Torrefaction of Spruce, Beech, and Oak Pellets in Order to Improve Calorific Value

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Native spruce, beech, and oak pellets were treated by torrefaction. The torrefaction was performed without oxygen, in an electric oven, at temperatures of 170 °C, 190 °C, and 210 °C and treatment periods of 1, 2, and 3 h. The results showed an increase in the calorific value of the pellets by raising the parameters of the treatment regime (temperature and duration), achieving maximum values of 18.93 MJ/kg in the case of beech pellets for 3 h and 190 °C, 18.97 MJ/kg for the roughest regime with 3 h and 210 °C in the case of spruce pellets, and 18.84 MJ/kg in the case of oak pellets with 3 h and 190 °C. The torrefaction process for beech and oak must be stopped at 190 °C and 3 hours, becoming ineffective after these values of the treatment regime. The shear strength of the pellets were decreased slightly (10 to 15%) by the torrefaction treatment, but ash content slightly increased from 17.7% for spruce pellets to 29.1% for beech pellets. The results suggest that direct torrefaction of native pellets is possible without the use of nitrogen, *i.e.*, by turning off the oxygen intake in the torrefaction oven, with good results for increasing the calorific value with 8.7% for beech pellets, 7.5% for spruce pellets, and 7.9% for oak pellets.

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INTRODUCTION

Wood pellets are considered renewable fuels because they are obtained by compacting fine wood material, usually sawdust. Pellets are made also using remnants from the furniture, veneer, plywood, and flooring industries, making them more renewable and environmentally friendly (CEN/TC 335 2004; CTI-R 04/5 2004; EN 14961-1 2010). The process of compression and compaction through extrusion of the small particles makes these pellets have high densities, over 1000 kg/m^3, which corresponds to a wood compression degree between 70 and 180%. Trnka *et al*. (2021) noted that the alternative biomass represents a significant part of biomass processing waste in forestry and agriculture. Other research proposed to create pellets from alternative biomass sources (grass chips, corn husks, lime leaves, and pine needles) (Toscano *et al*. 2013). The properties of these pellets were compared with those of spruce wood pellets. Most analyses of fuel properties focus on chemical composition, thermogravimetric analysis, volatile compounds, calorific value, and ash melting temperature (Chen *et al.* 2011). The problems in burning alternative types of biomasses are low calorific value, high gas emissions, high ash content, and low ash melting temperatures (Demirbas 2001; Demirbas and Demirbas

2004). The two main options for solving these problems are the production of wood fuel blends or the use of new combustion technologies (Boutin *et al*. 2007).

Usually, the pellet compaction of shredded material does not use adhesive or additives, but when the particles are heated to temperatures of 80 to 100 °C, the lignin in the lignocellulosic material is activated (Horvat *et al.* 2021). The small pellet diameter of about 6 mm obliges all manufacturers of wood-based thermal power plants to include in their equipment specific fireplaces for burning lignocellulosic pellets (Mitchual *et al.* 2013). Pellets are fairly inexpensive products that can be used in developing, medium, and highly developed countries.

The use of pellets in combustion promotes “green energy” and reduces the noxious emissions released into the atmosphere by fossil fuels (Cardozo *et al.* 2014; Arranz *et al.* 2021). Even though the price of wood pellets is double that of briquettes, their major advantages (they are renewable, decrease dependence on fossil fuels, don't degrade during storage, *etc.*) make them increasingly used for heating homes, but also for generating electricity from renewable sources (Dhillon and von Wuelhlisch 2013). Their major disadvantage is the susceptibility to moisture, which is why they are always stored, delivered, and transported wrapped (Plištil *et al*. 2005).

The pellet/sawdust torrefaction process is conditioned by the presence of an inert medium (usually nitrogen) during the heat treatment, with elimination of the possibility of self-ignition of the lignocellulosic material (Junginger *et al*. 2008). Torrefied pellets are usually obtained from torrefied sawdust, which is subsequently compacted (Jehlickova and Morris 2007). However, torrefied sawdust is flaky and does not compact as well as native sawdust, which is why natural ingredients such as molasses, glucose, adhesive extracted from resin cones, *etc*., must be added. Direct pellet torrefaction is a simple solution, but not currently used. Bridgeman *et al.* (2008) noted that torrefaction improves biomass properties relative to thermochemical processing techniques for energy generation (combustion, gasification). This study examines nitrogen torrefaction of 2 energy crops, feather reed grass and short rotation coppice willows. It was found that the properties of biomass fuel resemble those of coal (Omer 2012). The results indicate that the volatile component of the biomass is decreased, producing a thermally stable product (Kažimírová *et al*. 2013). The difference between mass and energy yield improved under the higher torrefaction temperatures that was also investigated (Tabarés *et al.* 2000).

Pellets have important dimensional, physical, chemical, and mechanical properties (Lu *et al.* 2014). Their diameter is usually 6 or 8 mm, and the length should not exceed 5 times the thickness, according to European standards (CEN/TC 335:2004; ISO 1928:2009; ASTM D3865-12:2000; EN 14916-2:2010). With complete combustion of pellets (as other type of fuels haven’t), there are significant reductions in CO pollutant emissions, namely higher than 40% for wood pellets and higher than 30% for agricultural crop pellets. These results improve the combustion efficiency by 2.6 to 3.7% (Horvat *et al.* 2021).

Kaliyan and Morey (2009) stated that 2 kg of pellets, 2.5 kg of firewood, 12.5 m^3 natural gas, or 3 L of oil should be used to obtain 10 kWh of thermal energy. They pointed out the advantages and disadvantages of conventional and torrefied pellets. Torrefaction is defined as a pyrolysis process, which is the process of heating without access to air and is produced at temperatures of 220 to 330 °C. Partial decomposition of cellulose and lignin in wood pellets decreased its weight by 20 to 30%, and increased its energy density by 10 to 15%, leading to a substantial increase in the specific heat of combustion (Kazagic and Smajevic 2009). Based on temperature and process duration, the combustion heat increases from 14 to 17 MJ/kg to 19 to 24 MJ/kg. The torrefied pellets acquired high hydrophobicity and resistance to atmospheric factors. These properties solved also the problems of transportation and storage.

Nielsen *et al.* (2009) studied pellets of the following forest species *A. xalapensis, J. deppeana*, and *Q. sideroxyla* with very good value of calorific power. Shear strength of the pellet was investigated by the same authors with the use of ASTM D 6128-0615:2006 standard (Czachor *et al.* 2016).

The vast majority of research uses primary torrefaction of sawdust in nitrogen medium and subsequent the compaction into pellets. Because of several shortcomings of sawdust torrefaction (low sawdust compaction, high price due to nitrogen use in reactors), the present research aimed to direct torrefaction of native pellets using a low-oxygen torrefaction medium, inside of oven with the air damper closed. Three types of pellets from different species were analyzed to observe their reaction during torrefaction.

EXPERIMENTAL

Three types of industrial pellets were made of beech (*Fagus sylvatica* L.), spruce (*Picea abies* Karst.), and oak (*Quercus robur* L.), taken from the market (Brico depot Brasov, Romania), by the same production company, so that there are no differences in consistency, compaction, *etc*., without the use of additives or other adhesives substances (EC 1997; Eurostat 2011).

The moisture content of the pellets was determined gravimetrically, by weighing-drying-weighing, using a precision balance with 2 decimal (Kern, China) and an electric furnace of Memmert type (Munich, Germany), which ensured a temperature of 105 °C. After obtaining the constant mass, the absolute moisture content of the pellets was determined with Eq. 1,

$$Mc = \frac{m_u - m_0}{m_0} \cdot 100 [\%] \tag{1}$$

where Mc is the moisture content, m_u is the mass of pellets before drying (g), and m_0 is the mass of pellets after drying (g). Ten specimens were used to obtain the average moisture content.

For determining the density of each individual pellet, the pellet ends were ground with an abrasive disc to obtain straight circular cylinders (Kim and Dale 2003). By determining the mass and volume of each pellet, the determination relationship for the individual pellets was as follows,

$$D_u = \frac{4 \cdot m}{\pi \cdot d^2 \cdot l} \cdot 10^6 \ [\frac{kg}{m^3}] \tag{2}$$

where D_u is the unit density of the pellets ($kg \cdot m^{-3}$), m is mass of the pellets (g), d is diameter of the pellet (mm), and l is length of the pellet (mm). For this determination, 20 individual pellets were used.

Torrefaction Process

The heat treatment process was carried out in a Memmert electronic furnace (Munich, Germany) at temperatures of 170 °C, 190 °C, and 210 °C and periods of 1, 2, and 3 h. Before being placed in the furnace, the pellets were dried to remove moisture content from them, at 105 °C for 2 h, after which they were weighed to 2 decimal precisions (Griu 2014; Arranz *et al.* 2021*)*. During the torrefaction the pellets were kept in a temperature resistant crucible (Van Dam *et al.* 2008; Sola and Atis 2012). After treatment they were cooled in a desiccator and weighed again (Kers *et al*. 2013). The mass loss was determined as the main factor in the evaluation of the torrefaction, with (Eq. 3),

$$ML = \frac{m_f - m_i}{m_i} \cdot 100[\%] \tag{3}$$

where ML is the mass loss (%), m_f is final mass of dry pellet (g), and m_i is initial mass of pellet, after torrefaction (g). Ten pellets of each type were tested.

Calorific Value

The calorific value of native/torrefied pellets was determined using an adiabatic calorimeter (XRY-1C oxygen bomb calorimeter, Shanghai Geological, China) on the base of DIN 51900-1:2000. Before of the one set of tests, the calorimeter was calibrated with benzoic acid, having a known calorific value of 26,454 kJ/kg, in order to determine the calorimetric coefficient k, which was then used by the calorimetric software for determining the calorific value with Eq. 4,

$$CV = \frac{k \cdot (T_f - T_i) - Q_s}{m} \quad [\frac{kJ}{kg}] \tag{4}$$

where k is coefficient of the calorimetry (kJ/ ºC), T_f is final temperature during the test (ºC), T_i is initial temperature during test (ºC), Q_s is additional heat achieved by burning the nickel wire and the cotton thread (kJ), and m is mass of the sample pellet, usually around 0.8 g. Ten pellets of each type were tested.

Ash Content

The calcined ash content was determined in accordance with ASTM D 1102-84:2013 and EN 14775:2010 by grinding the pellets and taking the fraction that passed through the 1×1 mm sieve. The dust obtained was first dried to constant mass in order to eliminate the influence of moisture on the ash content. The calcination was conducted by placing the dust crucibles in an electric furnace of ProTherm type (Ploiesti, Romania) at a temperature of 650 °C for at least 1.5 h. The calcination was finished when no traces of carbon or sparks were visible on the crucible in the furnace (Shang *et al.* 2012). The calculation relationship was based on the masses (measured to 3 decimal accuracy) of dust/ash in the crucible at the start and end of the test, as follows,

$$A_c = \frac{m_{c+p} - m_{c+a}}{m_{c+p}} \cdot 100\ [\%] \tag{5}$$

where m_{c+p} is the mass of sample plus mass of crucible (g), and m_{c+a} is mass of ash plus mass of crucible (g). Ten valid samples were considered.

Shear Strength

Among the mechanical properties, the shear strength was determined using a laboratory methodology (Rahman *et al*. 1989; Mark *et al*. 2006; Stelte *et al*. 2011). Primarily, a 6-mm thick steel plate was used, which had 5 holes of 6 mm diameter, into which 5 pellets were inserted. The second shear plate had a tip with 85° angle of sharpness, with a view to eliminating the possibility of cutting the pellets. The two plates were inserted between the jaws of the universal testing machine (Zwick, Ulm, Germany) in such a way as to create a central shear force. The calculation relation used to determine the shear strength of the pellets was as follows,

$$\tau_p = \frac{F_{max}}{5\cdot\pi\cdot d^2}\ \left[\frac{N}{mm^2}\right] \qquad (6)$$

where τ_p is the shear strength (N/mm^2), F_{max} is maximum shear strength (N), and d is the diameter of pellets, around 6 mm. Ten shear tests were used for each pellet type.

Increase/Decrease of Properties

The increase/decrease in the value of a given property was obtained using the following general relationship (Eq. 7),

$$Ip/Dp = \frac{V_{max}-V_{min}}{V_i}\cdot\ 100\ [\%] \qquad (7)$$

where Ip/Dp is increase/decrease of the property (%), V_{max} is maximum value of the property, V_{min} is minimum value of the property; and V_{i} is initial value of the property, usually the minimum in case of increase of the property.

Statistical Analysis

The results of the research were statistically processed, mainly by obtaining the survey median and standard deviation. The standard deviation was applied to the graphs made in MS Excel. Statistical graphs were also created, using the Minitab 18 program, for a 95% confidence interval and an alpha error of 0.05.

RESULTS AND DISCUSSION

Unit Density of Native Pellets

The density of native pellets without torrefaction was 1.33 g/cm^3 for spruce and was the highest one, 17.6% higher than that of beech pellets and 14.6% higher than that of oak pellets (Fig. 1). The cumulative distribution function (CDF) of unit density, achieved with the Minitab 18 program, obtained for the 20 test specimens, gives a standard deviation of 0.037 g/cm^3 for beech pellets, 0.038 g/cm^3 for spruce pellets, and 0.023 g/cm^3 for oak pellets.

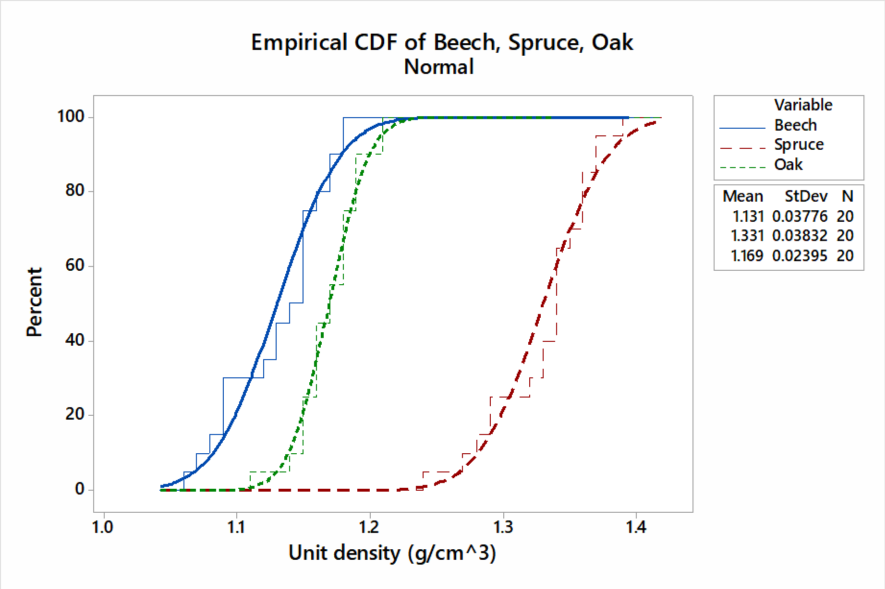

Fig. 1. Empirical CDF of unit density (expressed in g/cm^3) for all wood pellets type

These standard deviation values (Fig. 1) determined a range of 1.055 to 1.206 g/cm^3 in the case of beech pellets, of 1.254 to 1.407 g/cm^3 in the case of spruce pellets and 1.121 to 1.206 g/cm^3 in the case of oak pellets for the 95% confidence interval. The very high differences between densities may be related to the different structure of the wood species, the soft species being the most compressible.

Calorific Value for Native (Non-torrefied) Pellets

The calorific value of pellets differs between species, depending on their structural and chemical characteristics. The torrefaction process of wooden pellets increases the calorific properties close to inferior coal (Yeniocak *et al.* 2014). The thermal torrefaction process contributes to the increasing of the lignin content and degrades hemicelluloses, lignin, and cellulose in the wooden torrefied pellets (Obernberger and Thek 2004; Chen *et al.* 2012). Therefore, spruce pellets had a maximum calorific value HCV of 17.6 MJ/kg (Fig. 2) because of the higher cellulose content than that of deciduous species. The calorific value of spruce pellets is also explained by the existence of a certain resin content, resin having a very high calorific value of about 34.6 MJ/kg (Okello *et al*. 2013). Regarding the higher heating value, spruce pellets had a value 12.8% higher than that of beech pellets and only 0.5% higher than that of oak pellets. Moreover, the difference between the upper and lower calorific values were much higher for oak pellets than for the other two pellet categories, this being based on the structural differences of the three wood species.

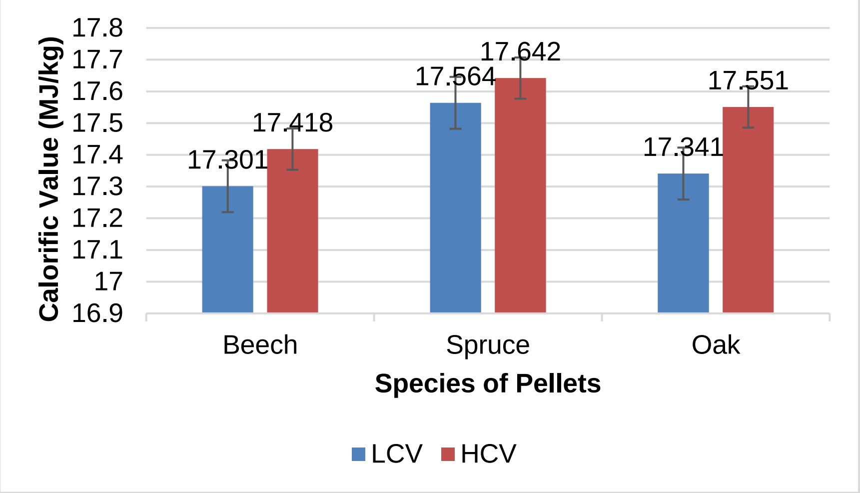

Fig. 2. Calorific value of native pellets: LCV-low calorific value, HCV- high calorific value

Calorific Value of Pellets During Torrefaction

Generally, during torrefaction the calorific value of pellets increased proportionally to the heat treatment regime (temperature and heat time) but differed between wood species (Bridgeman *et al.* 2008). Hence, in the case of beech pellets, the increase in calorific value from the native to the maximum torrefaction state was 8.7%; in the case of spruce pellets it was 7.5%; and in the case of oak pellets it was 7.9% (Fig. 3). It can be seen that the deciduous species (beech and oak) were torrefied more strongly than the larch. Furthermore, the optimal treatment temperature of the pellets of these wood species was 190 °C and the treatment time of 3 h.

If a sequential analysis is made for each pellet type and depending on the heat treatment regime, it can be noted that spruce pellets had the most homogeneous torrefaction process, the calorific value increasing constantly from a lower to a higher torrefaction step. In the case of beech pellets the maximum heating value of 18.934 MJ/kg was achieved in the second temperature step and for a duration of 3 h, and in the case of oak pellets the maximum value of 18.9 MJ/kg was achieved for the same treatment regime.

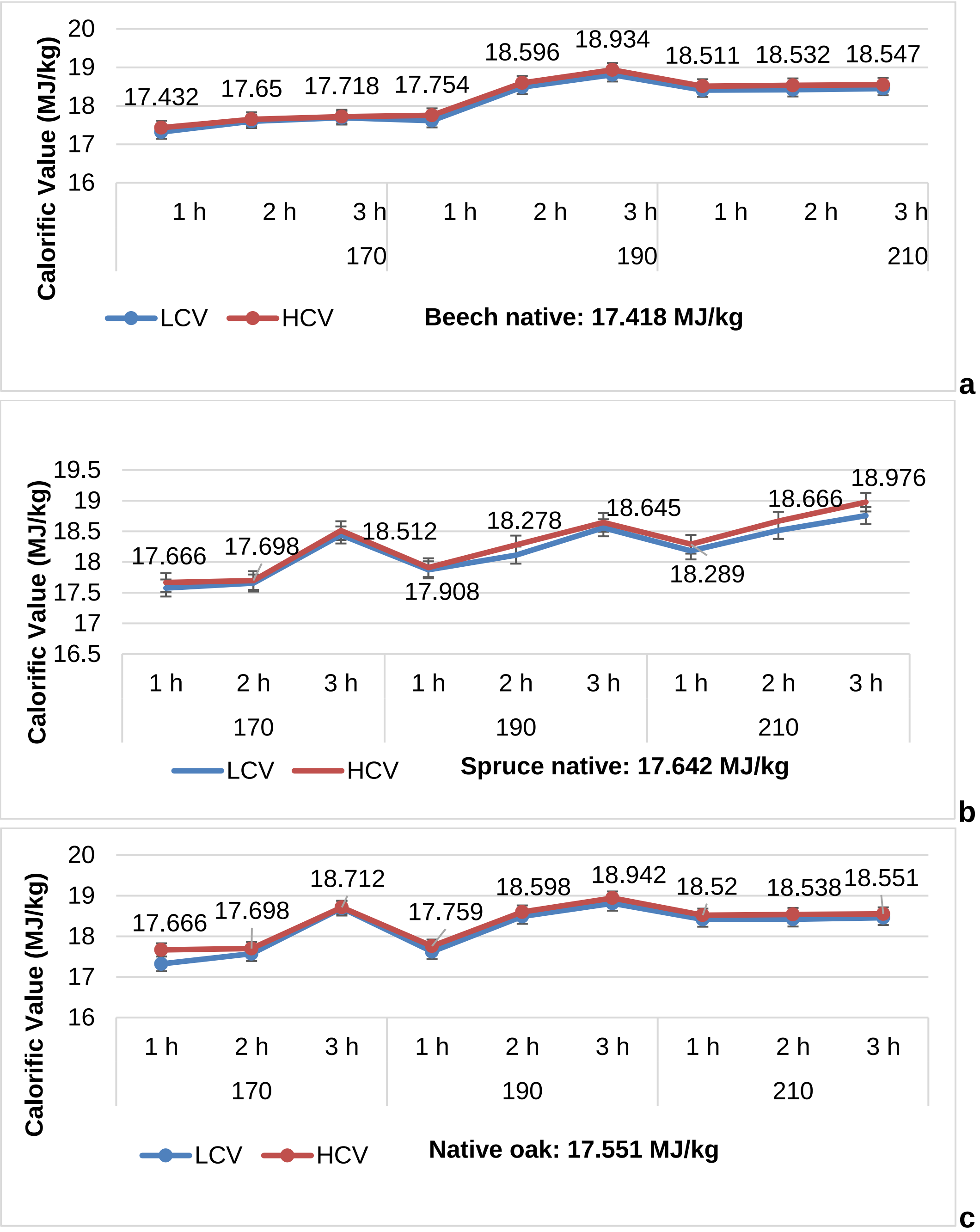

Fig. 3. Calorific value of pellets during torrefaction: a-beech pellets; b-larch pellets; c- oak pellets; LCV-low calorific value; HCV-high calorific value

Sequentially for each temperature step, the following were noted:

- In the case of beech pellets, in the temperature step of 170 °C, the increase was 1.7%, at temperatures of 190 °C the increase was 8.7%, and at temperatures of 210 °C the increase was 6.4%;
- In the case of spruce pellets in the temperature step of 170 °C the increase was 4.9%, at temperatures of 190 °C the increase was 5.6%, and at temperatures of 210 °C the increase was 7.5%;
- In the case of oak pellets in the temperature step of 170 °C the increase was 6.6%, at temperatures of 190 °C the increase was 7.9% and at temperatures of 210 °C the increase was 5.6%.

It is observed that the beech and oak pellets had a maximum increase in calorific value to 190 °C and 3 h of torrefaction, which means that the torrefaction of these pellets above this temperature is no longer justified and must be stopped. After this value of the torrefaction regime, the calorific value decreased slightly, by 2.04% in the case of beech and by 2.06% in the case of oak. Another conclusion derived from the above sentences is that the spruce pellets (softwood species) allow higher torrefying temperatures of 210 °C, while the other categories of oak and beech pellets (deciduous species) allow lower temperatures of maximum 190 °C.

Verna *et al.* (2009) notes that torrefaction is one of the pretreatment processes used to overcome the disadvantages of using biomass as a fuel such as low energy density, high moisture, and oxygen content. The same authors highlight in the article that pellet torrefaction can be conducted at temperatures of 220 °C and 290 °C for 30 min. Increasing the torrefaction temperature resulted in increased hydrophobicity but also decreased density, hardness, and durability of the pellets. The mass loss during torrefaction was due to the degradation of hemicellulose and cellulose, and the positive influence of lignin (Wannapeera *et al.* 2011).

Shear Strength

The shear strength values of native pellets are shown in Fig. 4. The maximum shear strength value was found for spruce pellets, beech pellets being 41.1% weaker and oak pellets with 31.3% weaker. A higher variation of values was noticed in the case of beech pellets, which is also visible in Fig. 4.

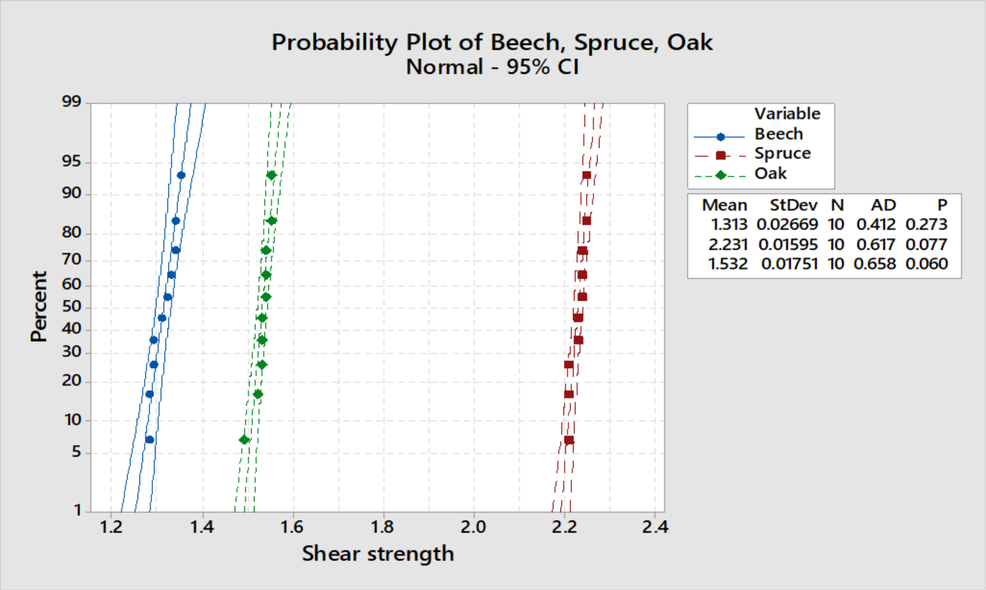

Fig. 4. Probability plot of shear strength (expressed in N/mm^2) for three wood pellets type

Moreover, by determining the variation of values for a 95% confidence interval (1.25 to 1.36 N/mm^2 for beech pellets, 2.23 to 2.26 N/mm^2 for spruce pellets, and 1.51 to 1.56 N/mm^2 for oak pellets), the maximum variation of strength for beech pellets and the minimum for spruce pellets is observed. By evaluating the Anderson-Darling coefficients and p-value (Fig. 4), the normality of the distribution of the three groups of values is demonstrated, which is also visible on the graphs in the Fig. 4.

The shear strength of the pellets decreased in time of torrefaction by 10 to 15%, but all torrefied pellets remained unitary and did not disintegrate. Larger differences in resistance were observed at spruce pellets (12 to 15%) and small differences in the case of beech and oak pellets (10 to 13%).

Dependence between Density and Shear Strength

As for both solid wood and other wood-based products such as fiberboards and wood chips, there is a direct correlation between density and strengths. A similar correlation between these properties was sought for the analyzed pellets. Figure 5 shows this correlation.

There is a certain correlation between the two properties through the two linear regression equations, which are almost parallel, the difference between the angles being only a few (2 to 3) sexagesimal degrees. This demonstrates once again that the properties of the pellets were in direct correlation, and that by increasing their density will lead to an proportionally increase in shear strength.

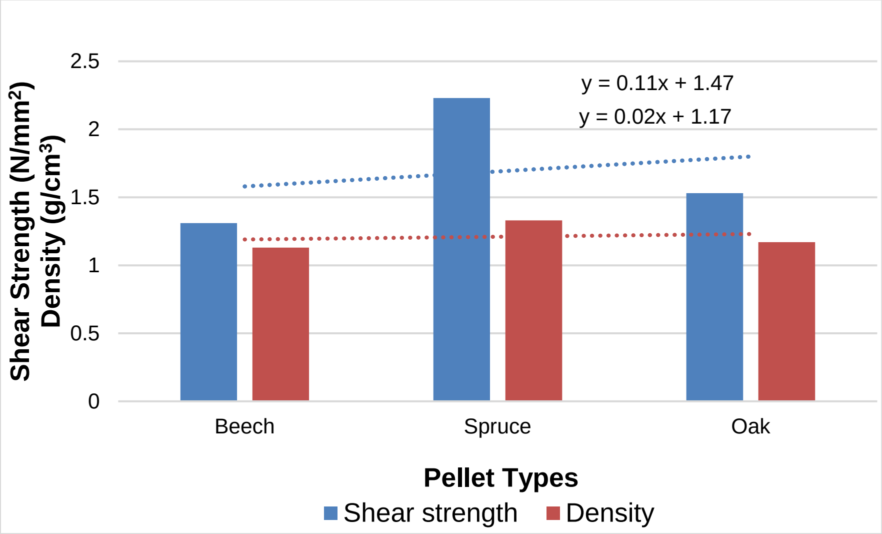

Fig. 5. Correlation between pellet density and shear strength

Ash Content Results

The ash content was differentiated by wood species and heat treatment regimes. In general, the ash content of the pellets corresponded to that of the analyzed species wood, as the pellets did not contain additional additives. As shown in Fig. 6, the ash content increased slightly during the heat treatment of roasting, the harsher the treatment, the higher the ash content. This is explained by the elimination of volatile substances from pellets, by the degradation of hemicelluloses and celluloses, but the intact of the secondary chemical compounds of wood from pellets (Griu 2014).

The average values of the calcined ash content of the native pellets were below 1%, lower for oak and beech, and higher for spruce. The increases of the ash content for the torrefied pellets with maximum regime were of 17.5% for spruce, of 20% for oak, and of 29.1% for beech.

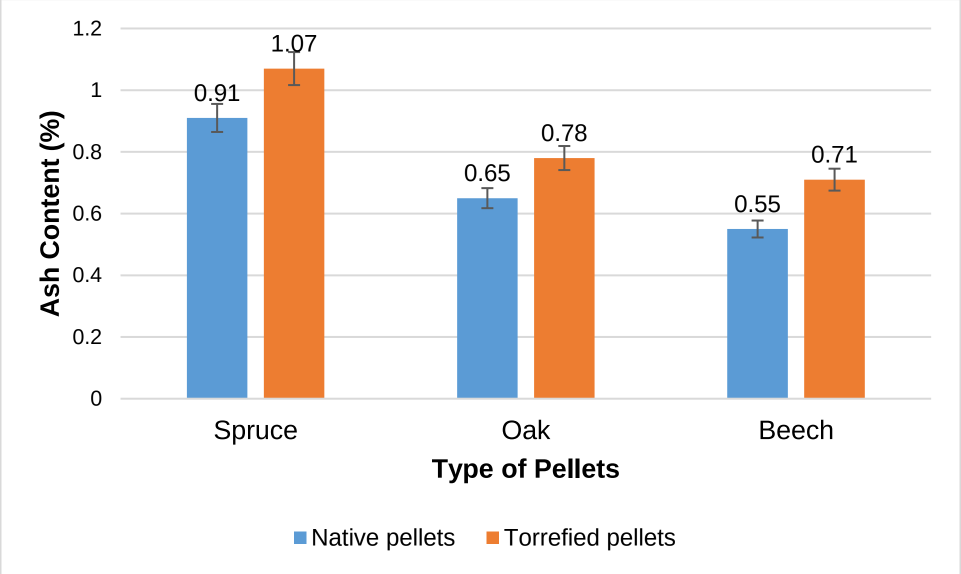

Fig. 6. Ash content of native and torrefied (3 h and 210 °C) pellets

This slight increase in the ash content for torrefied pellets was due to the elimination of some volatile substances during torrefaction, respectively the decrease of the denominator in the relationship (5) for determining the ash content.

CONCLUSIONS

1. Through whole research, it has been shown that the torrefaction process can be applied to the wooden pellets, in furnaces/oven without oxygen, with the main reason of calorific value increase.
2. Pellet density was higher for spruce pellets than for beech and oak pellets, demonstrating that this species is much more compressible than the others.
3. The calorific value increased during the pellet torrefaction treatment, as a result of the carbon enrichment of the pellets, obtaining maximum increase of 8.7% for beech, 7.5% for spruce and 7.9% for oak.
6. The shear strength decreased slightly following the torrefaction process. The torrefied pellets remained compact and with good strengths.

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