Article
Variability of Normative Properties of Wood Chips and Implications to Quality Control

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Abstract: The research was conducted with the goal to determine the variability of the quality parameters of the wood chips produced from the most favorable raw material (energy roundwood), and in the most controllable operational conditions (pellet factory), as the first step in identifying opportunities to optimize the wood chips’ quality monitoring. Four raw material types were tested: fir/spruce and beech debarked energy roundwood, as well as energy wood with bark of the same species. Sampling was conducted during six consecutive months along with laboratory testing, all according to the HRN EN ISO standards for solid biofuels. Interpretation of the results was done in relation to deviation from the first sampling results (as an indicator of the possibility to retain the quality of wood chips), and repeatability and reproducibility set in the standards (as an indicator of acceptable variability). The influence of the species and debarking process on the wood chips’ quality was analyzed as well. Relative deviation from the first sampling as well as the quality class change pointed moisture content as a normative property with the lowest possibility to retain initial values over the six-month period. Ash content results indicated a strong possibility to maintain the initial ash content class in the majority of the samples. In just three cases, the results of ash content were outside the reproducibility limits with first sampling as a reference. Gross calorific value results pointed only four samples outside the reproducibility limits with the first sampling results are set as a reference. Wood species influenced gross calorific value and the median value of the particle size distribution and debarking showed a significant positive effect on the moisture content reduction as well as on the ash content reduction. Presented findings are indicative for the investigated raw materials, however for the general conclusion on the subject of wood chips normative properties variation, various raw material types will have to be examined in further research.

Keywords: moisture content; ash content; calorific value; particle size distribution

1. Introduction

The EU adopted its first package of climate and energy measures back in 2008, setting three key targets to be attained by 2020: 20% reductions in greenhouse gas emissions (from 1990 levels), 20% share of renewable energy, and 20% improvement in energy efficiency [1]. Experiences of the 2020 framework indicate that European and national targets can drive strong action by the Member States and growth in emerging industries [1]. One of the sectors that was highly effected was the bioenergy sector, resulting with an increase of solid biomass shares in the gross final energy consumption in the 2010–2018 period from 6.1% to 8.0%, thus significantly increasing the share of renewables, while at the same time contributing to the overall greenhouse gas emission savings [2].

Development of the bioenergy sector, which was mostly driven by the national support schemes and emerging pellet markets, can be illustrated by the situation in Croatia in the 2014–2019 period. In 2014, only three CHP biomass facilities with an installed power
of 6.74 MWel (8% of the 85 MWel set as a 2020 goal in the Croatian National renewable energy action plan) were operational [3], and in 2019, the number of operational CHP biomass facilities reached 34 with the installed power of 73.71 MWel [4], thus almost completing the 2020 goal. In the same period, pellet production increased from 192,660 to 307,690 t [5], retaining strong export orientation, with the amount of export to EU Member States reaching 281,270 t in 2019 [5].

New policy framework for climate and energy in the period from 2020 to 2030 sets a greenhouse gas reduction target of 40% and a greater share of renewable energy in the EU of at least 27% and suggests that the EU level targets will drive continued investment in renewable energy, but at the same time, stresses a need for an improved biomass policy to maximize the resource efficient use of biomass [1]. As the generation of energy from biomass plays an important role in current international strategies to mitigate climate change and to enhance energy security [6], it can be expected that a resource-efficient use of biomass will have an impact on the future development of the bioenergy sector, but also on reaching the 2030 goals. Affordable and clean energy, approved by the United Nations General Assembly as one of the 17 Sustainable Development Goals (SDGs), was found to be one of the seven SDGs with the highest number of strong positive correlation with other SDGs [7]. Study of implementation of the SDGs in sustainable supply chain management, highlighted the need for active collaboration of producers with upstream suppliers and downstream customers on the value creation process [8]. One of the tools to ensure a resource-efficient use of biomass and increase the value of a biofuel in the supply chain is the quality control of solid biofuels with the aim to produce a biofuel of the highest possible quality, even from the lower quality raw material, and more importantly, to avoid the production of a low-quality biofuel from high-quality raw materials [9]. The quality requirements for wood-derived solid biofuels are regulated by international standards, setting the limits for technical parameters that affect the quality of solid biomass as a fuel [10]. Standards are designed in a way that enables the standardization of different solid biofuels including densified solid biofuels (pellets and briquettes) and thermally treated biofuels (charcoal), but also minimally processed fuelwood (firewood, hog fuel, and wood chips). Due to an increasing industrial scale use of wood chips for production of heat and electricity, it is important to acknowledge that wood chips quality is the major factor in energy production efficiency [10], and that the quality of wood chips is important for proper functioning of small scale non-industrial appliances [11,12]. Therefore, the standard that defines general requirements, in terms of fuel specification and classes—HRN EN ISO 17225-1:2014 [13] points out dimensions, moisture content, and ash content as the main normative properties of wood chips. Depending on the raw material origin, nitrogen, sulfur, and chlorine content are regarded as normative properties for chemically treated biomass, as well as informative properties for all fuels that are not chemically treated. Net calorific value (or energy density), bulk density, and ash melting behavior are listed as informative properties. Classification of graded wood chips for non-industrial use according to the HRN EN ISO 17225-4:2014 [14] adds bulk density on the list of normative properties for all classes and contents of N, S, Cl, and some minor elements for the lower quality classes (B), and most importantly, defines thresholds for certain properties by providing limitations concerning, among others, moisture and ash content (in certain quality classes).

Moisture content is the single most important quality attribute of wood chips or raw material for their production as it affects net calorific value, storage properties, chipping, and transportation costs of the fuel [15]. Boiler efficiency is strongly influenced by moisture content of the fuel; as increase in the moisture content reduces the net calorific value of the fuel and results in increasing the heat loss with escaping vapor [16]. Therefore, managing moisture content is a key way to improve the net calorific value and cost-efficiency of the energy wood supply [17]. Fortunately, natural drying of raw material in terms of transpirational drying of full-trees [18,19], seasoning of logging residues [20–22], energy roundwood [23–28], and firewood [29] proved to be an effective measure for reducing the moisture content. Reduction of moisture content is also possible while storing wood chips,
either naturally [26,30–32] or artificially [12,33–35]. However, in some cases of the natural drying process, negative effects such as re-wetting [31,36] and substantial dry matter loss in logging residues [21,36] and wood chips [17,30–32] can occur, which can lead to lowering of the total amount of available energy [17,31,37]. In the case of artificial drying of wood chips, financial viability has to be taken into account [38].

Ash content of wood chips is directly affected by the natural organic to inorganic matter ratio of the raw material. Apart from the naturally occurring negative effect of the present tree parts that contain significant amounts of leaves/needles or the unfavorable bark-to-wood ratio, common, for e.g., in the raw material originating from the SRC [39,40], the final product ash content can be negatively affected by inorganic matter (e.g., dirt, sand) contamination during the harvesting process [41] or by organic matter deterioration during storage [17]. Positive effects can be achieved by proper raw material manipulation; for instance, by seasoning of forest residues to lower the amount of foliage [20] or by debarking of the raw material prior to grinding, drying, and palletizing [42], which is a common practice of the quality upgrade in pellet production process when energy roundwood is used as raw material [43].

Particle size distribution of wood chips can effectively be influenced by selecting a proper raw material for desired particle size classes, as it is proven that particle size distribution is strongly affected by the type [44,45], species [46–48], and even the moisture content [45] of the raw material. Fortunately, the comminution process itself has a decisive effect on the particle size distribution. With the selection of an adequate chipper type and setting [49,50], together with regular maintenance of the chipper [49,50], substantial influence on achieving the best possible particle size distribution can be expected. The final step in upgrading the particle size distribution can, if needed and economically justified, be accomplished by screening [12], with the goal to lower the amount of fines and oversize particles. Variability of the wood chip quality parameters, as the consequence of the aforementioned naturally occurring variability of the raw material, as well as numerous options in the production chain, imposes a need for standardization. Standardization of wood chips can improve resource efficient use of biomass for energy production by identifying the key point in the production chain, where the quality upgrade might be achieved, ensuring the convergence of the appropriate wood chip quality classes and requirements of the end-user appliances, and enabling trade. Therefore, the set of solid biofuels’ standards, firstly introduced at the European level as EN standards, later harmonized with ISO standards, and currently comprising of 46 active documents at the Croatian national level [51], is an indispensable tool in the wood chips procurement chain. The next logical step, in using the potential that the solid biofuels’ standards provide, is the certification of solid biofuels. Following the successful implementation of the ENplus certification scheme for wood pellets, in 2015, the Biomassplus certification scheme for wood chips was introduced in Italy [52], and in 2018, the Good Chips certification scheme for wood chips was launched, both based on the EN ISO standards. However, the certification of a solid biofuel with such variable quality parameters as wood chips, arises certain challenges that are of greater extent than in the case of solid biofuels produced in a more controllable operational environment (wood pellets and briquettes). Some of the challenges already recognized with the application of the solid biofuels’ standards related to the sampling intensity [53] and incorporating the results of the quality parameters’ tests into the logistics chain in a timely matter, thus enabling the adjustment of the unit value of the delivered wood chips [41], can be expected with the application of the certification schemes for wood chips. The main issue that needs to be addressed is the challenges that wood chip producers might face with, in order to maintain the quality class of wood chips for which the certificate has been issued, having in mind the period for which the certificate has been issued. In this respect, due attention will have to be given to a continuous monitoring of the main wood chips’ quality parameters. Moreover, information on the expected variability of the quality parameters could be helpful in designing the appropriate procedures that would enable balancing the cost and benefit of the sampling procedure, as well as the laboratory testing.
Previous research was generally focused on individual wood chips’ quality parameters, foremost the change of moisture content during storage and influence of raw material on particle size distribution. Three main research designs were dominant: (a) interpreting the results of numerous laboratory tests to present general findings interpreted on the level of material type (harvesting residues, energy wood, . . . ) resulting with large sample database but with limitations regarding the information on wood species in question; (b) focusing on the differences between wood chips quality parameters with more details on the raw material, but without repetition; and (c) determining wood chips’ quality when testing chippers productivity. In addition, a research gap considering the effect of debarking on the wood chips’ normative properties is present. In this research a bottom-up approach was applied with limiting the influence of the raw material to only four types tested in six consecutive months. The research was set to determine the variability of the quality parameters of the wood chips produced from the most favorable raw material, and in the most controllable operational conditions, as the first step in identifying the opportunities to optimize the wood chips’ quality monitoring. In addition, the effect of the possible approaches for calculating the net calorific value results (on dry basis) of sampled wood chips was explored.

According to research goals following hypotheses were tested:

• H1—quality of wood chips produced from the same raw material type expressed by normative properties (dimensions, moisture content, and ash content) can be retained in the same quality class for six consecutive months;
• H2—ash content and gross calorific value of wood chips produced from the same raw material type can be retained for six consecutive months in ideal (repeatability and reproducibility) limits;
• H3—raw material type and debarking process significantly influences wood chips’ normative properties.

2. Materials and Methods
2.1. Data Collection

A pellet factory was chosen as the most controllable operational surrounding with the most favorable raw material. The selected pellet factory with an installed production of 70,000 t·year$^{-1}$ together with the sawmill residues uses nearly 65,000 m$^3$·year$^{-1}$ of energy roundwood as raw material. Energy roundwood mix consists of 63% silver fir and Norway spruce and 37% European beech, which is in the line with common raw material mix (approximately 70% coniferous wood and remaining part deciduous wood) previously reported [54]. Product range is ENplus A1 pellet and ENplus A2 pellet. For achieving the adequate ash content, crucial in the pellet classification, a part of the energy roundwood has to be debarked. In the specific case, 44% of the total amount of the silver fir and Norway spruce roundwood is processed as debarked, together with 12.5% of the total amount of European beech energy roundwood that is also being used as debarked. Energy roundwood is stored at the factory open landing for a maximum of 30 days or less prior to chipping. In the case of debarked energy roundwood, storage is continued at the open landing for a maximum of 10 days or less prior to chipping. Chipping is performed by a stationary electro-powered JENZ 561 StA drum chipper (JENZ GmbH, Petershagen, Germany). Wood chips produced in this way are stored in separate compartments of a roofed storage depending on the species (silver fir and Norway spruce/European beech) and of the debarking pretreatment (debarked/with bark). Data on the harvesting system, time of felling and time of natural drying in the forest stand and by the forest road was not available.

Selected research site was chosen as it represents the closest operational conditions to biomass upgrading terminal. Use of the terminal as a part of the supply chain offers better environment for controlling fuel properties, and the upgrading terminal offers the possibility to improve the quality of the fuel by introducing a pre-comminution or post-comminution artificial or natural drying and post-comminution sieving, blending, and
densifying [55]. Selected raw material represents the raw material of the highest possible quality, where it is expected that further processing will result with wood chips of the highest quality and low variability in terms of the normative parameters. In addition, selected raw material represents the tree species most commonly used in the research area and beyond.

Sampling of the wood chips produced by comminution of four different raw materials:

- **S**—debarked silver fir (*Abies alba* L.) and Norway spruce (*Picea abies* (L.) H. Karst.) energy roundwood;
- **SB**—silver fir (*Abies alba* L.) and Norway spruce (*Picea abies* (L.) H. Karst.) energy roundwood with bark;
- **H**—debarked European beech (*Fagus sylvatica* L.) energy roundwood;
- **HB**—European beech (*Fagus sylvatica* L.) energy roundwood with bark;

Sampling was done on a monthly basis during a six-month period, starting from May and ending in October 2020. Sampling was conducted following the HRN EN ISO 18135:2017 standard [56]. In total, 24 samples were collected (four samples of different raw material x 6 months). Nearly all samples were collected within 24 h after chipping (only two deviated from this procedure).

2.2. Laboratory Tests

Normative properties of the sampled wood chips, according to the standard HRN EN ISO 17225-1:2014 [13], were determined. For all of the collected samples, moisture content was determined by gravimetric method according to the HRN EN ISO 18134-2:2017 standard [57]; particle size distribution was determined by oscillating screen method according to the HRN EN ISO 17827-1:2016 standard [58]; and ash content following the HRN EN ISO 18122:2015 standard [59]. In addition, gross calorific value was determined using calorimeter according to the HRN EN ISO 18125:2017 standard [60]. According to the HRN EN ISO 14780:2017 standard [61], laboratory samples and general analysis samples were prepared; particle size reduction to nominal top size of 1 mm or less was done using a cutting mill.

For the first four samples of different raw material (sampled in May) and for all of the samples of wood chips produced by chipping the European beech energy roundwood with bark (as raw material with greatest expected variability), a total carbon, hydrogen, and nitrogen content was determined according to the HRN EN ISO 16948:2015 standard [62], total content of sulfur and chlorine was determined according to the HRN EN ISO 16994:2016 standard [63], while the oxygen content was calculated according to the HRN EN ISO 16993:2016 standard [64]. Net calorific value (on dry basis) was calculated based on the determined content of hydrogen, nitrogen, and oxygen for individual samples and based on the default values for “Stemwood without bark, needles and leaves” stated in the Annex G of the HRN EN ISO 18125:2017 standard [60].

Results of the particle size distribution testing were processed to calculate shares of the fines (<3.5 mm), accepts (3.15–63 mm), and oversize (>63 mm) fractions and to express the median value of the particle size distribution. Limits of the fractions were defined in order to uniform the data representation. However, for determining the quality classes, different limits were applied (main and coarse fraction) according to the standard.

2.3. Statistical Analyses

Statistical analyses were performed in the software package STATISTICA version 13. Normal distribution of the datasets was tested by Kolmogorov–Smirnov and Liliefors test. Influence of the raw material properties (i.e., wood species and debarking) on moisture content, ash content, gross calorific value, and median value of the particle size distribution was analyzed by using ANOVA. Scheffe post-hoc test was used to determine homogenous groups at a 5% significance level. t-test was used to determine statistical differences of net calorific values (on dry basis), calculated (i) based on the determined hydrogen, nitrogen,
and oxygen content for individual samples; (ii) based on the default values; and (iii) based on values determined in the first sampling.

Interpretation of the results was done concerning the quality class limits, determined variability, deviation from the first (May) sampling results, repeatability, and reproducibility limits defined in the standards.

3. Results

3.1. Moisture Content

The lowest average moisture content, but with the highest standard deviation, was found for wood chips produced by chipping of debarked fir/spruce energy wood. ANOVA results (Table A3 in Appendix B) detected debarking as an efficient way of reducing the moisture content, and Scheffe post-hoc test (Table 1) clearly delineated debarking effect in fir/spruce energy wood (with a 13% reduction of moisture content), while in the case of beech energy wood (that showed similar reduction of the moisture content, 11.2%), that was not clearly recognized by the same test.

Table 1. Results of the moisture content analyses.

| Raw Material                  | S Fir/Spruce Energy Wood Debarked | SB Fir/Spruce Energy Wood       | H Beech Energy Wood Debarked    | HB Beech Energy Wood       |
|-------------------------------|-----------------------------------|---------------------------------|---------------------------------|---------------------------|
| Average moisture content, %w  | 30.77<sup>a</sup>                | 43.76<sup>b</sup>              | 31.72<sup>ab</sup>             | 42.90<sup>ab</sup>         |
| Standard deviation of the moisture content, %w | ±9.67                             | ±7.15                           | ±5.76                           | ±5.30                     |
| Moisture content of the first sampling, %w (Moisture content class) | 34.6 (M35)                        | 47.4 (M50)                      | 36.6 (M40)                      | 45.1 (M50)                |
| Deviation of the 1st sampling moisture content, %w (Moisture content class) | 2nd sampling: +2.6 (M40)         | −4.5 (M45)                      | +0.7 (M40)                      | +6.1 (M55)                |
|                               | 3rd sampling: −4.5 (M35)          | +8.7 (M55+)                     | −4.8 (M35)                      | −7.8 (M40)                |
|                               | 4th sampling: −15.0 (M20)         | −10.6 (M40)                     | −4.0 (M35)                      | −7.4 (M40)                |
|                               | 5th sampling: −14.9(M20)          | −9.8 (M40)                      | −6.0 (M35)                      | −4.0 (M45)                |
|                               | 6th sampling: +9.0(M45)           | −5.6 (M45)                      | −15.3 (M25)                     | 0.0 (M50)                 |

<sup>1</sup> Different letters denote significant differences at the 5% level.

3.2. Ash Content

Initial and average ash content was below 1.0% for all types of raw material tested, with only one case in beech energy wood with bark, where the ash content was slightly above the before mentioned threshold (Table 2). Wood species and debarking both showed statistically significant influence on the ash content (Table A4), resulting with the most favorable ash content in the case of debarked fir/spruce energy wood, and the least favorable in the case of beech energy wood with bark.

When analyzing deviations from the first sampling, as indicators of the possibility to retain the quality parameters set in the initial sampling, it is important to notice that initial ash content class was maintained (or upgraded) for all fir/spruce samples, while in the case of beech energy wood, ash content class was higher in two cases of debarked wood and in one case of wood with bark. It is also important to stress the fact that for nine samples, deviation from the initial sampling ash content falls into repeatability limits (0.1% absolute) and in only three cases, the results are outside reproducibility limits (0.2% absolute).
Table 2. Results of the ash content analyses.

| Raw Material                      | S Fir/Spruce Energy Wood Debarked | SB Fir/Spruce Energy Wood | H Beech Energy Wood Debarked | HB Beech Energy Wood |
|-----------------------------------|-----------------------------------|---------------------------|-----------------------------|---------------------|
| Average ash content, %w           | 0.51\(^a\)                        | 0.76\(^{bc}\)            | 0.66\(^{ab}\)              | 0.91\(^c\)          |
| Standard deviation of the ash content, %w | ±0.14                             | ±0.10                     | ±0.13                       | ±0.18               |
| Ash content of the first sampling, %w | (Moisture content class)           |                           |                             |                     |
| 2nd sampling                      | +0.02 (A0.7)                      | +0.07 (A1.0)             | +0.14 (A1.0)               | +0.24 (A1.5)        |
| 3rd sampling                      | +0.02 (A0.7)                      | −0.16 (A0.7)             | −0.12 (A0.7)               | +0.09 (A1.0)        |
| 4th sampling                      | −0.19 (A0.5)                      | −0.11 (A0.7)             | +0.19 (A1.0)               | +0.07 (A1.0)        |
| 5th sampling                      | −0.27 (A0.5)                      | +0.07 (A1.0)             | −0.08 (A0.7)               | +0.06 (A1.0)        |
| 6th sampling                      | +0.07 (A0.7)                      | −0.11 (A0.7)             | −0.12 (A0.7)               | −0.31 (A0.7)        |

\(^1\) Different letters denote significant differences at the 5% level.

3.3. Calorific Value

Higher gross calorific values were found for fir/spruce energy wood compared to beech energy wood, and debarking itself showed no statistical effect on the gross calorific value (Table A5).

Deviation from the first sampling gross calorific results reveals that for only two samples, repeatability limits (140 J/g) were not exceeded, but on the other hand, only four samples are outside reproducibility limits (400 J/g); all of them with lower values than determined in the first sampling (Table 3).

Table 3. Results of the calorimetric analyses.

| Raw Material                      | S Fir/Spruce Energy Wood Debarked | SB Fir/Spruce Energy Wood | H Beech Energy Wood Debarked | HB Beech Energy Wood |
|-----------------------------------|-----------------------------------|---------------------------|-----------------------------|---------------------|
| Average gross calorific value, MJ/kg \(^1\) | 20.065\(^{a}\)                  | 20.002\(^{a}\)          | 19.446\(^{b}\)             | 19.593\(^{ab}\)    |
| Standard deviation of the gross calorific value, MJ/kg | ±0.270                          | ±0.337                    | ±0.204                      | ±0.322              |
| Gross calorific value of the first sampling, MJ/kg | 20.134                          | 20.172                    | 19.503                      | 19.816              |
| Deviation of the 1st sampling gross calorific value, MJ/kg | −0.159                          | −0.174                    | −0.207                      | −0.307              |
| 2nd sampling                      | −0.294                          | −0.747                    | −0.200                      | −0.424              |
| 3rd sampling                      | −0.409                          | −0.341                    | −0.279                      | −0.590              |
| 4th sampling                      | +0.154                          | +0.046                    | +0.103                      | −0.322              |
| 5th sampling                      | +0.299                          | +0.194                    | +0.240                      | +0.302              |

\(^1\) Different letters denote significant differences at the 5% level.

Net calorific value results for all samples of the first sampling (Table 4) were calculated based on the results of the CHN(S) analyses presented in the Table A1 in Appendix A, and based on the HRN EN ISO 18125:2017 default values for stemwood without bark, needles, and leaves.

The effect of using sample specific (determined) values of hydrogen, oxygen, and nitrogen content (Table A2), default values, and the first sampling values in calculation of the net calorific value of beech energy wood with bark is presented in Table 5. \(t\)-test showed statistically significant differences between the net calorific value based on the results of CHN(S) analyses and net calorific value based on the first sampling H, O, N values \((t = −3.03064, \ p = 0.029060)\), as well as between the net calorific value based on the results of CHN(S) analyses and net calorific value based on the EN ISO 18125:2017 default values \((t = 4.64683, \ p = 0.005598)\).
Table 4. Results of the net calorific value calculation.

| Raw material | S Fir/Spruce Energy Wood Debarked | SB Fir/Spruce Energy Wood | H Beech Energy Wood Debarked | HB Beech Energy Wood |
|--------------|-----------------------------------|--------------------------|-----------------------------|---------------------|
| Net calorific value based on the results of CHN(S) analyses, MJ/kg | 18.81 | 18.19 | 18.26 | 18.57 |
| Net calorific value based on the HRN EN ISO 18125:2017 default values, MJ/kg | (−0.03) | (−0.09) | (−0.11) | (−0.10) |

Table 5. Results of the net calorific value calculation of beech energy wood.

| Sampling | 1 | 2 | 3 | 4 | 5 | 6 |
|----------|---|---|---|---|---|---|
| Net calorific value based on the results of CHN(S) analyses, MJ/kg | 18.57 | 18.18 | 18.08 | 17.92 | 18.23 | 18.85 |
| Net calorific value based on the HRN EN ISO 18125:2017 default values, MJ/kg | 18.47 | 18.16 | 18.04 | 17.88 | 18.14 | 18.77 |
| (Deviation from Net calorific value based on the results of CHN(S) analyses, MJ/kg) | (−0.10) | (−0.02) | (−0.04) | (−0.04) | (−0.09) | (−0.08) |
| Net calorific value based on the 1st sampling H, O, N values, MJ/kg | 18.57 | 18.27 | 18.15 | 17.98 | 18.25 | 18.87 |
| (Deviation from Net calorific value based on the results of CHN(S) analyses, MJ/kg) | (+0.09) | (+0.07) | (+0.06) | (+0.02) | (+0.02) | (+0.02) |

3.4. Particle Size Distribution

In general, tested samples showed favorable particle size distribution, both in terms of fines fraction (with only one sample of fir/spruce energy wood with bark over the initial F05 class threshold) and in terms of the high accepts (3.15–63 mm) fraction share (Table 6, Figure 1). However, classification in the main fraction class showed high variability; eight samples were classified as P16 class, five samples had to be classified as P31S because of the less than 60% of the main fraction for P16, and nine samples in total could not have been classified in the initial (first sampling) class (P16, P31S) because their maximal length of particles are >150 mm-eight samples and one sample with the maximum cross sectional area larger than 4 cm² (of which 6 samples in addition had less than 60% of the main fraction for classification to P16).

ANOVA (Table A6) showed statistically significant effect on the median value of the particle size distribution only for the wood species parameter. A detailed presentation of the individual fractions’ share (Figure 1) reveals reasons for an increased median value of the particle size distribution in the case of beech energy wood; a decreased share of the fraction >3.15–8 mm and an increased share of the >16–31.5 mm fraction compared to fir/spruce energy wood.
Table 6. Results of the particle size distribution analyses.

| Raw Material       | S Fir/Spruce Energy Wood Debarked | SB Fir/Spruce Energy Wood | H Beech Energy Wood Debarked | HB Beech Energy Wood |
|--------------------|----------------------------------|---------------------------|-----------------------------|----------------------|
| Median value of the particle size distribution, mm \(^1\) | 12.22 \(^a\)                  | 12.38 \(^a\)             | 14.25 \(^b\)              | 14.92 \(^b\)         |
| Standard deviation of the median value of the particle size distribution, mm | ±0.86                       | ±1.07                     | ±0.62                      | ±1.40                |
| Average share of the fine fraction, %w | 2.57                        | 4.03                      | 2.46                       | 2.32                 |
| Standard deviation of fine fraction share, %w | ±1.49                       | ±1.28                     | ±1.14                      | ±0.59               |
| Share of the fine fraction of the first sampling, %w (Fine fraction class) | 3.66 (F05)                  | 4.16 (F05)               | 2.17 (F05)                | 1.74 (F05)          |
| Deviation of the 1st sampling share of the fine fraction, %w (Fine fraction class) | +0.09 (F05)                | +0.29 (F05)              | +0.95 (F05)               | +1.16 (F05)         |
| Deviation of the 3rd sampling share of the fine fraction, %w (Fine fraction class) | +0.23 (F05)                | −0.39 (F05)              | +1.89 (F05)               | +1.20 (F05)         |
| Deviation of the 4th sampling share of the fine fraction, %w (Fine fraction class) | −2.94 (F05)                | −2.38 (F05)              | −0.78 (F05)               | +0.94 (F05)         |
| Deviation of the 5th sampling share of the fine fraction, %w (Fine fraction class) | −2.91 (F05)                | +0.12 (F05)              | +0.80 (F05)               | −0.12 (F05)         |
| Deviation of the 6th sampling share of the fine fraction, %w (Fine fraction class) | −1.03 (F05)                | +1.54 (F10)              | −1.12 (F05)               | +0.30 (F05)         |
| Average share of the accepts fraction, %w | 96.75                      | 95.55                     | 97.25                      | 97.39               |
| Standard deviation of accepts fraction share, %w | ±1.16                      | ±1.13                     | ±1.15                      | ±0.65               |
| Share of the accepts fraction of the first sampling, %w (Main fraction class) | 96.30 (P16)                | 95.67 (P16)              | 97.49 (P16)               | 97.75 (P31S)        |
| Deviation of the 1st sampling share of the accepts fraction, %w (Main fraction class) | −0.29 (P16)                | −0.45 (P45)              | −0.93 (P31S)              | −0.85 (P31)         |
| Deviation of the 3rd sampling share of the accepts fraction, %w (Main fraction class) | −0.78 (P31)                | +0.05 (P31)              | −1.75 (P31S)              | −0.95 (P31S)        |
| Deviation of the 4th sampling share of the accepts fraction, %w (Main fraction class) | +0.80 (P45)                | +1.79 (P31)              | +1.02 (P31)               | −0.99 (P31)         |
| Deviation of the 5th sampling share of the accepts fraction, %w (Main fraction class) | +2.54 (P31)                | −0.37 (P31)              | −0.88 (P31S)              | +0.54 (P16)         |
| Deviation of the 6th sampling share of the accepts fraction, %w (Main fraction class) | +0.43 (P16)                | −1.72 (P16)              | +1.12 (P31)               | +0.08 (P16)         |
| Average share of the oversize fraction, %w | 0.68                       | 0.42                      | 0.28                       | 0.30                |
| Standard deviation of oversize fraction share, %w | ±0.77                      | ±0.20                     | ±0.12                      | ±0.20               |
| Share of the oversize fraction of the first sampling, %w | 0.04                       | 0.17                      | 0.34                       | 0.51                |
| Deviation of the 1st sampling share of the oversize fraction, %w | +0.20                      | +0.16                     | −0.02                      | −0.31               |
| Deviation of the 3rd sampling share of the oversize fraction, %w | +0.54                      | +0.34                     | −0.14                      | −0.26               |
| Deviation of the 4th sampling share of the oversize fraction, %w | +2.14                      | +0.59                     | −0.23                      | +0.05               |
| Deviation of the 5th sampling share of the oversize fraction, %w | +0.38                      | +0.25                     | +0.08                      | −0.42               |
| Deviation of the 6th sampling share of the oversize fraction, %w | +0.60                      | +0.18                     | +0.00                      | −0.38               |

\(^1\) Different letters denote significant differences at the 5% level.
4. Discussion

All tested wood chip quality parameters showed variation to some extent in terms of the raw material type and month (number) of sampling. The observed variation was assumed due to a natural origin of the raw material, as well as numerous factors affecting the production process. For reasons of limiting the effect of the raw material properties on the quality parameters, energy roundwood was selected as the most suitable in terms of the expected variability compared to logging residues and whole trees. Variation was expected, as a result of different wood species and debarking process.

Moisture content of the sampled wood chips showed greatest variation in terms of relative deviation from the first sampling and more importantly, highlighted the inability to retain the moisture content class over the research period of six months. The obtained results were expected, having in mind that the research was conducted in a pellet factory where the artificial conditioning of moisture content is an integral part of the pellet production process, and therefore moisture content of the raw material is a matter to be considered mainly regarding the energy efficiency, and less important for the final product quality. In contrary, when producing wood chips for a direct use, moisture content optimization is an integral part of the supply chain and benefits of the raw material seasoning must be exploited. In this respect, options of either procuring seasoned raw material of known and adequate duration of seasoning or fresh raw material and seasoning on the facility landing are recommended. Using the potential of seasoning for moisture content reduction should also contribute to balancing the expected variability, especially when lower moisture content values are reached. Nevertheless, given the fact that moisture content is the single most important quality parameter of wood chips and the parameter that shows the highest variation, frequent sampling and testing are a necessity.

The debarking process proved to be an effective measure to reduce the moisture content, contrary to reports of previous research of partial debarking of energy roundwood by harvester head [23,24]. However, since a relatively high effect of a 10%-moisture content reduction was observed in only a 10-days period (which could also be related to the possible higher moisture content of the bark compared to wood), this finding should be investigated in more detail.

In addition to the positive effect on the moisture content reduction, debarking had a significant influence on the ash content reduction. Ash content of debarked energy
wood is within typical variation range for coniferous and broad-leaf wood stated in the Annex B of the HRN EN ISO 17225-1:2014 standard [13]. Energy wood with bark also showed favorable ash content results, considerably lower than average values for similar raw material reported in the previous research [50], which could indicate a proper raw material handling in the procurement chain in terms of selecting adequate harvesting equipment and proper storage. Previous research found extreme differences between wood and bark ash contents, the latter being up to ten times greater for instance in the case of SRC poplar [40]. On the roundwood level, ash content is a result of natural levels of bark and wood ash but also favorable wood to bark ratio. High levels of ash content, common in logging residue wood chips [50], are seldomly reached when chipping energy wood (with bark), and would indicate contamination with dirt or sand in the harvesting process [41]; that situation was evidently successfully avoided in this research. Although results indicate a strong possibility to maintain the ash content class from the initial (first) sampling in the majority of the following samplings (with a number of cases with improved ash content class), ash content should still be regularly tested, especially if the raw material contamination occurred. In this research, low ash content variation for each raw material respectively can be substantiated when compared to reproducibility limits (0.2% absolute). Only in three cases, the results of ash content were outside the reproducibility limits with first sampling as a reference.

Gross calorific value was found to be independent of the debarking process, but statistically dependent of the species type showing results similar to average values for coniferous and broadleaved wood chips on the Italian energy market [65]. Only four samples were outside the reproducibility limits (400 J/g) when the first sampling results are set as a reference. This indicates that a possibility to determine site specific and raw material specific gross calorific value should be explored in further research. This might be a useful tool in reducing the cost of quality control of wood chips produced from energy roundwood, but probably would not be the best option for wood chips produced from logging residues, a raw material that often consists of a mix of different wood species and different tree parts. In addition, chemical composition and its variability has to be taken into account. Use of the HRN EN ISO 18125:2017 standard default values for hydrogen, oxygen, and nitrogen content in the net calorific calculation should be verified by laboratory analyses given the fact that Annex G of the HRN EN ISO 18125:2017 standard does not provide default values for all raw material types (e.g., stemwood with bark). If the option of using site specific and raw material specific values for hydrogen, oxygen, and nitrogen content is considered, first sampling results should be confirmed either by comparison with results of the previous research of similar raw material, following the results of the second sampling, or ideally by forming and continuous updating of the hydrogen, oxygen, nitrogen contents of the site and raw material laboratory database.

Main findings in the particle size distribution analyses indicate that classification is under a strong influence of the oversized particles (which are in most of the samples present in negligible share), even in the case of a high quality and uniform raw material. Therefore, for retaining the desired particle size class, additional efforts should be put on the proper maintenance of the chipper, especially on the condition of the chipper knives [49]. One of the reasons for relatively high class variability is also the fact that P16 was the first sampling main fraction in three raw material types. Although a P16 might be considered a logical target class in the pellet production, wood chips for direct energy use are usually not produced in this class. Focusing on the target class of larger dimensions could resolve the oversize particle problem in terms of classification, since in most of the samples, dimensions of the oversize particles would be within the limits. In addition, it is interesting to notice that chipping of beech energy roundwood, with the same chipper settings, produced wood chips of a higher median value as a result of the lower share of particle class >3.15–8 mm, and a higher share of particle class >16–31.5 mm, compared to fir/spruce energy roundwood. Wood species effect on chip size was also reported in the research of pine and chestnut whole tree wood chips [46], pine and poplar roundwood chips, latter resulting
with larger wood chips possibly related to different wood properties [44], birch chips being slightly larger than pine and spruce chips when chipping delimbed stemwood logs [47], as well as in the research of beech and poplar chips, determining significantly larger average size of beech chips than for poplar chips, possibly due to the higher strength of beech wood [48].

5. Conclusions

Relative deviation from the first sampling as well as the quality class change pointed moisture content as a normative property with the lowest possibility to retain initial values over the six-month period. Results indicate the need for frequent sampling and a better coordination within the supply chain in order to benefit from the moisture reduction by seasoning of the raw material.

Ash content results indicate a strong possibility to maintain the initial ash content class in the majority of the samples. Low ash content variation is further substantiated when compared to reproducibility limits. In just three cases, the results of ash content were outside the reproducibility limits with first sampling as a reference.

Results of the particle size distribution analyses indicate that classification is under a strong influence of the oversized particles that were in most of the samples present in negligible share.

Gross calorific value results pointed only four samples outside the reproducibility limits with the first sampling results are set as a reference that indicates the possibility to use site specific and raw material specific gross calorific value.

Wood species influenced gross calorific value and the median value of the particle size distribution and debarking showed a significant positive effect on the moisture content reduction as well as on the ash content reduction.

In this research, options for optimizing the quality control of wood chips were investigated in terms of balancing the intensity of sampling and laboratory analyses with the information needed to confirm the expected quality class of delivered wood chips over the longer period. Findings are indicative for the investigated raw materials, however for the general conclusion on the subject of wood chips normative properties variation, various raw material types will have to be examined in further research.

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Appendix A. Results of the CHN(S) Analyses

Table A1. Results of the CHN(S) analyses.

| Raw Material | S Fir/Spruce Energy Wood Debarked | SB Fir/Spruce Energy Wood | H Beech Energy Wood Debarked | HB Beech Energy Wood |
|--------------|----------------------------------|---------------------------|----------------------------|---------------------|
| Hydrogen content, w−% d | 6.1 (−0.1) | 5.8 (−0.4) | 5.7 (−0.5) | 5.7 (−0.5) |
| Oxygen content, w−% d | 42.80 (−0.2) | 42.80 (−0.2) | 44.30 (+1.3) | 43.30 (+0.3) |
| Nitrogen content, w−% d | 0.11 (+0.01) | 0.15 (+0.05) | 0.13 (+0.03) | 0.15 (+0.05) |

Table A2. Results of the CHN(S) analyses of beech energy wood.

| Sampling | 1 | 2 | 3 | 4 | 5 | 6 |
|----------|---|---|---|---|---|---|
| Hydrogen content, w−% d | 5.7 (−0.5/) | 6.1 (−0.1/+0.4) | 6.0 (−0.2/+0.3) | 6.0 (−0.2/+0.3) | 5.8 (−0.4/+0.1) | 5.8 (−0.4/+0.1) |
| Oxygen content, w−% d | 43.30 (+0.30/) | 43.20 (+0.20/−0.10) | 44.04 (+1.04/+0.74) | 44.34 (+1.34/+1.04) | 43.10 (+0.10/−0.20) | 42.20 (−0.80/−1.10) |
| Nitrogen content, w−% d | 0.15 (+0.05/) | 0.20 (+0.10/−0.05) | 0.16 (+0.06/+0.01) | 0.28 (+0.18/+0.13) | 0.20 (+0.10/+0.05) | 0.14 (+0.04/−0.01) |

Appendix B. Results of the ANOVA

Table A3. ANOVA table for the moisture content.

| Effect | SS   | DF | MS   | F    | p    |
|--------|------|----|------|------|------|
| Intercept | 33375.53 | 1 | 33375.53 | 648.4461 | <0.000001 |
| Wood species | 0.01 | 1 | 0.01 | 0.0002 | 0.990328 |
| Debarking | 875.32 | 1 | 875.32 | 17.0065 | 0.000527 |
| Wood species*Debarking | 4.82 | 1 | 4.82 | 0.0936 | 0.762846 |
| Error | 1029.40 | 20 | 51.47 | | |
Table A4. ANOVA table for the ash content.

| Effect                  | SS      | DF | MS           | F        | p          |
|-------------------------|---------|----|--------------|----------|------------|
| Intercept               | 12.11734| 1  | 12.11734     | 616.8504 | <0.000001  |
| Wood species            | 0.14209 | 1  | 0.14209      | 7.2333   | 0.014098   |
| Debarking               | 0.37500 | 1  | 0.37500      | 19.0899  | 0.000297   |
| Wood species*Debarking  | 0.00000 | 1  | 0.00000      | 0.0001   | 0.992349   |
| Error                   | 0.39288 | 20 | 0.01964      |          |            |

Table A5. ANOVA table for the gross calorific value.

| Effect                  | SS       | DF | MS         | F   | p     |
|-------------------------|----------|----|------------|-----|-------|
| Intercept               | 9386.656 | 1  | 9386.656   | 113114.9 | <0.000001 |
| Wood species            | 1.589    | 1  | 1.589      | 19.1| 0.000292 |
| Debarking               | 0.011    | 1  | 0.011      | 0.1 | 0.724496 |
| Wood species*Debarking  | 0.066    | 1  | 0.066      | 0.8 | 0.381344 |
| Error                   | 1.660    | 20 | 0.083      |     |       |

Table A6. ANOVA table for the median value of the particle size distribution.

| Effect                  | SS       | DF | MS    | F    | p     |
|-------------------------|----------|----|-------|------|-------|
| Intercept               | 4336.282 | 1  | 4336.282 | 4121.941 | <0.000001 |
| Wood species            | 31.282   | 1  | 31.282 | 29.735 | 0.000024 |
| Debarking               | 0.375    | 1  | 0.375  | 0.356 | 0.557176 |
| Wood species*Debarking  | 1.042    | 1  | 1.042  | 0.990 | 0.331587 |
| Error                   | 21.040   | 20 | 1.052  |      |       |

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