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On the Rheological Behavior of Pine (*Pinus sylvestris* L.) Shavings and the Briquettes Obtained from Them

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Abstract: The aim of this paper is to carry out a rheological study of the pressing of pine wood sawdust, after which obtaining briquettes from the same wood material with a hydraulic installation and analyzing their properties. In order to know the rheological behavior during pressing, the fractions resulting from sorting the sawdust with 4 × 4, 3 × 3, 2 × 2, and 1 × 1 mm^2 sieves were used, respectively, six fractions (the fraction larger than 4 × 4 mm^2 and the smaller one of 1 × 1 mm^2 are added) and a specific pressing device, placed on a universal testing machine. The results obtained in the rheological study showed that the obtained density does not increase proportionally with the pressure, the best results (density of 1030 kg/m^3) being obtained at a pressure of 180 MPa. Within the briquettes, higher densities were obtained for the sawdust fraction smaller than 1 × 1 mm^2, but the breaking strength was higher for the fraction larger than 4 × 4 mm^2. As a general conclusion, it was found that pine sawdust is easily compressible, and the briquettes obtained from it have good properties for use in combustion.

Keywords: rheology; pressing; pine wood; shavings; briquette; wood waste; calorific value; briquette resistance

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1. Introduction

In Europe, wood waste represents a renewable energy source and plays an important role in the field of energy. Wood waste represents a substantial part of the biomass wood waste, by regulating its properties but also by optimizing their use. Currently, the inexhaustible source of wood waste is the forestry processing industry. This must be evaluated in order to find the ones efficient methods of energy. For example, the amount of sawdust produced differs from one woodworking process to another, by 13% for softwood sawmills with a capacity of 50,000 m^3/year, 9.6% for hardwood lumber of 180,000 m^3 capacity/year, 3% for flitch with 4300 m^3 logs/year capacity, 17% to 22% for solid wood doors and solid wood furniture, 3% for strip parquet flooring, 15% for wood parquet, etc. [1,2].

Wood briquettes are densified lignocellulose materials that contain a large amount of concentrated energy in a low volume. Their calorific value is higher because of low moisture content and could be kept because they are follies. Their main advantage refers to the fact that briquettes use wood biomass, respectively, the remains that fall to the primary processing of wood in the form of lumber or logs [3].The decomposition of these lignocellulose materials in nature (regardless of the shape and size of these remains) would lead to the elimination of a quantity of carbon dioxide in nature, equivalent to that absorbed by the tree during its life [3–5]. To this ecological aspect of the briquettes, the existence of a renewable and sustainable fuel product can be added that meets the needs of mankind for heating homes and cooking food in the countryside, sometimes even for obtaining electricity by cogeneration.

The use of different wood species in as many combinations as possible can lead to the realization of a great category of combustible materials that could be used in the heating of rooms [6] obtained hollow-core wood briquettes made from two species such as spruce and oak wood. Comparison with the classic types of briquettes was carried out in order to be able to find some advantages and disadvantages of these briquettes. The results obtained show that there are few differences between the characteristics of hollow briquettes and those of classic ones [6]. Also, the biomass briquettes market size is expected to reach USD 612.6 million by the end of 2026. Classic briquettes differ from hollow core briquettes by the pressing method and equipment [6–9]. Given the economic character, it is completely possible to fit these briquettes in the field of cooking and heating [10,11].

Among the softwood species, pine trees have a dominant growth both at European and world level. The forested area worldwide is very different, pine trees being among the most widespread softwood species on the globe, with almost half of the world's forests area. Also, the top three countries with the largest forest reserves are the Russian Federation, Brazil, and Canada, from which the largest quantities of wood remain is resulted (Figure 1) [12–14].

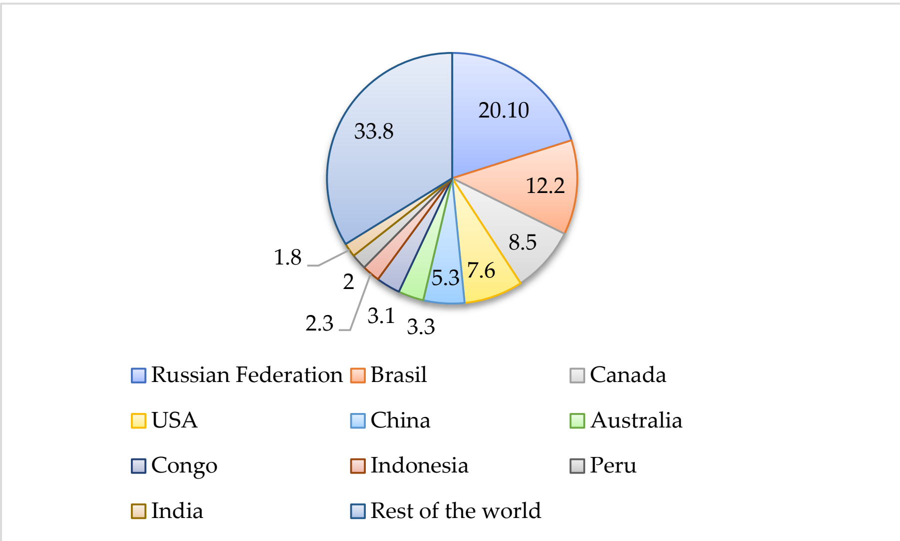


Figure 1. Global forest fund distribution [12–14].

Wood species with a low quality represent one of the main directions of development of the forest industry [15]. The reorientation of the industry from the export of unprocessed wood to the production of products with high added value implies the improvement new technologies for the processing of extracted wood from forestry [15–18].

Usually, branches and tree tops remain at the cutting site, which leads to the loss of raw materials that could be used in the woodworking industry. Moreover, this practice leads to the pollution of forest ecosystems and an increased risk of fires during the warm season of the year [1,16]. At enterprises specializing in the production of timber products, production waste such as sawdust and wood chips are not used to the required extent; however, these are valuable raw materials that can be used for the production of new materials as briquettes and other purposes [19–22].

When logs are cut and lumber is produced, various sizes and shapes of waste are generated. The main constituent of this waste is shredded wood as the particle size, configuration, and production method can vary. Wood waste, including sawdust and fine chips, is then sent for recycling, used to produce steam and energy, or stored in piles and taken to a landfill. The share of chip sifting can be up to 8% of the total volume of chopped wood. One of the approaches to solving the problem of using shredded wood is its processing and subsequent use in wood briquetting production and household use. An example of the implementation of this approach is the grinding of wood with subsequent pressing in the form of fuel briquettes [23–25].

To develop the theory of compaction, it is important to take into account the dependence of the deformation properties of a conglomerate on its density. In other words, for the effective use of wood waste, place an important role the rheological behavior of crushed wood during the processing of the raw material. The study of the rheological behavior of crushed wood is an important aspect when considering the technology of its processing and use [26–28]. Rheology refers to the study of the flow properties of materials when subjected to mechanical forces and deformations. Shredded wood is a composite material consisting of various particles of different sizes and shapes. Rheological properties are determined by the interaction of particles, their concentration, and structure. Important parameters characterizing the rheological behavior of crushed wood are compressibility, viscosity, plasticity, elasticity, and flowability of the material under various conditions. The study of the rheology of crushed wood during uniaxial pressing is promising, both in the fundamental aspect of the study of bulk materials, and in the significance applied, for example, for the production technology of briquettes [29–31] or building materials. In the process, the material is subjected to pressure in one direction, which leads to its compaction and the formation of semi-finished of a certain shape and size. The rheological characteristics of the material affect its ability to be pressed, formed and the strength of the resulting briquettes. A deeper study of the rheology of shredded wood makes it possible to optimize the parameters of the pressing process, to select optimal conditions and technologies for processing wood waste. Such research help to increase the efficiency of the wood use and decrease the negative impact on the environment.

The objectives. This paper was directed in two main directions. First of all, the purpose of this paper is to study the rheological behavior of crushed wood pine during uniaxial pressing, to determine the optimal parameters of pressing, and also to study the effect of fractional composition on compressibility and burst resistance of the resulting briquettes. Secondly, the objectives of the paper are directed towards the briquettes obtained from pine sawdust by means of a briquetting machine with hydraulic drive, determining the mechanical, physical, and calorific properties of the compacted piece, including the rheological behavior.

2. Materials and Methods

2.1. Rheological Behavior of Shredded Pine During Uniaxial Pressing

Studying the rheological aspects of the uniaxial pressing of pine (*Pinus sylvestris* L.) wood crushed was made by various fractional compositions. This material was obtained from a planning machine from Reghin (Romania) when some pieces of timber with a medium annual ring width of 2.2 mm and a medium width of latewood of 0.9 mm were processed. The kinetics of the deformation of crushed pine wood was studied on a constant speed of movement of the press also using some rheological aspects. Rheological aspects–foreign curves were made for various fractions with ten replicates for each.

Materials are presented to study the rheological aspects of uniaxial pressing of crushed pine wood of various fractional compositions. The deformation modality of crushed woods was studied by displacement of the press piston using a rheological aspect. Rheological deformation curves have been constructed for six different fractions ($d < 1$ mm; 1 mm $< d < 2$ mm; 2 mm$< d < 3$ mm; 3 mm $< d < 4$ mm; $d > 4$ mm). The object of study was pine wood from shaving waste with six fractional compositions and dried at the 10% in the drying pot (Figure 2).

Figure 2. The sawdust used during the experiment (fractional composition of pine wood in the form of shavings): (**a**)-fraction greater than 4 mm; (**b**)-fraction of 3–4 mm; (**c**)-fraction of 2–3 mm; (**d**)-fraction of 1–2 mm; (**e**)-fraction less than 1 mm; (**f**)-unfractionated sawdust (whole).

Before the weighing operation, the wood was laid successively layer by layer in a metal mold (Figure 3) to the edges, after which it was weighed on an electronic balance from Bucharest (Romania) with a measurement accuracy of 0.1 g. The diameter of the working volume of the mold was 25 mm and the maximum height was 80 mm.

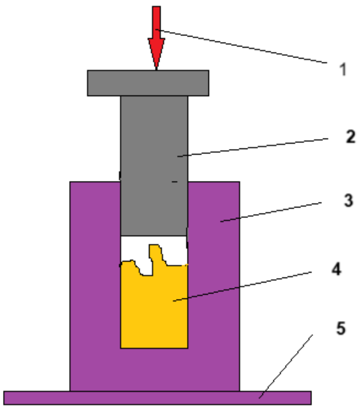


Figure 3. Scheme of a metal mold used in the compression of briquettes. 1—rod; 2—cylindrical shape of the piston; 3—body; 4—filling of the cylindrical space with the studied sawdust composition; 5—device base.

Calculation of bulk density is carried out according to the following formula:

$$\rho = \frac{m}{S \times h} \quad (1)$$

where

m is the mass of the sawdust, g;

s—cross-sectional area of loading mold, cm^2;
h—is the height of the mass layer, cm.

$$\rho_r = \frac{\rho}{\rho_{ws}}, \quad (2)$$

where ρ—actual density, in g/cm^3; ρ_{ws}—density of the wood substance, equal to 1.53 g/cm^3.

The deformation of the studied compositions was carried out on a universal test machine "INSTRON 3369" at a constant speed of 5 mm/s. The measurement accuracy provided was 1%. The entire experimental series was carried out in room conditions. The prepared metal form shown in Figure 3, with the composition under study, was installed under the piston of the test machine. A load was applied to the piston, under the influence of which the rod deformed the volume of the filler, and the mixture was compacted to a maximum load, upon reaching which the load was removed, and the compact was removed. With the help of the software of the test machine INSTRON 3369, rheological curves of the relationship between stress and deformation were constructed (the general characteristic shape of the curve is shown in Figure 4), from which one can appreciate the rheological properties of the studied composition and the optimal compressibility conditions.

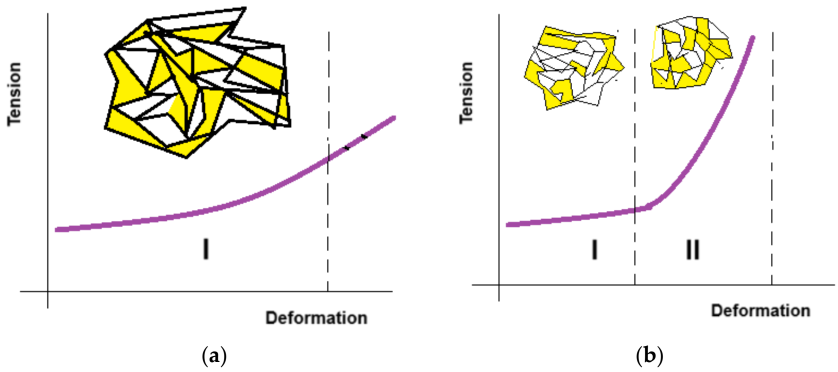


Figure 4. Typical appearance of the pressure–rheological deformation curve: (**a**): first stage; (I)-weak increase in tension with increasing deformation; (**b**)-2nd stage (II)-intense nonlinear increase in tension with a slight increase in deformation.

With the universal machine, the dependence of the density on compaction was observed. The density dependence of compacts on the compaction pressure was determined using a universal compression machine. The applied pressure ranged from 20 to 200 MPa in increments of 20 MPa. The form with the mixture was installed under the piston of the compression machine to which constant pressure was applied. After compaction and holding the compact under constant pressure for 30 s, the compact was removed, then its mass and height were measured, and the density was calculated at each applied pressure.

The result of the diverse composition of shredded pine wood on the strength of compacts when was obtained without the use of adhesive was studied using a developed installation (Figure 5).

As a strength criterion, resistance to destruction during a free fall from a height of 2 m was used. To do this, a compacted product was installed on the platform, then the locking rod holding the platform with a rope was pulled, and the sample under the influence of gravity falls on a steel plate, always in the same place. Experiments were carried out with samples obtained at low values of compaction pressure from 20 to 60 MPa, since more compacted samples are less susceptible to destruction under these conditions. After each fall, a change in the mass of the compact was recorded, a critical indicator was a loss of mass of the compact by more than 20%.

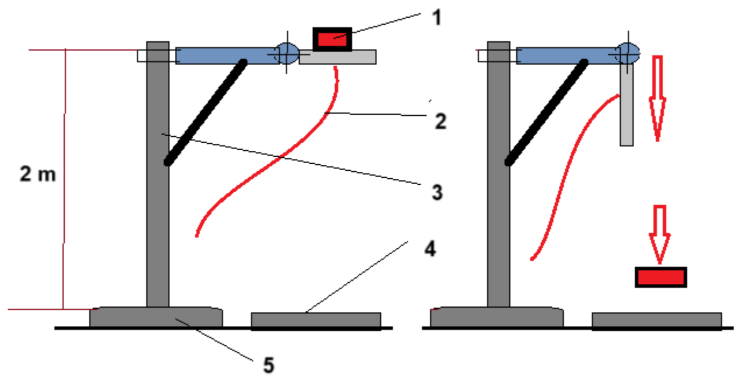


Figure 5. Scheme of the installation in free fall: 1—sample; 2—string for tablet unlocking; 3—locking rod; 4—steel plate; 5—device base.

2.2. Methodology for Briquettes

Pine wood shavings obtained from a planning machine had so-called dimensional characteristics, which is why its granulometric composition was determined using the following dimensions of the sorting sieves: 4 × 4, 3 × 3, 2 × 2 and 1 × 1 mm^2, meaning five different fractions were determined (with ten replicates for everyone). To this, unfractionated sawdust was added. The electrical fractionation installation is shown in Figure 6, where the body of the device, the sieves, and the columns for compact fixing of the sieves during vibrations are observed.

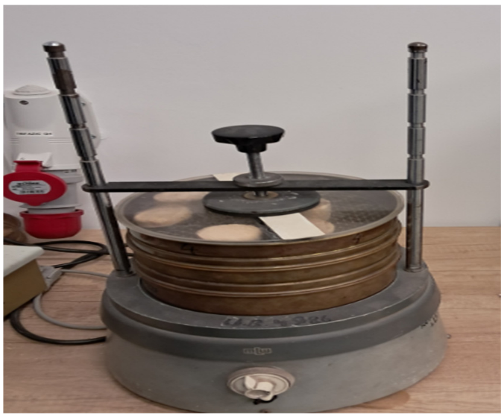


Figure 6. Device with electric vibrations for sieves sorting of pine shavings.

Of the same material used in the previous sub-chapter, namely pine wood shavings (*Pinus sylvestris* L.) briquettes were made using a hydraulic installation with two pistons (one for supplying material and the other for compressing the agglomerate to the density of briquette). The typology of the briquettes and the size of the chips of the various chips are visible in Figure 7.

Density of briquettes. The wood shavings conditioned and sorted into the six distinct fractions (less than 1 mm, between 1 and 2 mm, between 2 and 3 mm, between 3 and 4 mm, larger than 4 mm and unfractionated) were subjected to briquetting operation on a briquetting installation with hydraulic actuator type GOLD Star (Brasov, Romania). The compression parameters used were the temperature of 18 °C and the relative humidity of 65% to obtain a constant moisture content of about 12% [32–34]. Then, the dimensions of each of the 14 briquettes of each group were determined (Table 1), and the density was determined using the following calculation relation:

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

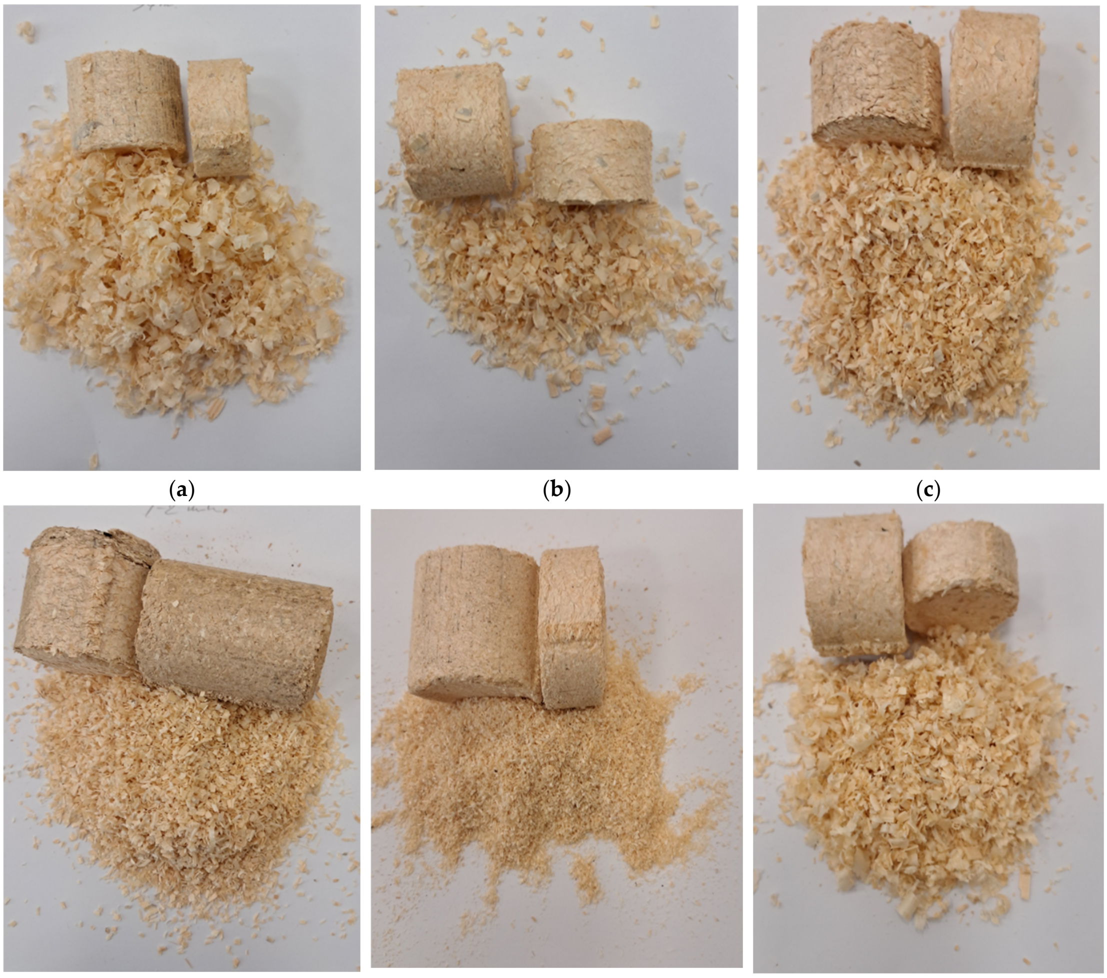
where

m—is mass of samples, in g;

d—diameter of the briquette, in mm;

l—length of the briquette, in mm;

14 replications of the test were performed.



(**a**) (**b**) (**c**) (**d**) (**e**) (**f**)

Figure 7. Briquettes made from pine shavings: (**a**)—higher than 4 mm; (**b**)—between 3 and 4 mm, (**c**)—between 2 and 3 mm, (**d**)—between 1 and 2 mm, (**e**)—lower that 1 mm, (**f**)—from total saw-dust.

Table 1. Initial parameters of pine wood shavings and values of obtained compaction density.

Particle Size	Mass, g	Height, cm	Pressing Height, cm	Bulk Density, g/cm	Compacted Density, g/cm^3	Relative Density g/cm^3
A: d < 1 mm	3.122	4.9	0.6	0.131	1.060	0.693
B: 1 < d < 2 mm	3.195	5.3	0.7	0.123	0.930	0.608
C: 2 < d < 3 mm	3.382	5.7	0.8	0.121	0.862	0.563
D: d > 4 mm	3.358	6.7	0.8	0.103	0.855	0.559

Determination of agglomerate density and compression ratio between briquettes and wood shavings. To determine the density of uncompressed wood shavings, a graduated cylinder in units of volume [cm^3] was used, which was filled up to a certain height. By weighing the distinction between the mass of the wood shavings cylinder, the mass of the hollow cylinder was determined. This represents the mass of the wood material used to determine this density. The density in the loose state of the wood shavings was determined by dividing mass to volume [35].

There were 10 replications of the same test.

Calorific value of pine wood shavings. To determine the calorific value of crushed pine material, it was tableted in small pellets of about 0.8 g, corresponding to the technology but also the dimensions of the installation for determining the upper calorific value (HCV) and lower (LCV) [31]. The determination installation was prepared by placing the nichrome wire and cotton wire in the calorimetric bomb and filling it with technical oxygen up to a pressure of 30 atm [36–38].

The results obtained for the 10 replications of the test, in the form of high and low calorific value, were analyzed statistically.

The formula used by calorimetric bomb was as follows:

$$CV = k \cdot \frac{T_f - T_i}{m} - Q_{nw} - Q_{cw} \, [\frac{\text{MJ}}{\text{kg}}] \quad (4)$$

where

k—calorimetric coefficient;

T_f—final temperature in calorimetric vessel, in °C;

T_i—initial temperature in calorimetric vessel, in °C;

m—mass of the analyzed sample, in g;

Q_{nw}—the quantity of heat eliminated by the nichrome wire, in MJ/kg;

Q_{cw}—the quantity of heat eliminated by burning the cotton wire, in MJ/kg.

Ash content of briquettes. Ash content was determined by taking a few grams of very fine material that passed through the 1×1 mm^2 sieve [39]. It was dried to anhydrous value in order to eliminate moisture as an influencing factor of the ash content. The actual determination was made by using a ProTerm calcination furnace (Ploiești, Romania), at a temperature of 750 °C [39,40]. The crucibles and wood samples were weighed using an electronic balance with an accuracy of 3 decimals. The calcination was carried out by means of metal crucibles, resistant to high temperature, whose mass was taken into account when determining the ash content, with the following formula:

$$A_c = \frac{m_a}{m_s} \cdot 100 \, [\%] \quad (5)$$

where

m_a—mass of calcined ash, in g;

m_s—mass of dried material sample.

In order to protect the calcination furnace against smoke and soot resulting from the calcination of the wood material, before placing in the calcination furnace, the crucible with the wood material was arranged above a flame from a butane gas cylinder until the flame was stopped, thus obtaining a black ash (as opposed to be calcined one that had a light gray color), whose black ash content was determined with a similar relationship to the previous one (Equation (5)).

Ten replications were made for this test.

Compression resistance. This mimics the crushing action of the briquettes while stationary under the action of the weight that is above them [41,42]. It is considered that the burst area of briquette is its longitudinal section, which is why the calculation formula is as follows:

$$\sigma_c = \frac{F_{max}}{d \times l} \, [\frac{N}{mm^2}] \quad (6)$$

where

F_{max}—maximum crushing force, in N;

d—briquette diameter, in mm;

l—briquette length, in mm.

Ten replications were made for this test.

Abrasion of briquettes. This determination mimics the friction action of the briquettes between them during transport to the beneficiary. For this, a set of 5 briquettes were taken from each batch of manufacture, having a maximum length of 25 mm in order to abrase them.

Ten replications were made for this test.

Statistical analysis. The main statistical parameters were determined, namely the arithmetic mean, the survey median, the standard deviation and the coefficient of variation. Also, when using variation graphs, statistical parameters of standard statistical deviation type and Pearson's coefficient R^2 were used.

3. Results

3.1. Results Regarding Agglomerate Compaction

In the case of these tests, the density compression curves were obtained as a function of pressure. The optimal compressibility parameters in the case of fractions with particle size smaller than 1 mm, reach some high densities at low pressures (ρ_{zag} = 1.03 g/cm^3 la P = 180 MPa). Other fractions studied had about the same compressibility and density ρ_{zag} in the range of 0.85–. . .0.90 g/cm^3 at P = 140–. . .160 MPa. Due to the elastic expansion after the removal of the load, the additional increase in the pressing pressure did not lead to an increase in the density.

Also, regarding the mechanical properties of the compacts made, the greater strength of the compacts was obtained when a part of size greater of 4 mm fell. These results can be used to optimize the process of pressing shredded wood and find more efficient methods of using wooden resources. Then, the dependence of applied pressure and compact density was established. On this basis, an assessment of the compactness of bulk materials was made, which allows, in the future, the selection of the pressure necessary to ensure a certain density value (Figure 8).

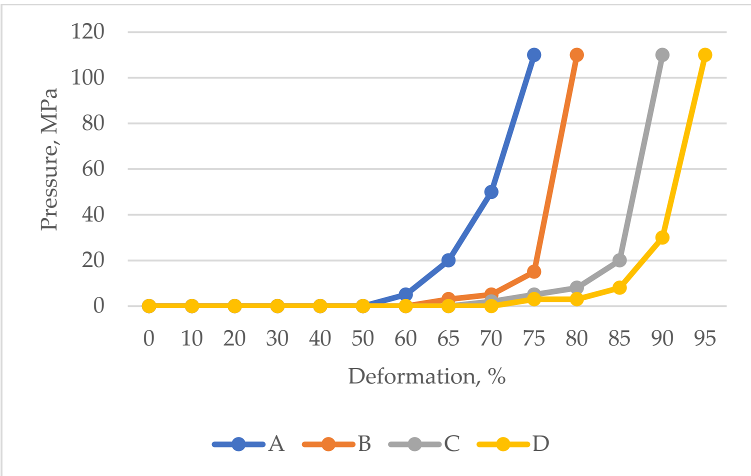


Figure 8. Some rheological curves for different fractional compositions of shredded pine wood: A: d < 1; B: 1 < d > 2; C: 2 < d > 3; D: 3 < d < 4.

The density of compaction is equal or approximately equal to the density of the maximum compact (density of woody substance) (Table 1).

As a result of the experiments, compression curves were obtained graphs of the dependence of the compaction density on the applied pressure. Figure 9 shows the resulting dependency graphs.

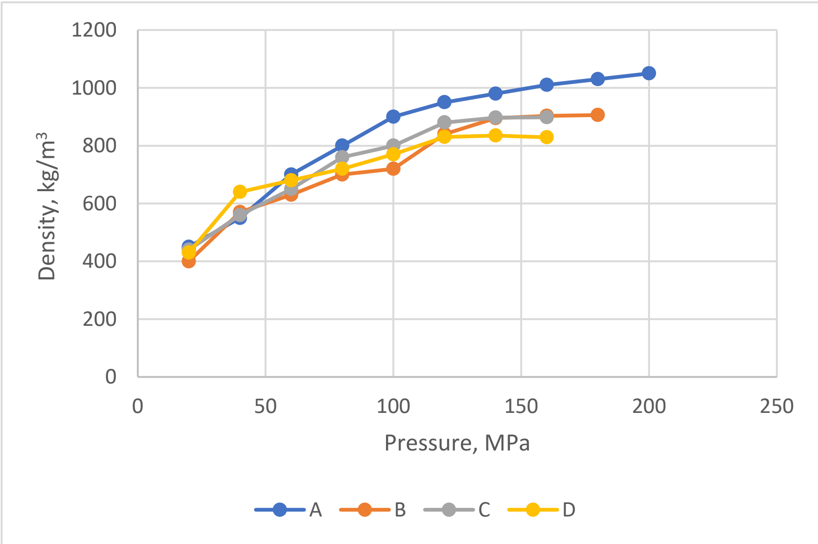
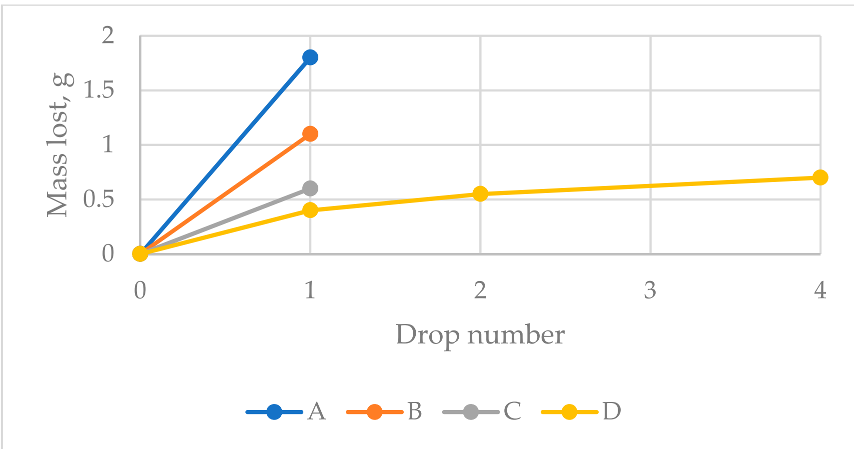


Figure 9. Compression curves "density-pressure" for different fractional compositions of shredded pine wood: A: d < 1; B: 1 < d < 2; C: 2 < d < 3; D: d > 4.

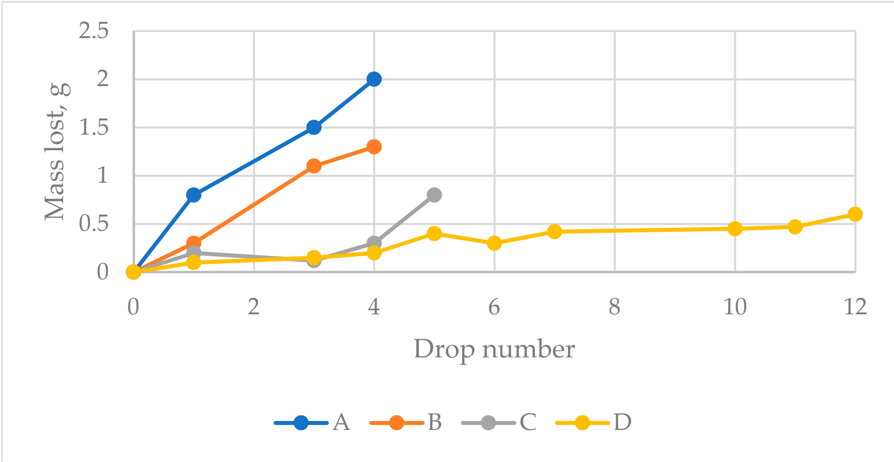
The study of the results of the different compositions of shredded pine wood on strength was carried out on pressed parts in a low range of pressing pressures (20, 40, and 60 MPa). The use of compacts obtained at high pressures did not allow us to trace the influence of fractional composition using this test method due to their high strength. The test results are shown in Figure 10.

In a free fall from a height of 2 m, all parts pressed under a pressure of 20 MPa (Figure 10a) of the fractions d < 1, 1 < d < 2 and 2 < d < 3 mm lost more than 20% of their mass after the first all. The compact with larger fractions d > 4 mm failed after four falls.



(**a**)

Figure 10. *Cont.*



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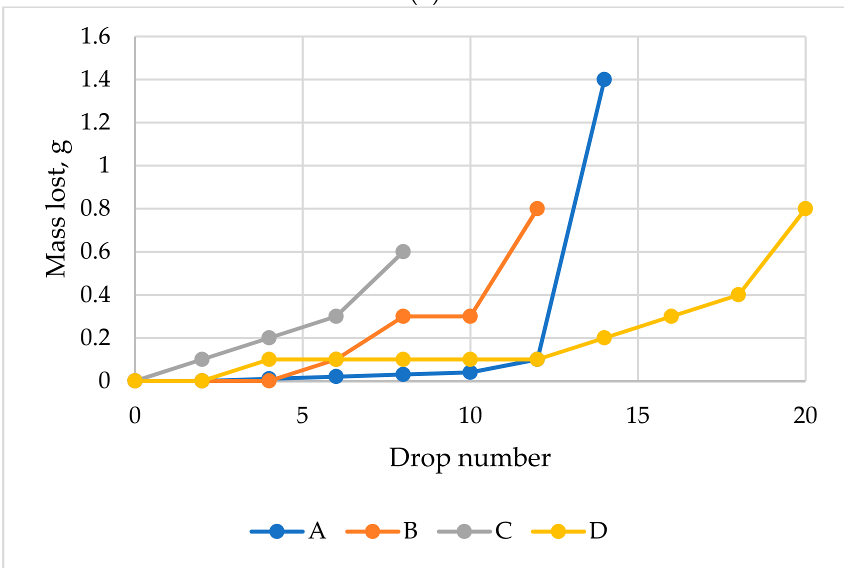


(**c**)

Figure 10. Absolute reduction in mass during free fall of samples from the studied compositions of shredded pine (A: d < 1; B: 1 < d < 2; 2 < d < 3; d > 4), compressed under pressure: (**a**) 20 MPa; (**b**) 40 MPa; (**c**) 60 MPa.

The results of compacts obtained at a pressure of 40 MPa (Figure 10b) correlate with past performance. Compacts made of a coarse fraction d > 4 mm had the highest resistance to destruction during free fall, the loss of critical mass occurred after 14 falls. Other compacts in fractions d < 1, 1 < d < 2 and 2 < d < 3 mm failed after four, three, and five falls, respectively. When testing compacts obtained at a pressure of 60 MPa (Figure 10c), the same tear resistance during free fall was demonstrated by compacts from the fraction d > 5 mm, critical fracture, which occurred after the 20th fall. Other compacts in fractions d < 1, 1 < d < 2 and 2 < d < 3 mm failed after 14, 12, and 7 falls, respectively.

3.2. Results Regarding Wood Briquettes

Wood shavings granulometry. Based on the 10 rows of mass values determined at the sieves sorting of pine wood shavings, different percentages were determined for each fraction (by dividing the mass fraction by the total mass of the sample taken), as seen in Figure 11.

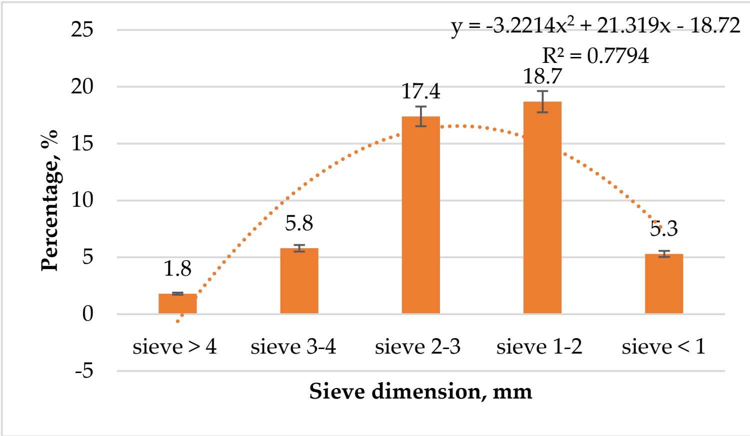


Figure 11. Wood shavings granulometry.

Bulk density of wood shavings. Following the methodology expressed in Section 2 of the paper, we obtained the bulk density of unfractionated wood shavings (113.7 kg/m^3, with a standard deviation of 10.5 kg/m^3 and a coefficient of variation of 0.092), but also of each fraction (Figure 12).

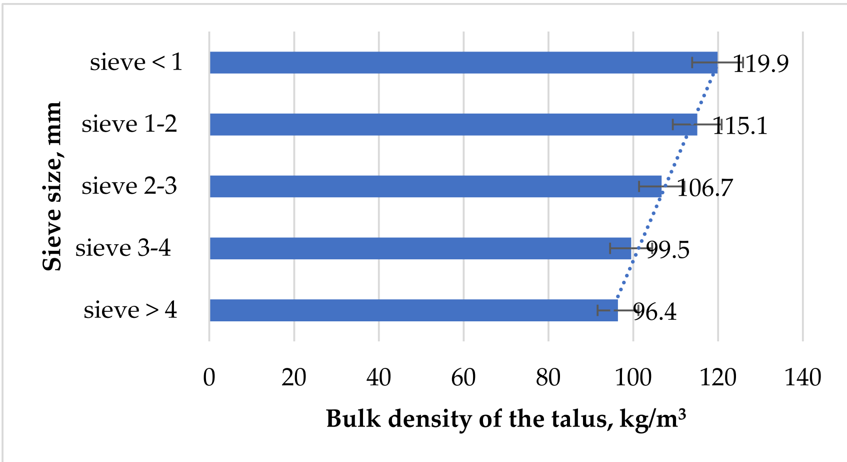


Figure 12. Bulk density of talus is related to sieve size.

Density of briquettes. The density of the obtained briquettes for each fraction was expressed separately, determining the arithmetic mean, the standard deviation, and the coefficient of variation for the 14 determined values (Table 2).

By repeating the procedure for determining the effective density of briquettes for all five distinct fractions considered, it was found that the variation range of briquettes was between 820.5 and 915.1 kg/m^3 (Figure 13).

From the previous figure it can be seen that the general tendency of the density of briquettes is to increase once the particle size decreases, demonstrating once again that small particles compact much better than large ones.

The degree of compaction of the briquette. The compaction of briquettes was determined both according to the density level of the bulk sawdust compared to the density of briquettes (850.7/113.7 = 7.48 times) and the level of the wood species used (density Pinus sylvestris 560 kg/m^3) (850.7/560 = 1.34 times, or an increase of 34%). Regarding the variation in this degree of densification with respect to the particle size of the wood shavings, the principles exposed to the density variation according to the particle size were observed, obtaining compaction coefficients of 8.5, 8.29, 8.12, 7.63, and 7.62 times.

Table 2. Density of briquettes made of pine shavings; fraction greater than 4 mm.

Number Sample	Mass (g)	Diameter (mm)	Length (mm)	Density (kg/m^3)
1.	22.1	41.66	19.6	827.61
2.	22.9	40.92	19.4	858.81
3.	30.9	40.89	28.93	813.77
4.	27.1	40.87	25.62	806.69
5.	22.4	40.54	20.35	853.19
6.	30.9	40.85	27.83	847.61
7.	35.5	41.81	31.67	816.86
8.	31.1	41.27	29.33	793.06
9.	50	41.51	42.46	870.59
10.	36.9	41.69	34.81	776.94
11.	31.4	40.66	32.6	742.17
12.	21.8	41.25	19.78	825.11
13.	33.1	41.15	29.7	838.42
14.	25.9	41.23	22.04	880.62
Mean	30.14	41.16	27.43	822.84
			Std.dev.	4.86
			Coeff. of variation	0.006

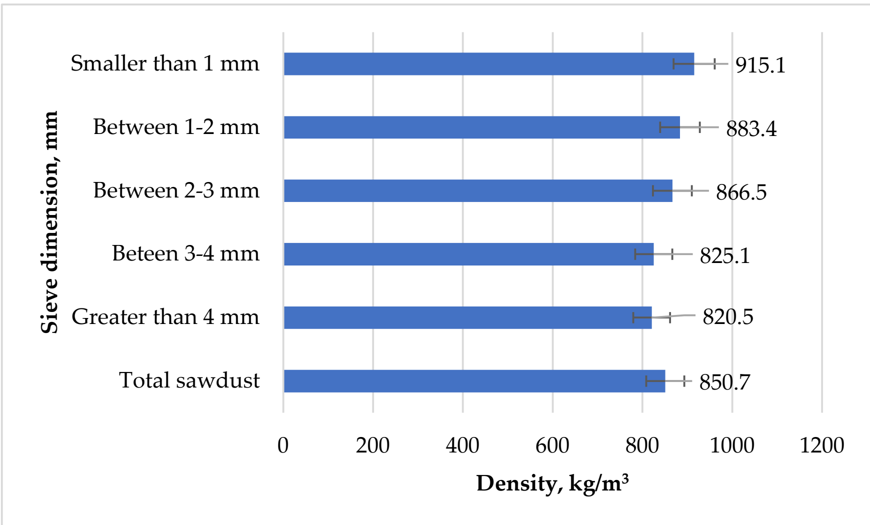
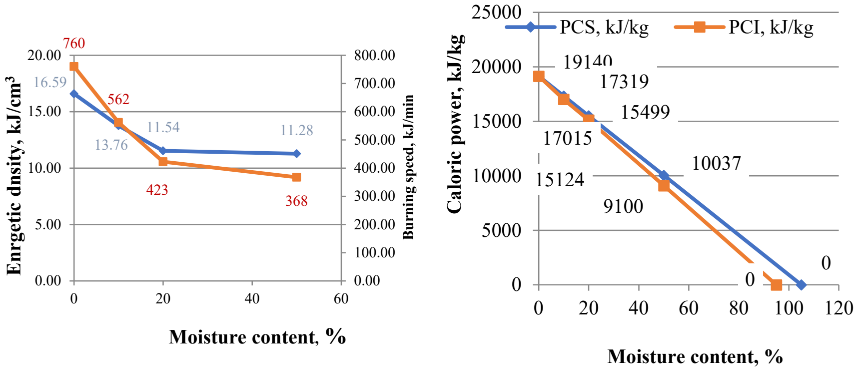


Figure 13. Density of briquettes depending on particle size (for o pressure of 20 MPa).

If we take into account the wood density of pine wood of 1.53 g/cm^3, we will find a ratio of densities or relative density of 0.53, 0.54, 0.56, 0.57, and 0.59, respectively, a reserve of compression-compaction of briquettes resulting from various fractions, of 46.3%, 46.1%, 43.3%, 42.2%, and 40.1%, respectively.

Calorific value of pine shavings. The calorific value of pine wood in the form of pellets with mass around 0.8 g was 19.14 MJ/kg for anhydrous wood, and when the briquette moisture content was 10% the high calorific value was 17.32 MJ/kg and the low one was 17.02 MJ/kg (Figure 14a). These values are among the highest of resins, along with those of Virginia juniper and yew (values of only 18.8 MJ/kg are identified in fir and spruce), all of these due to the high resin content of 2.8%–4.3% [42]. The average calorific value or energy of the other wood species is around 18.6 MJ/kg [42]. Also, based on these values and other values determined during combustion in the calorimetric bomb, a combustion speed of 760 kJ/min and a calorific density of 16.59 kJ/cm^3 was determined (Figure 14b) [42,43].



(**a**) (**b**)

Figure 14. Energy density (**a**) and calorific value (**b**) depend on the moisture content.

Breaking resistance of briquettes. The resistances were small, due to the fact that the entire section of the briquette was taken into account, respectively, A = d × l. Other authors did not take into account the cross-section of the briquette [43] or took into account only the deformed surface or their linearity (Figure 15).

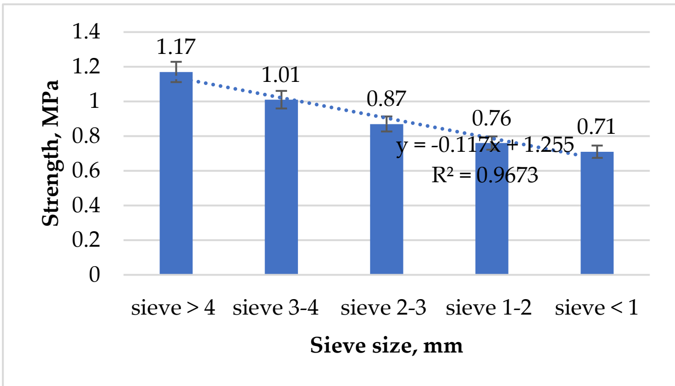


Figure 15. Breaking resistance of briquettes.

Abrasion of briquettes. This determination showed that the specimen was quite compact and do not crumble when rubbed on a sorting sieve with mesh dimensions of 4 mm (Figure 16).

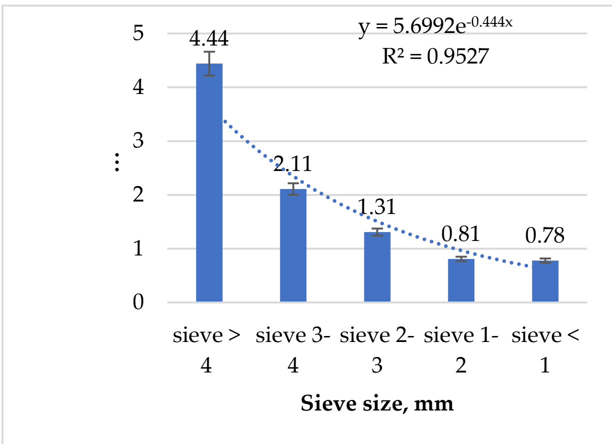


Figure 16. Abrasion of briquettes.

Ash content. The resulting value of the ash content was 0.55%, a good value considering that softwood species contain silicates and oxide (0.06% N, 0.42% P, 18% Ca, 0.97%, Mg and 2.27% K), which could easily increase the ash content. The explanation is given by the fact that a high content of resin was removed at the time of drying. Black ash with a value of 14.12% generally contains fine charcoal, after the volatile substances have been removed from the wood and the flame has disappeared (Figure 17).

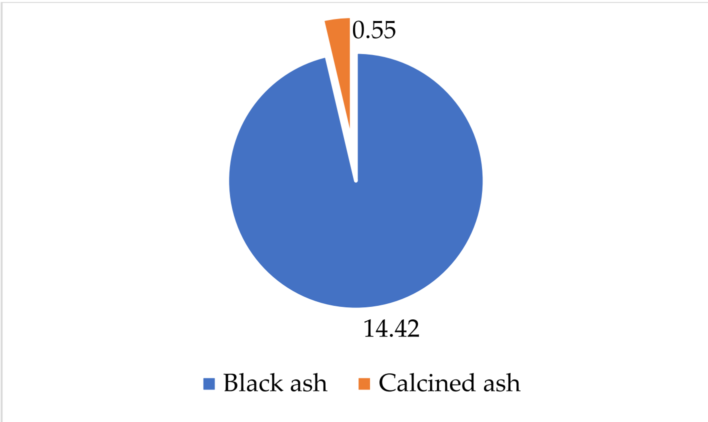


Figure 17. Content of calcinated and black ash of pine wood shavings.

4. Discussion

The first stage of the rheological curve (observed in Figure 8) was characterized by a linear increase in tension with increasing deformation. Deformation of the volume of the material occurs as a result of the movement of particles relative to each other, that is, the voids are filled. These aspects were identity to other studies [43]. The second stage of the rheological curve corresponds to a steep nonlinear increase in tension with a slight increase in deformation. At this stage, the movement of particles occurs due to accommodation (adjustment) between them and partly due to deformation of the volume of particles. This stage requires great effort to further deform the material. It is this stage that is of the greatest technological interest, since at this stage a certain plastic deformation accumulates in the material, providing strength [43]. For the third stage, with a significant increase in pressing pressure, the deformation practically does not increase.

For a more detailed analysis of the pressing process at the second stage of the rheological curve, the dependence of the compact pressure density (compression curves) was built. In other words, for the pressure range from 20 MPa to 200 MPa, the density values were obtained. When the maximum density was reached (the height of the compaction does not decrease, deformation does not occur), we moved on to the text composition.

It was established that the fraction $d < 1$ mm has the best compressibility indicators (maximum density $\rho = 1.03$ g/cm^3 at a pressure of 180 MPa). Other fractional compositions that were studied $1 < d < 2$ mm; $2 < d < 3$ mm; $d > 5$ mm have approximately the same indicators of compressibility ($\rho = 0.90$ g/cm^3 at P = 160 MPa; $\rho = 0.88$ g/cm^3 at P = 140 MPa; $\rho = 0.85$ g/cm^3 at P = 140 MPa). This is because smaller particles have a larger contact area with each other, which contributes to better adhesion and the formation of a denser compact structure. As a result, at the same pressing pressure, the fraction with a particle size lesser than 1 mm reaches higher density [43].

A further increase in compaction pressure above a certain level does not lead to an increase in compaction density. This may be due to the phenomenon of elastic relaxation after removal of the load. When the pressure is released, the compact can easily return to its original shape and increase in volume, limiting the possibility of increasing the density.

The increased strength of the fraction of shredded wood with a particle size of more than 4 mm can be associated with several factors [43]. First, large particles have a higher initial density compared to small particles, which can help strengthen the internal bonds and structure of the compact. Higher density can lead to increased resistance to mechanical forces, which contributes to increased strength. Second, a fraction with larger particles can provide a larger contact area between particles when forming a compact. This can contribute to better adhesion among particles and increase the mechanical strength of the compact. The combination of these factors leads to the fact that the fraction with larger particles can form a stronger and more stable structure inside the compact. This promotes better distribution and reduces the likelihood of failure when exposed to external forces.

Different percentages of granulometry explain why the density of briquettes obtained from different fractions is different, and the density of unfractionated wood shavings does not overlap the average density of all fractions. The characteristics shape of the curve has been identified in other specialized works in the field for willow or oak [43]. It was observed from Figure 8 that those smaller particles settle better on the surface of the cylinder, obtaining a higher density, but the maximum difference from the density of unfractionated wood shavings did not exceed 20.6%. A lower value of the density of wood shavings, lower than that of sawdust by about 200 kg/m^3 [43], was also observed and having smaller particles lays better and significantly increases the bulk density. The values of calorific power are among the highest of resins, along with those of Virginia juniper and yew (values of only 18.8 MJ/kg are identified in fir and spruce), all of these are due to the high resin content of 2.8%–4.3%. The average calorific value of the other wood species is in addition 18.6 MJ/kg [43].

Even if it is clearly seen that once the particles of the briquettes decrease, the percentage loss by friction also decreases, as observed in Figure 16, it is the exponential regression curve that best approximates (R^2 = 0.95) the variation in friction losses depending on the grain of the briquettes. It is observed, in this respect, that the regression curve flattens at small grain values, respectively, starting with the 1–2 mm sieve the decrease that is no longer as large (a decrease of 0.03%) as at the first two sieves (a decrease of 2.33%) [43].

Considering the complexity of the problem addressed, the authors intend to continue research in the field of this work, considering a multiaxial pressing of pine sawdust with different specifical pressures between 20 and 60 MPa. An objective is also to obtain briquettes from pine sawdust with mechanical briquetting installations, which would make it possible to obtain densities of over 1.000 kg/m^3.

5. Conclusions

The obtained results highlight the importance of fractional composition, compaction pressure, and compact resistance to optimizing the compaction process of the crumbled wood material. The best compressibility results (ρ = 1.03 g/cm^3 at P = 180 MPa) were obtained when using a fraction with a particle size d < 1 mm. The other studied fractions with particle sizes from 1 to 4 mm demonstrate approximately the same compressibility (ρ = 0.90 g/cm^3 at P = 160 MPa; ρ_{zag} = 0.88 g/cm^3 at P = 140 MPa; respective ρ = 0.85 g/cm^3 at P = 140 MPa). A further increase in pressure does not lead to an increase in compact density.

The methodology developed and applied to assess the strength of compacts by measuring the absolute loss of mass during their free fall from a height of 2 m showed that compacts with a particle size fraction d > 4 mm have the highest resistance to fall, which allows us a fundamental understanding of the influence of the particle size of shredded wood on the strength characteristics of compacted parts.

It was established that fractions with particle sizes less than 1 mm had the best compacity parameters and achieved high densities at low pressures (ρ = 1.03 g/cm^3 at pressure of 180 MPa). The other fractions that were studied had the same compacity and density ρ in an interval of 0.85–0.90 g/cm^3 at a pressure of 140–160 MPa. A greater increase in pressing did not lead to a density increase due to elastic deformation after removal of the load. The greater strength of compacts was determined for a fraction more than 4 mm in size fell is used.

All the results obtained by this kind of research could be used to optimize the process of pressing shredded wood and develop more efficient methods for using wooden resources.

An optimum particle size of 2–3 mm was found to correspond to an acceptable density of 866.5 kg/m^3 and an acceptable resistance to compression breakage of 0.87 MPa.

The degree of expansion of the briquettes was found after their exit from the extrusion channel with a rheological level of briquettes at 34%.

We found a degree of compaction of the structure in relation to the bulk density of the wood shavings of 7.48% and 1.34.% in comparison to the density of the pine, respectively, a very good compaction–compression reserve of 40.1% in relation to the density of the wood substance of this wood species.

Increasing the wooden density of briquettes by 40.1% compared to pine wood represents a major advantage in the subsequent uses of these wood briquettes, broadening the base of their uses.

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References

1. Griu, T. Evaluation of the Calorific Value of Woody Biomas. Ph.D. Thesis, Universitatea Transilvania din Brasov, Brasov, Romania, 2014. (In Romanian)
2. Krajnic, N. *Wood Fuel Handbook*; Food and Agriculture Organization (FAO) of the United Nations: Pristina, Kosovo, 2015.
3. Prasertsan, S.; Sajakulnukit, B. Biomass and Bioenergy in Thailand: Potential, Opportunity and Barriers. *Renew. Energ.* **2006**, *31*, 599–610. [CrossRef]
4. Demirbas, A. Biomass Resource Facilities and Biomass Conversion Processing for Fuels and Chemicals. *Energ. Convers. Manag.* **2001**, *42*, 1357–1378. [CrossRef]
5. Demirbas, A.; Demirbas, A.S. Briquetting Properties of Biomass Waste Materials. *Energ. Source Part A* **2004**, *26*, 83–91. [CrossRef]
6. Spirchez, C.; Lunguesa, A.; Matei, M. Particularities of hollow-core briquettes obtained out of spruce and oak wooden waste. *Maderas Cienc. Tecnol.* **2018**, *20*, 139–152. [CrossRef]
7. Ramírez-Ramírez, M.A.; Carrillo-Parra, A.; Ruíz-Aquino, F.; Pintor-Ibarra, L.F.; González-Ortega, N.; Orihuela-Equihua, R.; Carrillo-Ávila, N.; Luján-Álvarez, C.; Rutiaga-Quinones, J.G. Valorization of briquettes fuel using *Pinus* spp. sawdust from five regions of Mexico. *BioResources* **2021**, *16*, 2249–2263. [CrossRef]
8. Borowsky, G. Possibilities of Utilisation of Energy Briquettes. *Electr. Enginering Res. Rep.* **2011**, *1*, 48–51. Available online: http://eeeic.org/proc/papers/74.pdf (accessed on 2 December 2024).
9. Marreiro, H.M.P.; Peruchi, R.S.; Lopes, R.M.B.P.; Andersen, S.L.F.; Eliziário, S.A.; Junior, P.R. Empirical Studies on Biomass Briquette Production: A Literature Review. *Energies* **2021**, *14*, 8320. [CrossRef]
10. Lundborg, A. A Sustainable Forest Fuel System in Sweden. *Biomass Bioenerg.* **1998**, *15*, 399–406. [CrossRef]
11. Lakó, J.; Hancsók, J.; Yuzhakova, T.; Marton, G.; Utasi, A.; Rédey, A. Biomass—A Source of Chemicals and Energy for Sustainable Development. *Environ. Eng. Manag. J.* **2008**, *7*, 499–509. Available online: http://www.eemj.icpm.tuiasi.ro/pdfs/vol7/no5/2_Lako_J.pdf (accessed on 7 December 2024). [CrossRef]
12. FAO. FAO: State of the World's Forests 2024. Available online: https://www.donorplatform.org/post/fao-state-of-the-worlds-forests-2024/ (accessed on 9 December 2024).

13. Šturc, M. Eurostat, Statistics in Focus 44/2012. Environment and Energy. Available online: https://ec.europa.eu/eurostat/web/products-statistics-in-focus/-/ks-sf-12-044 (accessed on 12 December 2024).
14. European Commission (EC). Energy for the Future: Renewable Sources of Energy. White Paper for a Community Strategy and Action Plan, COM (97)599 Final (26/11/1997). Available online: http://ec.europa.eu/energy/library/599fi_en.pdf (accessed on 17 November 2024).
15. Plepys, A.; Boutin, J.-P.; Gervasoni, G.; Hlep, R.; Seyboth, K.; Lamers, P.; Ratton, M.; McCormick, K.; Mundaca, L. Alternative Energy Sources in Transition Countries. The Case of Bio-energy in Ukraine. *Environ. Eng. Manag. J.* **2007**, *6*, 3–11. Available online: http://www.eemj.icpm.tuiasi.ro/pdfs/vol6/no1/2_Jean-Philippe%20Boutin.pdf (accessed on 16 December 2024). [CrossRef]
16. Kim, S.; Dale, B.E. Cumulative Energy and Global Warming Impact from the Production of Biomass for Biobased Products. *J. Ind. Ecol.* **2003**, *7*, 147–162. Available online: http://arrahman29.files.wordpress.com/2008/02/lca-use.pdf (accessed on 2 May 2012). [CrossRef]
17. Junginger, M.; Bolkesjø, T.; Bradley, D.; Dolzan, P.; Faaij, A.; Heinimö, J.; Hektor, B.; Leistad, Ø.; Ling, E.; Perry, M.; et al. Developments in International Bioenergy Trade. *Biomass Bioenergy* **2008**, *32*, 717–729. [CrossRef]
18. Verma, V.K.; Bram, S.; de Ruyck, J. Small Scale Biomass Systems: Standards, Quality Labeling and Market Driving Factors—An EU Outlook. *Biomass Bioenergy* **2009**, *33*, 1393–1402. [CrossRef]
19. Roser, D.; Asikainen, A. *Sustainable Use for Forest Biomass for Energy*; Springer Series in Wood Science; Springer: Berlin/Heidelberg, Germany, 2006.
20. Okello, C.; Pindozzi, S.; Faugno, S.; Boccia, L. Bioenergy potential of agricultural and forest residues in Uganda. *Biomass Bioenergy* **2013**, *56*, 515–525. [CrossRef]
21. Aebiom (European Biomass Association). Wood Fuels Handbook. Available online: https://www.lemvigbiogas.com/BiogasHandbook.pdf (accessed on 18 December 2024).
22. Kostanaki, E.; Vamvuka, D. A comparative reactivity and kinetic study on the combustion of coal-biomass char blends. *Fuel* **2006**, *85*, 1186–1193. [CrossRef]
23. Petrovici, V.; Popa, N. *Chemistry and Chemical Wood Processing*; Lux Libris Print House: Brasov, Romania, 1997. (In Romanian)
24. *DIN 51731 [1996]*; Testing of Solid Fuels—Compressed and Untreated Wood—Requirements and Testing. German Institute for Standardization: Berlin, Germany, 1996.
25. *EN ISO 17225-1: 2014 [2014]*; Solid Biofuels—Fuel Specifications and Classes—Part 1: General Requirements. European Committee of Standardization: Brussels, Belgium, 2014.
26. Equilabo, Catalogue 1200. Parr. Oxygen Bomb Calorimeters and Accessories. Available online: https://www.parrinst.com/products/oxygen-bomb-calorimeters/ (accessed on 8 July 2012).
27. *EN 14774-1:2009*; Solid Biofuels-Determination of Moisture Content-Oven Dry Method-Part 1: Total Moisture-Reference Method. European Committee of Standardization: Brussels, Belgium, 2009.
28. Hahn, B. Existing Guidelines and Quality Assurance for Fuel Pellets. Pellets for Europe 2004, Umbera. Available online: http://www.pelletcentre.info/resources/1020.pdf (accessed on 18 December 2024).
29. *ÖNORM M 7135 [2000]*; Pellets and Briquettes—Requirements and Test Conditions. Austria Standards Institute: Vienna, Austria; European Committee of Standardization: Brussels, Belgium, 2000.
30. Gocławski, J.; Korzeniewska, E.; Sekulska-Nalewajko, J.; Kiełbasa, P.; Dróżdż, T. Method of Biomass Discrimination for Fast Assessment of Calorific Value. *Energies* **2022**, *15*, 2514. [CrossRef]
31. *DIN 51900-1*; Determining the Gross Calorific Value of Solid and Liquid Fuels Using the Bomb Calorimeter and Calculation of Net Calorific Value. Part 1: General Information. German National Standard: Berlin, Germany, 2000.
32. Nurek, T.; Gendek, A.; Dąbrowska, M. Influence of the Die Height on the Density of the Briquette Produced from Shredded Logging Residues. *Materials* **2021**, *14*, 3698. [CrossRef]
33. *ISO 1928:2009*; Solid Mineral Fuels. Determining the Gross Calorific Power by Calorimeter Bomb and Calculus of Net Calorific Power. International Organization for Standardization: Geneva, Switzerland, 2009.
34. *ASTM E1755-01*; Standard Method for the Determination of Ash in Biomass. American Society for Testing and Materials, International: Philadelphia, PA, USA, 2000.
35. Kers, J.; Kulu, P.; Aruniit, A.; Laurmaa, V.; Križan, P.; Šooš, L.; Kask, Ü. Determination of physical, mechanical and burning characteristics of polymeric waste material briquettes. *Est. J. Eng.* **2013**, *19*, 307–316. Available online: http://www.kirj.ee/18098/?tpl=1061&c_tpl=1064 (accessed on 18 December 2024). [CrossRef]
36. 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. Bioenerg.* **2011**, *35*, 910–918. [CrossRef]
37. Mitchual, S.J.; Frimpong-Mensah, K.; Darkwa, N.A. Effect of Species, Particle Size and Compacting Pressure on Relaxed Density and Compressive Strength of Fuel Briquettes. *Int. J. Energy Environ. Eng.* **2013**, *4*, 30. [CrossRef]
38. Sola, O.C.; Atis, C.D. The Effects of Pyrite Ash on the Compressive Strength Properties of Briquettes. *KSCE J. Civ. Eng.* **2012**, *16*, 1225–1229. [CrossRef]

39. Plištil, D.; Brožek, M.; Malaták, J.; Roy, A.; Hutla, P. Mechanical Characteristics of Standard Fuel Briquettes on Biomass Basis. *Res. Agric. Eng.* **2005**, *51*, 66–72. Available online: http://agriculturejournals.cz/publicFiles/57241.pdf (accessed on 18 December 2024). [CrossRef]
40. Kaliyan, N.; Morey, R.V. Factors Affecting Strength and Durability of Densified Biomass Products. *Biomass. Bioenergy* **2009**, *33*, 337–359. [CrossRef]
41. Rahman, A.N.E.; Masood, M.A.; Prasad, C.S.N.; Venkatesham, M. Influence of Size and Shape on the Strength of Briquettes. *Fuel Process Technol.* **1989**, *23*, 185–195. [CrossRef]
42. Tabarés, J.L.M.; Ortiz, L.; Granada, E.; Viar, F.P. Feasibility Study of Energy Use for Densificated Lignocellulosic Material (briquettes). *Fuel* **2000**, *79*, 1229–1237. [CrossRef]
43. Mikheev, M.V.; Komina, A.V.; Gorbacheva, G.A.; Kalinina, A.A.; Bolotskaya, A.V.; Sanaev, V.G.; Stolin, A.M. Study of disintegrated pine wood rheological behavior under uni-axial compressing]. *Lesn. Vestn. For. Bull.* **2024**, *28*, 116–126.