the crucible plus the mass of the sample in g.

Ten replicate samples were tested in order to obtain significant statistical values of the percentage of ash content.

If the test material was not dry and had a certain moisture content, the relationship for determining the ash content changed significantly, as follows (Equation (12)):

\[ Ac(\%) = \frac{m_{ac}}{m_{MCs}} \times (100 + MC) \% \]  

(12)

where \( m_{ac} \) is the mass of calcined ash in g; \( m_{MCs} \) is the mass of the material sample at a certain moisture content in g and \( MC \) is the moisture content of the sample in g.

Equation (11) can also be expressed in kg/kg (\( Ac, \text{kg/kg} \)) if multiplication by 100 is omitted; that is, the amount of ash in kg resulting from one kilogram of straw can be obtained. Knowing the production of straw per hectare, it is possible to determine the ash content obtained per hectare of wheat plants, with the following relationship (Equation (13)):

\[ A(c,ha) = Ac,kg/kg \cdot m(s,ha) \text{ [kg/ha]} \]  

(13)

where \( A(c,ha) \) is the ash content in kg/ha; \( Ac,kg/kg \) is the ash content in kg/kg; and \( m(s,ha) \) is the mass of straw per surface unit of the wheat crop in kg/ha.

The previous relationship could also be determined by the amount of ash resulting from the controlled burning of stubble, even if this option is not recommended due to the danger of fires. Due to the large amounts of ash in straw [41,42], modeling of the calorific value was undertaken depending on the ash content. Thus, based on the laboratory data from the current research on wheat and beech, and also from other previous research [43,44], a database was drawn up, based on which a calorific graph was created for the ash content of the vegetable materials.
2.7. Determination of Proximate and Elemental Analysis

A proximate analysis usually considers the moisture content, volatile matter, fixed carbon, and ash content. When the moisture content is zero, the sum of the other three must be 100%. The elemental analysis evaluates the main chemical elements, such as carbon, hydrogen, oxygen, ash, etc. This test was determined by calcining samples at high temperatures of 900 °C, without oxygen supply, inside the calcination oven, for 5 min [45,46]. The calcination of the crushed material took place in a crucible with a lid, arranged inside the calcination installation on a special stand which did not allow the crucible to come into contact with the walls of the installation. At the end of the test, taking the amount of residue left in the crucible, the active carbon content was determined. The volatile content was determined by the difference between the fixed carbon and calcined ash content, using the following relationship (Equation (14)):

\[ VM + CA + Ac = 100 \% \]  

(14)

where VM is the volatile matter in %; CA is the active carbon in % and Ac is the ash content in %.

Ten valid samples were used to determine the statistical parameters of dispersion and the tendency of the test of volatiles, active carbon, and calcined ash.

2.8. Statistical Analysis

For each group of values, the median of the survey and the standard deviation was determined as the main statistical parameters of tendency and dispersion. The statistical analysis program Minitab 18 was also used, with empirical Cumulative Distribution Function (eCDF) statistical diagrams. The entire statistical analysis was carried out with a confidence interval of 95% and an alpha error of 0.05. Referencing values of briquette properties were used from some European standards, especially ÖNORM M7135 from Austria, which contains a specific section for briquettes.

3. Results

3.1. Dimensions and Expansion in Diameter after Pressing

The T1 type of briquettes without voids had a mean thickness of 59.96 mm, with a standard deviation of 1.07 mm. The inner diameter of the extrusion channel was 59.00 mm, and a value of 1.6% of the radial expansion of the diameter after exiting the briquetting machine was found. For the T2 type of briquettes without voids, an average thickness of 73.22 mm was obtained, with a standard deviation of 0.93 mm. Bearing in mind that the diameter of the extrusion channel was 72 mm, an expansion of the briquette diameter after extrusion of 1.69% was determined. A slight increase in the expansion in diameter of the briquettes with the larger diameter was observed, as stated by other authors [10]. In the T3 type of briquettes, an average outer diameter of 71.12 mm was obtained, with a standard deviation of 0.53 mm, and an average inner diameter of 26.88 mm with a standard deviation of 1.43 mm, but it was not possible to evaluate the thickness expansion of the briquette, because it spread over both the exterior and the interior. Moreover, the very large standard deviation of the inner diameter of the briquettes can be explained by the irregular radial expansion of the briquettes toward their hollow. Normally, the stresses on the outside and inside of the briquette should be equal, but due to the smaller surface of application of these stresses on the inside (a surface 2.6 times smaller), their influence will be greater. The effect is also amplified by the fact that the direction of these efforts is radial, with more prominent areas and less prominent areas. This larger and irregular expansion towards the inside is due to the appearance of additional forces towards the interior of the briquettes.

3.2. Density of Briquettes

The moisture content of the briquettes at the time of the density tests [47] was 10%. The densities of the briquettes without hollows were differentiated, respectively, as 1237 kg/m³
for briquettes without hollows and 59 mm diameter and 1169 kg/m$^3$ for those with hollows and a diameter of 73 mm (Figure 2). If a 95% confidence interval and the standard deviations of the values are taken into account (Figure 2), the densities will have the following ranges of variation: 1.199–1.274 kg/m$^3$ for T1 briquettes without voids and the diameter of 59 mm, 1.124–1.213 kg/m$^3$ for T2 briquettes without gaps and a 73 mm diameter, and 1.117–1.238 kg/m$^3$ for T3 briquettes of 71 mm diameter with an internal hollow of 26 mm.

The densities of the three types of briquettes were different for the same reasons. Regarding the two types of briquettes without voids, T1 and T2, the thinner briquettes had a higher density, because the same pressure of the piston acted on a smaller surface. For the same reason, the density of T3 hollowed briquettes was slightly higher than the T2 without voids, even though the outer diameter was almost the same.

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**Figure 2.** Empirical Cumulative Distribution Function (CDF) for the density of straw briquettes.

### 3.3. The Compressive Strength of Briquettes

The compressive strength of the briquettes was different from one batch to another, with higher values being obtained for the samples without hollows, type T1 and T2. It can be seen from Figure 3 that the range in variation of the compressive strengths was between 0.5 and 2.5 N/mm$^2$, with the lowest value of 1.15 N/mm$^2$ being assigned to the briquettes with hollows, and the highest value of 2.1 N/mm$^2$ to briquettes without holes with a diameter of 59 mm. It was concluded that thicker briquettes without hollows (T2) have a lower resistance than thin ones (T1), and briquettes with hollows (T3) have a lower compressive strength than those with voids (T1 and T2). These variations in compressive strength were determined by the type of specimen (with voids or without voids) and the density of the briquettes [48–50]. In this sense, the briquettes with hollows had lower resistances than those without holes, due to the inner hole greatly reducing the resistance. The range of variation calculated for a 95% confidence interval (for plus/minus two standard deviations) was 1.6–2.6 N/mm$^2$ for briquettes without holes with a diameter of 59 mm, 0.57–1.67 N/mm$^2$ for briquettes without hollows with a diameter of 73 mm, and 0.17–2.13 N/mm$^2$ for those with holes [51].
By adding additives, the compressive strength could be improved by 10–15% [39], through superior compaction and consistency of the crushed material in the briquette.

The maximum number of rows in a stack (Table 1) depended to the greatest extent on the average compressive strength (or average breaking force). From this point of view, the samples without hollows, type T1, had the maximum number of rows of 23 pieces. The group of specimens of T3 presented the lowest number of rows of 17 pieces because they had the lowest compression force of 395 kg; 33.9% lower than the specimens of T1.

Table 1. Modeling to find the maximum number of existing rows in a stack depending on the compression resistance.

<table>
<thead>
<tr>
<th>Type</th>
<th>Mean Mass, g</th>
<th>Mean Force, kg</th>
<th>Mean Resistance, N/mm²</th>
<th>Mean Diameter, mm</th>
<th>Mean Length, mm</th>
<th>Maximum Rows</th>
</tr>
</thead>
<tbody>
<tr>
<td>T1</td>
<td>208.1</td>
<td>598</td>
<td>2.10</td>
<td>59.95</td>
<td>59.49</td>
<td>23</td>
</tr>
<tr>
<td>T2</td>
<td>297.3</td>
<td>501</td>
<td>1.16</td>
<td>73.22</td>
<td>60.38</td>
<td>19</td>
</tr>
<tr>
<td>T3</td>
<td>253.8</td>
<td>395</td>
<td>1.14</td>
<td>D₉₀ = 71.12</td>
<td>D₉₅ = 26.8</td>
<td>17</td>
</tr>
</tbody>
</table>

3.4. Splitting Strength

This resistance had low values of 0.17–0.39 N/mm² (Figure 4), 81–85% lower than the compression resistance, for all three types of briquettes analyzed. The hollowed briquettes had the highest split resistance, and of the two categories of non-void briquettes, the smaller diameter briquettes had a higher split resistance than the larger diameter briquettes. This difference was explained by a slightly higher density of the briquettes with hollows, but also different working methods of the two mechanical properties of the briquettes.
mean of linear geometry, an intersection with the horizontal axis of the two linear equations was found for the value of 104% in the case of LCV and 164.5% for HCV. These means of linear geometry, an intersection with the horizontal axis of the two linear equations was therefore recommended when the calorific efficiency is over 94%. By observing after a value of 60% moisture content. A maximum moisture content of 10% for observed after a value of 60% moisture content. A maximum moisture content of 10% for calorific value of HCV and LCV decreased. Significant decreases in yield below 42% were calorific value of HCV and LCV decreased. Significant decreases in yield below 42% were observed. Based on the data obtained with the adiabatic calorimeter, the average values of the upper calorific value (HCV) of 17,260 kJ/kg and lower (LCV) of 17,010 kJ/kg were obtained for an average moisture content of briquettes of 4%. With the help of the previous relationship (8), the calorific value for absolutely dry briquettes (at a moisture content of 0%) was found to be 17,690 kJ/kg. Using some elements of linear geometry (generating a linear equation through two points), the two relationships of the high and lower calorific power was found to be 17,690 kJ/kg. Using some elements of linear geometry (generating a linear equation through two points), the two relationships of the high and lower calorific power (LCV = 17,690–170 Mc; HCV = 17,690–107.5 Mc) were found, with the help of which the graph in the following figure was created (Figure 5).

3.5. Calorific Value

It was observed that, with the increase in the moisture content of the briquettes, the calorific value of HCV and LCV decreased. Significant decreases in yield below 42% were observed after a value of 60% moisture content. A maximum moisture content of 10% for coated briquettes is, therefore, recommended when the calorific efficiency is over 94%. By means of linear geometry, an intersection with the horizontal axis of the two linear equations

Figure 4. Empirical Cumulative Distribution Function (CDF) for splitting strength of briquettes: T1 briquettes without voids with a diameter of 59 mm; T2 briquettes without voids with a diameter of 71 mm; T3 briquettes with inner hollows.

Figure 5. The influence of moisture content on the calorific value for straw briquettes: red line–HCV, blue line–LCV.
was found for the value of 104% in the case of LCV and 164.5% for HCV. These moisture content values, for which the calorific value is zero, point to some limiting values when the energetic effect is zero; that is, the amount of heat obtained through combustion is equal to that consumed for drying the wood briquettes. These values obtained on the basis of the graph in Figure 5 are referred to as the limiting moisture content of combustible materials.

The calorific value obtained per hectare of cultivated wheat, obtained with the help of (9), was 91,988 MJ/ha.

The calorific value of wheat straw was also modeled according to the carbon content. As shown by other authors [27,29,43], wheat straw has a lower carbon content than wood at 47.9%. This low carbon content is translated into a lower calorific value. Correspondence between the calorific value and the carbon content is, therefore, necessary to take into consideration, along with wheat straw, European beech wood (Fagus sylvatica L.) and wood charcoal obtained from beech wood. The dependency relationship developed by Channiwala and Parikh [18,19,43], adapted to the requirements of this modeling, was used:

\[
CV = 33.910 \cdot C + 11.783 \cdot H + 1.034 \cdot O - 0.211 \cdot Ac \quad [MJ/kg] \tag{15}
\]

where 33.910, 11.783, 1.034, and 0.211 are the calorific values of carbon, hydrogen, oxygen, and ash content, in MJ/kg; C is the carbon content in kg/kg, dry weight; H is the hydrogen content, kg/kg, dry weight; O is the oxygen content, kg/kg, dry weight; and Ac is the ash content, kg/kg, dry weight.

By substituting the percentage values of the carbon, hydrogen, and oxygen content into (15), the predicted value of the calorific value will be obtained, as given in the last column of Table 2. Based on these values (of the carbon content and the calorific value), the graph of Figure 6 was obtained. According to the methodology from point 2.2, the problem that Table 2 solves is that the calorific value of any plant material is dependent on the percentages of the component chemical elements and the calorific value of each.

<table>
<thead>
<tr>
<th>Materials</th>
<th>Carbon, %</th>
<th>Hydrogen, %</th>
<th>Oxygen, %</th>
<th>Ash Content, %</th>
<th>Calorific Value, MJ/kg</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wheat straw</td>
<td>47.8</td>
<td>5.1</td>
<td>38.1</td>
<td>9.1</td>
<td>17.402</td>
</tr>
<tr>
<td>Beech waste</td>
<td>49.9</td>
<td>6.4</td>
<td>43.0</td>
<td>0.7</td>
<td>18.119</td>
</tr>
<tr>
<td>Charcoal</td>
<td>81.6</td>
<td>7.1</td>
<td>10.1</td>
<td>1.2</td>
<td>28.753</td>
</tr>
</tbody>
</table>

\[ y = 0.3362x + 1.3184 \quad R^2 = 1 \]

Figure 6. The influence of carbon content on the calorific value of straw.
In this way, a new equation was obtained for determining the calorific value depending on the carbon content of lignocellulosic biomass, which can be quickly used to determine the calorific value (Equation (16)):

\[
CV = 0.366C + 1.318 \text{ [MJ/kg]}
\]  

(16)

For example, to analyze reed biomass with a 42.41% carbon content [13], by applying (16), we find a calorific value of 16,840 MJ/kg. The carbon content obtained per 1 ha of wheat was between 1.1 and 3.9 t/ha, in agreement with the results of others [13].

3.6. Calorific Density

Average calorific density values of 20,000–103,000 MJ/m³ were obtained but were differentiated for the three categories of briquettes. T1 non-hollowed briquettes had a calorific density of 21.882 MJ/m³, T2 non-hollowed briquettes had a calorific density of 20.679 MJ/m³, and T3 hollowed briquettes had a calorific density of 20.856 MJ/m³. These values were in agreement with the results obtained by other authors [43].

3.7. Ash Content

The calcined ash content of straw briquettes was, on average, about 9.1%. This value was 10 times higher than that of beech briquettes (Fagus sylvatica L.), which had a value of 0.9%.

Figure 7 is a statistical diagram of the average value, standard deviation, number of tests, \(p\)-value, and Anderson–Darling’s coefficient. A statistical analysis of the values was made and the range of variation of the values was found for a confidence interval (CI) of 95%. The small values of the two statistical parameters of the graph in Figure 7 of the Anderson–Darling coefficient (AD) and \(p\)-Value, as well as the values falling between the two limits of the graph, showed the normality of the distribution of values. If the arithmetic medium of the values and the statistical confidence interval of 85% were taken into consideration, there was a range of variation in the calcined ash content of 8.89–9.33%. The high ash content of straw briquettes also has an influence on the deposits in the combustion plant, especially when the temperatures exceed the melting values of fly ash [52].

![Figure 7. Probability plot of ash content for straw briquettes.](image_url)

The calorific value of wheat straw was modeled according to the ash content and was based on the analysis of the calorific value and ash content of several plant biomass [10]...
and on the correlations found in the research for beech (with an ash content below 1% and a calorific value of 18.67 MJ/kg) and for wheat straw (with an ash content of almost 10% and a calorific value of 17.69 MJ/kg). In this way, the linear variation equation (Figure 8) of those two parameters \( CV = 19.164 - 0.49 \cdot Ac \) was found, based on which the calorific value of various plant biomass could be approximated in relation to the ash content. For example, in the case of exotic biomass such as bamboo, with an ash content of 3.5%, a calorific value of 17,449 MJ/kg was obtained, which is close to that found in the specialized literature [53].

![Figure 8. The influence of the ash content on the calorific value.](image)

In the range of 0–20% of calcined ash, the calorific value decreased from 18.67 MJ/kg to 16.65 MJ/kg, which means a decrease of 0.1 MJ/kg for each percentage of calcined ash. Considering that the average amount of straw is 5.2 t/ha, an average amount of ash of 473 kg/ha was obtained.


The average value of volatile content, fixed carbon, and ash content, obtained on the basis of the work methodology from chapter 2 and Equation (14), are shown in Figure 9. The average values for volatile content of 68.44 ± 5.47% and fixed carbon of 22.45 ± 2.11% for wheat straw were obtained.

![Figure 9. Content of ash, volatile content, and fixed carbon for wheat straw.](image)

### 4. Discussion

The density was different for hollowed and non-hollowed briquettes. Smaller diameter hollow briquettes (T1) compressed better than larger diameter ones (T2), as the same pressure of press was exerted on a smaller surface area [54]. The standard deviation
was lower for briquettes with a smaller diameter T1 (with a value of 18.24 kg/m$^3$), even though the effective density value of 1237 kg/m$^3$ was the highest. Briquettes with T3 hollows had a slightly higher density than those without T2 voids, for the same reason as the T1–T2 comparison, namely due to the reduced pressure application surface on the ground straws [55–58].

With a view to a comparative analysis, the data on the straw characteristics are given in Table 3, next to similar characteristics of European beech biomass (Fagus sylvatica L.).

Table 3. Comparison between briquettes of wheat straw, T1, and European beech.

<table>
<thead>
<tr>
<th>No.</th>
<th>Characteristics</th>
<th>Triticum aestivum L.</th>
<th>Fagus sylvatica L.</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Moisture content, %</td>
<td>10</td>
<td>10</td>
</tr>
<tr>
<td>2.</td>
<td>Effective density, kg/m$^3$</td>
<td>1.21</td>
<td>1.26</td>
</tr>
<tr>
<td>3.</td>
<td>Calorific value CV, MJ/kg</td>
<td>17.69</td>
<td>18.38</td>
</tr>
<tr>
<td>4.</td>
<td>Calorific density, MJ/m$^3$</td>
<td>20.679</td>
<td>21.856</td>
</tr>
<tr>
<td>5.</td>
<td>Volatile, %</td>
<td>68.44</td>
<td>66.3</td>
</tr>
<tr>
<td>6.</td>
<td>Fixed carbon, %</td>
<td>23.45</td>
<td>32.8</td>
</tr>
<tr>
<td>7.</td>
<td>Compressive strength, N/mm$^2$</td>
<td>2.1</td>
<td>4.6</td>
</tr>
<tr>
<td>8.</td>
<td>Expansion coefficient, %</td>
<td>1.6</td>
<td>1.8</td>
</tr>
<tr>
<td>9.</td>
<td>Ash content, %</td>
<td>9.1</td>
<td>0.9</td>
</tr>
</tbody>
</table>

In terms of the general values of the properties of wheat straw briquettes, the experimental briquettes obtained from wheat straw were made with powerful installations, thus obtaining a high density. Due to the high densities (1.1–1.2 g/cm$^3$), the other mechanical properties (compression and splitting) also had high values (over 1.6 N/mm$^2$).

The three types of wheat straw briquettes had similar characteristics, such as a higher ash content (8.8–9.3%) and good calorific value (17.4–17.8 MJ/kg). The high ash content was due to the large amounts of secondary compounds, such as oxalates and carbonates, at 6–8% compared to 1–3% for solid wood [43,59]. This is why a comparison was needed with briquettes made from hardwood residues (beech biomass). This also emerged from the fact that the limiting values of the properties according to the ONORM standard were expressed for the entire range of briquettes, especially for those based on wood, which was the most common on the world market. For this, research was carried out for a comparison with briquettes made from beech chips (Fagus sylvatica L.).

An increase of 1% of ash led to a decrease of 0.2 MJ/kg of the calorific value since the ash does not contribute substantially to the heat developed by combustion, although some mineral elements in the ash can act as catalysts for the thermal decomposition of the fuel. Additionally, an increase in the ash content led to a proportional decrease in the carbon content, the latter being the main chemical compound that determines the increase in the calorific value of wood.

Compared to other woody biomass such as pine [10], the volatile content of straw was much lower, by about 17%, and the fixed carbon content of wheat straw was 30.4% lower. Compared to beech wood, which has a volatile content of 66.3% and fixed carbon of 32.8%, wheat straw had higher values of volatiles by 3.2% and lower values of fixed carbon by 28.5%.

5. Conclusions

In general, all briquettes obtained from wheat straw can be considered a consistent source of ecological fuel, with properties and uses comparable to briquettes obtained from wood chips or firewood of beech biomass.

The density of briquettes made of wheat straw of 1237 kg/m$^3$ for briquettes without hollows and 1178 kg/m$^3$ for briquettes with hollows was comparable to that of the usual wood waste used as briquettes.
The calorific value per hectare of cultivated wheat of 91,988 MJ/ha represents an increase in renewable and sustainable energy at the level of each household. The calorific value of 17.69 MJ/kg of wheat straw was similar to that of beech waste, with a decrease of only 3.7%.

The particularly high ash content per hectare of 473 kg/ha shows that methods for the efficient use of ash must be considered. Ash collection must be undertaken at shorter intervals, and larger and covered storage spaces will be needed.

The mechanical properties, such as the compressive strength of 2.1 N/mm² for briquettes without hollows and 1.15 N/mm² for briquettes with hollows, placed these types of briquettes in the category of solid and strong briquettes.

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References


