

## Article

# Energy Efficiency of Conifer Cones and Seed Extraction Residue Biomass

Jacek Kwiatkowski <sup>1,\*</sup> and Zdzisław Sztejna <sup>2</sup>

<sup>1</sup> Department of Genetics, Plant Breeding and Bioresource Engineering, Faculty of Agriculture and Forestry, University of Warmia and Mazury in Olsztyn, 10-724 Olsztyn, Poland

<sup>2</sup> The State Forests, Forest District Jedwabno, Seed Extraction Plants, 12-122 Jedwabno, Poland; [zdzislaw.sztejna@olsztyn.lasy.gov.pl](mailto:zdzislaw.sztejna@olsztyn.lasy.gov.pl)

\* Correspondence: [jacek.kwiatkowski@uwm.edu.pl](mailto:jacek.kwiatkowski@uwm.edu.pl)

**Abstract:** Sustainable forest management, which accounts for the multiple roles played by forests, includes seed collection from selected areas for forest renewal and regeneration. The process of harvesting conifer seeds generates considerable amounts of waste biomass that can be used as a source of energy to supplement the local solid fuel market. Therefore, their quality is an important consideration. The mass fraction of Scots pine seed extraction residues was determined in this study. The thermophysical properties and elemental composition of the residues and spent Norway spruce and European larch cones (after seed extraction) were evaluated. An analysis of Scots pine seed extraction residues revealed that only cones had practical application. They accounted for more than 99% of total residue biomass and were characterized by the lowest content of ash, sulfur, and chlorine. The calorific value of cones of the analyzed tree species ranged from 17.08 to 18.29 MJ kg<sup>-1</sup>, the chlorine content was 0.010–0.041% DM, and the sulfur content was 0.019–0.043% DM. Due to the specificity of the extraction process, the generated waste, including cones, had a very low moisture content of 6.86–10.02%, which significantly increased their value as solid fuel.

**Keywords:** cones; seed extraction residues; Scots pine; Norway spruce; European larch; lignocellulosic biomass; HHV; LHV; ash content; sulfur content; chlorine content



**Citation:** Kwiatkowski, J.; Sztejna, Z. Energy Efficiency of Conifer Cones and Seed Extraction Residue Biomass. *Sustainability* **2024**, *16*, 2693. <https://doi.org/10.3390/su16072693>

Academic Editor: Jun Wang

Received: 28 February 2024

Revised: 19 March 2024

Accepted: 23 March 2024

Published: 25 March 2024



**Copyright:** © 2024 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (<https://creativecommons.org/licenses/by/4.0/>).

## 1. Introduction

The main aim of the European Union's (EU) energy policy is to increase the share of renewable energy in total energy consumption [1]. Solid biofuels have the largest share of renewable energy production, which exceeded 40% in the EU and 64% in Poland in 2022 [2]. Solid biomass for energy generation is derived mainly from forests and the timber industry [3–9], and it consists of sawmill waste such as branches, woodchips, bark, and sawdust [10,11]. Forest residues account for around 1% of waste biomass used in energy generation [12]. These residues are processed by forest biorefineries into value-added products [13–17].

At the end of 2022, the total forest area in Poland was estimated at 9,275,000 ha, marking an increase of 5.9% from 1990. Coniferous trees cover 68.7% of the total forest area, mostly Scots pines (85%), as well as spruces (7.5%) and larches (4.9%) [18].

Rational forest management practices promote the continuous and sustainable development of forests, as well as the regeneration of forests where timber is harvested [19]. In recent years, around 66,000 ha of forests were regenerated and planted in Poland each year on average [20]. Despite the steady increase in the percentage of naturally regrown forests (from 4.1% in 1990 to 19.8% in 2022), artificial regeneration continues to play the main role in forest restocking [18].

Seeds for forest restocking are obtained from dedicated seed stocks, including parent trees and seed tree stands. At the end of 2022, the area of seed tree stands registered in Poland reached nearly 253,000 ha, and Scots pine seeds were harvested in around 60% of these stands [18]. Seed production in these stands varies across years and is largely influenced by seed yields (seed

years) and current demand. According to Aniszewska and Kuszpit [21], between 2009 and 2012, more than 254 Mg of conifer cones, including Scots pine cones (89.9%), spruce cones (4.6%), fir cones (3.0%), and larch cones (2.5%), were harvested each year in Poland on average. An analysis of certificates of origin for forest reproductive materials revealed that approximately 360 Mg of Scots pine cones, 48.8 Mg of fir cones, 17.9 Mg of spruce cones, and 11 Mg of larch cones were harvested in 2022 in Poland [22]. A similar amount of Scots pine cones (350 Mg) was harvested for the same purpose in 2021 [22].

The seed extraction process generates waste biomass, including empty cones, seed wings, empty seeds, and other minor impurities. Cones are most difficult to manage because their volume increases during drying, and they require considerable storage space. Research has shown that cones can be a source of various valuable raw materials [14]. However, in practice, they are usually used for ornamental purposes, such as Christmas decorations. Cones are also used as fuel in some seed extraction plants.

The rational management of seed extraction residues plays an important role in sustainable forestry practices [23]. Spent cones, the by-products of seed extraction, constitute a secondary source of forest biomass and can be effectively used in energy generation. Seeds are extracted from cones by drying, which decreases the moisture content of cones, and their calorific value is not influenced by weather. Cones are a stable and supplementary source of biomass in local energy markets [24,25].

The properties of biomass from the process of seed extraction should be determined if it is to be used for energy production [7]. The content of fixed carbon, volatile matter, and ash is important in the process of biomass combustion, and the elemental composition of biomass is analyzed to estimate its calorific value (C, H, O) and environmental impact (N, S, Cl) [26]. The nitrogen content of biomass affects nitrogen oxide emissions during combustion [27]. Nitrogen oxides are among the major air pollutants responsible for the greenhouse effect and acid rain [28]. Chlorine and sulfur also contribute to the formation of deposits and corrosion in boilers [29].

The aim of this study was to determine the proportions of mass fractions and selected physical parameters of Scots pine seed extraction residues, and to evaluate the thermophysical properties and elemental composition of Scots pine, Norway spruce, and European larch cones after seed extraction.

## 2. Materials and Methods

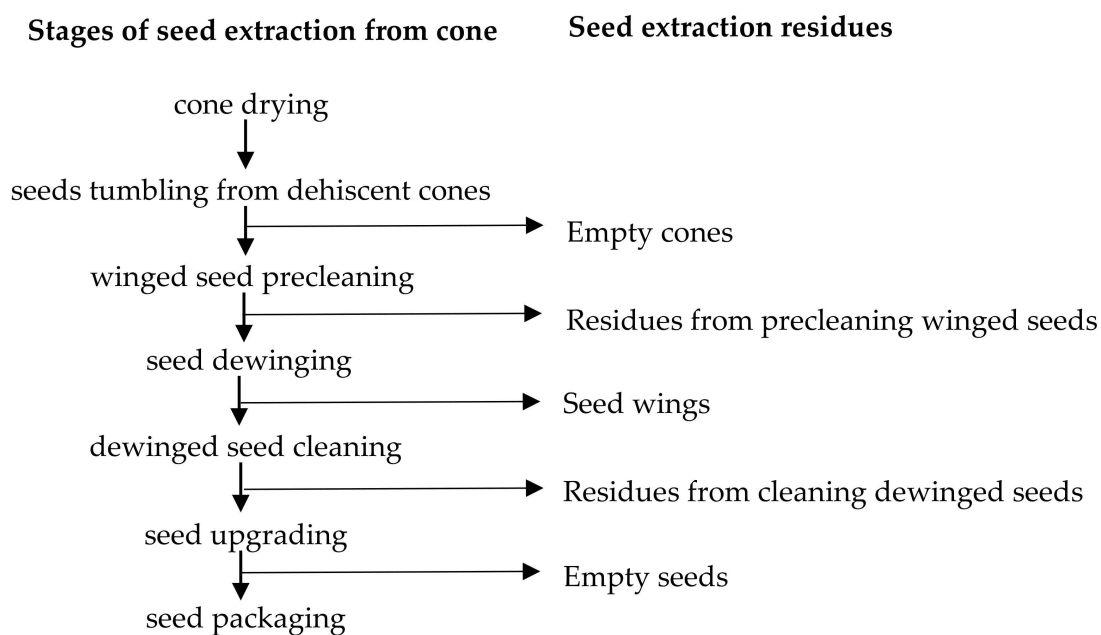
### 2.1. Materials

Residual biomass from the process of extracting seeds from the cones of female coniferous trees was obtained from a seed extraction plant in Jedwabno (Regional Directorate of State Forests in Olsztyn, Poland). The plant processes around 25 Mg of cones per year on average. Seed extraction residues from three species of coniferous trees (Scots pine, Norway spruce and European larch) were analyzed in the study. These species have their natural habitats in the plant's operation area. The residues were obtained from spent cones that were harvested in the 2021/2022 season in local forest districts for the purpose of forest restocking. In Scots pines, waste biomass fractions were examined in all stages of seed extraction and processing (cleaning, dewinging, fractioning) (Figures 1 and 2). Only spent cones were analyzed in Norway spruces and European larches. European larch seeds are extracted in a thermal and mechanical process, and the resulting waste biomass consists of two sub-fractions—central cone stems and woody seed scales—which were evaluated separately (Figure 2).

### 2.2. Laboratory Analyses

Scots pine seed extraction residues were divided into mass fractions by weighing each fraction in three different batches of Scots pine residues. The proportions of mass fractions were determined based on the mean values noted in each batch. The specific weight of each fraction was determined. The bulk density of each fraction ( $\text{kg m}^{-3}$ ) was calculated

by placing each fraction in a cylindrical container with a volume of 5.0 dm<sup>3</sup> (cones/seed wings) or 0.5 dm<sup>3</sup> (remaining fractions) and determining their net mass.



**Figure 1.** Stages of seed extraction from cones and generation of residues.



**Figure 2.** Samples of cones and seed extraction residues (LCSc—woody seed scales of larch cones; LCS—central larch cone stems; SC—spruce cones; PC—pine cones; RP—residues from precleaning winged pine seeds; SW—pine seed wings; RC—residues from cleaning dewinged pine seeds; ES—empty pine seeds).

The moisture content of each fraction was determined by drying the samples in a BINDER FD 53 oven (Tuttlingen, Germany) at a temperature of 105 °C (%)

(PN-EN ISO 18134-1:2015-11 [30]) and weighing the samples on RADWAG WBT 2000 laboratory scales (Radom, Poland) to the nearest 0.01 g. The samples were broken and ground in a Retsch SM 200 cutting mill (Haan, Germany) equipped with a bottom sieve with 1.0 mm mesh size. The mass of each sample was reduced to around 50 g according to Standard PN-EN ISO 14780:2017-07 [31]. The samples were stored in closed laboratory containers until further analysis.

The higher heating value (HHV) of biomass in each fraction was determined by the thermodynamic method in an IKA C2000 basic calorimeter (Taufen, Germany). The lower heating value (LHV) was calculated based on the HHV and the previously determined hydrogen content and moisture content (PN-EN ISO 18125:2017-07 [32]).

The ash content of the biomass was determined in an ELTRA TGA-Thermotest thermogravimetric analyzer (Neuss, Germany) according to Standard PN-EN ISO 18122:2016-01 [33]. The total content of carbon (C), hydrogen (H), and sulfur (S) in seed extraction residues was determined with the ELTRA CHS-500 elemental analyzer (Neuss, Germany) according to Standards PN-EN ISO 16948:2015-07 [34] and PN-EN ISO 16994:2016-10 [35]. The device was calibrated using Eltra Coal Standard 92510-29. Nitrogen (N) content was determined by the Kjeldahl method with a BUCHI K-435 digestion unit and a BUCHI B-324 distillation unit (Flawil, Switzerland). Chlorine (Cl) content was determined with the use of Eschka's mixture according to Standard PN-ISO 587:2000 [36]. Ground samples with the addition of Eschka's mixture were combusted in a muffle furnace at a temperature of 650 °C. The samples were mineralized and neutralized with nitric acid, and they were titrated with silver nitrate in the presence of potassium dichromate, in accordance with Mohr's method.

All laboratory analyses were conducted in triplicate for each type of seed extraction residue.

### 2.3. Statistical Analysis

The results were processed statistically by one-way analysis of variance. Arithmetic means were calculated for all examined parameters. Homogeneous groups were determined by Tukey's HSD test at a significance level of  $p < 0.05$ . Descriptive statistics (mean, median, minimum value, maximum value, lower quartile, upper quartile, standard deviation, and coefficient of variation) were also determined for the entire dataset. Seed extraction residues were subjected to a hierarchical cluster analysis. Before the cluster analysis, data were standardized in matrix columns. Data were clustered by Ward's method. Subsets (clusters) were determined with the use of Sneath's criterion. Two cut-off lines were applied at  $2/3 D_{\max}$  and  $1/3 D_{\max}$ , where  $D_{\max}$  is the maximum distance  $D$  between clusters. All statistical analyses were conducted and a dendrogram was constructed in Statistica 13 (TIBCO Software Inc., Palo Alto, CA, USA).

## 3. Results and Discussion

### 3.1. Proportions (%) of Scots Pine Seed Extraction Residues (Percentage by Weight)

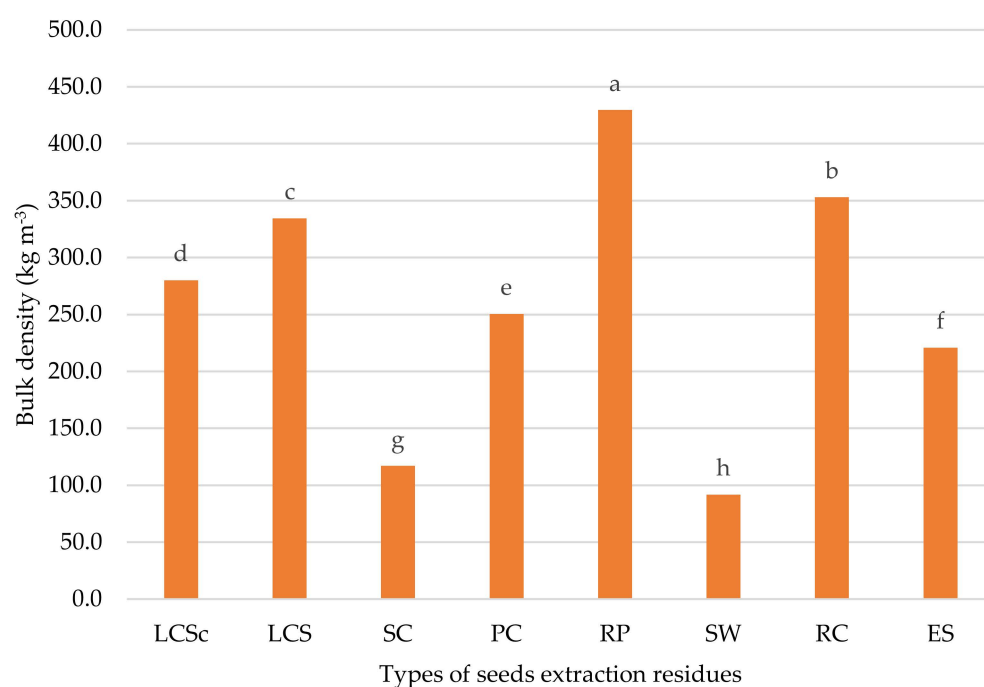
The proportions of the fractions of Scots pine seed extraction residues (percentage by weight) are presented in Table 1. Spent cones accounted for more than 98% of all fractions, while the remaining fractions represented around 0.5–0.6% each, excluding empty seeds that accounted for less than 0.1% of the total weight of all Scots pine waste biomass fractions. Minor differences can be observed in the proportions of waste fractions from cleaning dewinged seeds between batches of Scots pine cones, but these variations do not significantly affect the share of spent cones that constitute the dominant fraction in terms of both mass and volume (own study, data not published). According to Tyszkiewicz [37], the volume of spruce and larch cones doubles after drying. In larch waste biomass, the proportion of the empty seed fraction is somewhat higher because the seed coat has a much higher share of seed mass [38]. Therefore, cleaned larch seeds have to be sorted into size fractions before the final separation of empty seeds.

**Table 1.** The proportions (%) of Scots pine seed extraction residues (percentage by weight).

Seed Extraction Residues	Percentage
cones	98.35
waste from precleaning winged seed	0.59
seed wings	0.46
waste from seed cleaning	0.51
empty seeds	0.09

### 3.2. Bulk Density of Seed Extraction Residues

The fractions of seed extraction residues differed significantly in bulk density (Figure 3).



**Figure 3.** Bulk density of forest tree seed extraction residues (LCSc-woody seed scales of larch cones; LCS-central larch cone stems; SC-spruce cones; PC-pine cones; RP-residues from precleaning winged pine seeds; SW-pine seed wings; RC-residues from cleaning dewinged pine seeds; ES-empty pine seeds; <sup>a-h</sup> homogenous groups for the main source of variation).

Bulk density was highest in the waste fraction from precleaning winged seeds of Scots pine, which was composed of needles, cone fragments, and other impurities. Bulk density was also relatively high in the waste fraction from cleaning dewinged seeds and in the fraction of central larch cone stems. The seed wing fraction was characterized by the lowest bulk density. The analyzed parameter was only 25% higher in spent spruce cones. Other researchers [39] also found that bulk density was twice higher in empty pine cones than in empty spruce cones. In the analyzed tree species, the bulk density of cones can differ considerably across geographic locations [40]. The values of the bulk density of cones, noted in this study, are comparable to the bulk density of woodchips. In the work of Stolarski et al. [10], the bulk density of woodchips was determined to be in the range of 245.47–258.64 kg m<sup>-3</sup>. In turn, Spinelli et al. [41] found that the bulk density of woodchips from various species of forest trees ranged from 266 to 347 kg m<sup>-3</sup>. The bulk density of spruce cones increased more than six-fold (to approx. 670 kg m<sup>-3</sup>) after pelletizing (data not published). Such a big difference can be attributed to the fact that empty spruce cones are large and rigid and have thin scales that are deflected away from the cone axis, which causes them to snag and creates large empty spaces during storage [42].

### 3.3. Thermophysical Properties of Seed Extraction Residues

All fractions of seed extraction residues differed significantly in all thermophysical properties, i.e., moisture content (MC), content of fixed carbon (FC), volatile matter (VM) and ash, as well as the higher heating value (HHV) and the lower heating value (LHV) (Table 2).

**Table 2.** Thermophysical characteristics of solid biofuels derived from different types of forest tree seed extraction residues.

Types of Seed Extraction Residues	MC (%)	Ash (% DM)	FC (% DM)	VM (% DM)	HHV (GJ Mg <sup>-1</sup> DM)	LHV (GJ Mg <sup>-1</sup> )
Woody seed scales of larch cones	6.86 <sup>d</sup> ± 0.110	0.99 <sup>f</sup> ± 0.021	28.59 <sup>b</sup> ± 0.353	70.42 <sup>e</sup> ± 0.333	20.79 <sup>e</sup> ± 0.009	18.00 <sup>c</sup> ± 0.037
Central larch cone stems	9.99 <sup>a</sup> ± 0.054	0.71 <sup>g</sup> ± 0.017	28.08 <sup>c</sup> ± 0.031	71.20 <sup>d</sup> ± 0.048	20.77 <sup>e</sup> ± 0.027	17.33 <sup>d</sup> ± 0.039
Spruce cones	6.93 <sup>d</sup> ± 0.055	1.35 <sup>e</sup> ± 0.008	31.93 <sup>a</sup> ± 0.097	66.72 <sup>g</sup> ± 0.105	21.07 <sup>cd</sup> ± 0.025	18.29 <sup>b</sup> ± 0.004
Pine cones	8.98 <sup>b</sup> ± 0.008	0.93 <sup>f</sup> ± 0.035	24.06 <sup>e</sup> ± 0.170	75.02 <sup>b</sup> ± 0.205	20.30 <sup>f</sup> ± 0.036	17.08 <sup>e</sup> ± 0.048
Residues from precleaning winged pine seeds	7.40 <sup>c</sup> ± 0.250	17.69 <sup>a</sup> ± 0.015	17.66 <sup>h</sup> ± 0.025	64.66 <sup>h</sup> ± 0.040	21.14 <sup>c</sup> ± 0.073	18.28 <sup>b</sup> ± 0.123
Pine seed wings	9.85 <sup>a</sup> ± 0.024	2.27 <sup>d</sup> ± 0.080	25.50 <sup>d</sup> ± 0.220	72.23 <sup>c</sup> ± 0.300	20.91 <sup>de</sup> ± 0.091	17.47 <sup>d</sup> ± 0.077
Residues from cleaning dewinged pine seeds	8.72 <sup>b</sup> ± 0.191	3.63 <sup>c</sup> ± 0.080	18.55 <sup>g</sup> ± 0.110	77.82 <sup>a</sup> ± 0.030	25.43 <sup>a</sup> ± 0.122	21.62 <sup>a</sup> ± 0.158
Empty pine seeds	10.02 <sup>a</sup> ± 0.143	7.93 <sup>b</sup> ± 0.050	22.56 <sup>f</sup> ± 0.015	69.52 <sup>f</sup> ± 0.035	21.81 <sup>b</sup> ± 0.026	18.21 <sup>bc</sup> ± 0.016

<sup>a-h</sup> Homogenous groups for the main source of variation; ±—standard deviation.

All types and fractions of seed extraction residues were characterized by a very low moisture content in the range of 6.86–10.02% (Table 2). Moisture content was highest in the empty seed fraction obtained after the final cleaning step in a gravity separator, and in the seed wing fraction separated in the wet dewinging process. Woody seed scales of larch cones and spent spruce cones formed a homogeneous group with the significantly lowest moisture content. Only some types of biomass are characterized by such a low moisture content [39,40]. Seed extraction residues are classified as secondary forest biomass waste [22]. This type of waste biomass is obtained after thermal (spruce and pine) or thermal and mechanical (larch) seed extraction processes. In drying cabinets, cones are dried under controlled conditions to open up the scales (woody seed scales are deflected away from the cone axis). Dried cones have a low moisture content, and therefore they can be used as solid biofuel [8]. In the present study, spent spruce cones and larch cone stems were characterized by significantly lower moisture content than Scots pine cones and woody scales of larch cones. In contrast, Aniszewska and Gendek [39] found that open spruce cones had significantly higher moisture content than open Scots pine and larch cones. These discrepancies could be attributed to different cone-drying processes in other seed extraction plants. Due to their low moisture content, seed extraction residues are better suited for energy generation than other types of forest biomass [8,10,11,43]. The moisture content of seed extraction residues is similar to that of processed solid biofuels, such as pellets and briquettes derived from forest waste biomass [7,44–46].

Similarly to bulk density, considerable differences were also observed in the ash content of seed extraction residues (Table 2). These differences resulted from a very high content of minerals in waste from precleaning winged seeds (17.69% DM), empty seeds (7.93% DM), and waste from cleaning dewinged seeds (2.27% DM). The lowest ash content was noted in larch cone stems, followed by spent Scots pine cones and woody scales of larch cones, which formed homogeneous group *f*. In these fractions, the concentration of minerals did not exceed 1% DM. Ash content was relatively low (0.93–1.35% DM) in cones (pine and spruce) and cone fragments (larch), which is a desirable trait in solid biofuels [7]. The examined cones contained less ash than other types of forest biomass, including timber and sawdust of the same tree species, and their ash content was similar to that noted in certain types of woodchips [46–48]. The concentration of ash in seed wings, waste from cleaning dewinged seeds, and empty seeds was similar to that reported in pine and larch bark [47]. The high ash content of waste from precleaning winged seeds could be associated with the cone harvesting method. Depending on the type of seed stock, cones are harvested

from standing or fallen trees. Cones harvested from fallen trees are often significantly contaminated with sand and soil [49].

Fixed carbon (FC) content was highest in spruce cones, followed by woody seed scales and stems of larch cones (Table 2). Fixed carbon content was lowest in waste from precleaning winged Scots pine seeds and waste from cleaning dewinged Scots pine seeds (17.7% and 18.6% DM, respectively). The difference between fractions with the highest (spruce cones) and lowest FC content was 14.2 pp, i.e., nearly twice the lower value of FC in seed extraction residues. The content of VM was highest in waste from cleaning dewinged seeds, followed by spent Scots pine cones (Table 1), and lowest in waste from precleaning winged seeds and in empty seeds. The difference between extreme values of VM was 13.1 pp, which accounted for less than 1/5 of the lower value of this parameter. Fixed carbon content and VM content are closely related traits due to the method of their determination [50]. The content of VM in the cones of the examined tree species was considerably lower than that reported by Patel et al. [51], and it was similar to that noted in bark [52]. The observed differences in the content of FC and VM in the analyzed seed extraction residues can be attributed to differences in their anatomical structure [8].

The HHV was significantly higher in waste from cleaning dewinged seeds than in the remaining fractions of seed extraction residues (Table 2). Empty Scots pine seeds were characterized by the second highest HHV, which was 17% lower relative to waste from cleaning dewinged seeds. The analyzed parameter was lowest in Scots pine cones, and it was only somewhat higher in both fractions of larch cones (20.77–20.79 GJ Mg<sup>-1</sup> DM), which formed a homogeneous group in this respect. The HHV of Scots pine cones was 25% lower than that noted in waste biomass from cleaning dewinged seeds.

The waste from cleaning dewinged seeds was also characterized by the significantly highest LHV relative to the remaining fractions (Table 2). Spruce cones, waste from precleaning winged seeds, and empty seeds formed homogeneous group *b* in terms of their LHV values. The LHV of empty seeds did not differ significantly from that of woody seed scales of larch cones. The analyzed parameter was lowest in spent Scots pine cones. Similar values of HHV and LHV in the cones of the examined tree species were reported by other researchers [7,35,41]. The HHV and LHV of conifer cones were higher than the values noted in pinewood chips, other types of sawmill waste (sawdust, woodchips) from coniferous trees, and the biomass of forest residues [10,47,52]. These parameters can be increased through torrefaction [53].

### 3.4. Elemental Composition of Seed Extraction Residues

The content of carbon, hydrogen, sulfur, nitrogen, and chlorine differed significantly in the biomass of the analyzed fractions of seed extraction residues (Table 3).

**Table 3.** Elemental composition of solid biofuels derived from different types of forest tree seed extraction residues.

Types of Seed Extraction Residues	C (% DM)	H (% DM)	S (% DM)	N (% DM)	Cl (% DM)
Woody seed scales of larch cones	53.35 <sup>e</sup> ± 0.010	6.27 <sup>bc</sup> ± 0.026	0.029 <sup>f</sup> ± 0.0010	0.58 <sup>f</sup> ± 0.008	0.035 <sup>c</sup> ± 0.0022
Central larch cone stems	53.33 <sup>e</sup> ± 0.026	6.11 <sup>de</sup> ± 0.011	0.019 <sup>g</sup> ± 0.0003	0.40 <sup>g</sup> ± 0.002	0.020 <sup>d</sup> ± 0.0009
Spruce cones	58.58 <sup>b</sup> ± 0.422	6.05 <sup>e</sup> ± 0.076	0.043 <sup>e</sup> ± 0.0005	0.69 <sup>e</sup> ± 0.018	0.041 <sup>b</sup> ± 0.0021
Pine cones	54.52 <sup>d</sup> ± 0.055	6.34 <sup>b</sup> ± 0.075	0.029 <sup>f</sup> ± 0.0020	0.44 <sup>g</sup> ± 0.025	0.010 <sup>e</sup> ± 0.0000
Residues from precleaning winged pine seeds	53.22 <sup>e</sup> ± 0.110	5.89 <sup>f</sup> ± 0.000	0.069 <sup>d</sup> ± 0.0005	2.13 <sup>a</sup> ± 0.082	0.030 <sup>c</sup> ± 0.0000
Pine seed wings	55.41 <sup>c</sup> ± 0.100	6.18 <sup>cd</sup> ± 0.000	0.086 <sup>c</sup> ± 0.0015	1.96 <sup>b</sup> ± 0.008	0.010 <sup>e</sup> ± 0.0000
Residues from cleaning dewinged pine seeds	64.30 <sup>a</sup> ± 0.085	7.41 <sup>a</sup> ± 0.015	0.115 <sup>b</sup> ± 0.0015	1.26 <sup>c</sup> ± 0.004	0.010 <sup>e</sup> ± 0.0000
Empty pine seeds	54.63 <sup>d</sup> ± 0.250	6.39 <sup>b</sup> ± 0.035	0.203 <sup>a</sup> ± 0.0020	0.82 <sup>d</sup> ± 0.000	0.055 <sup>a</sup> ± 0.0050

<sup>a–g</sup> Homogenous groups for the main source of variation; ±—standard deviation.

Waste biomass from cleaning dewinged seeds was characterized by the highest carbon content, which was 5.7 pp higher than in spruce cones and 11 pp higher than in both larch

cone fractions and waste from precleaning Scots pine seeds. The carbon content of cones ranged from 53.3% DM (larch) to 58.6% DM (spruce).

Hydrogen content was below 6% DM only in waste biomass from precleaning winged pine seeds (Table 3). This parameter was highest in waste from cleaning dewinged seeds. Hydrogen content was highest in Scots pine cones (6.34% DM) and woody seed scales of larch cones, which formed homogeneous group *b* in this respect. Hydrogen content was lowest in spruce cones (6.05% DM) and larch cone stems, which formed homogeneous group *e* within the terms of this parameter.

Empty seeds and waste biomass from cleaning dewinged seeds were characterized by the highest sulfur content, which was an order of magnitude higher than in the remaining fractions of seed extraction residues. Sulfur concentration was lowest in the biomass of larch cone stems. In the evaluated cones, sulfur content ranged from 0.019% DM (larch cone stems) to 0.043% DM (spruce cones). Sulfur concentration was identical in Scots pine cones and woody seed scales of larch cones (Table 3).

The nitrogen content of the analyzed fractions ranged from 0.40% DM (larch cone stems) to 2.13% DM (waste from precleaning winged Scots pine seeds) (Table 2). Three residue fractions (waste biomass from seed precleaning and cleaning, and Scots pine seed wings) were characterized by increased nitrogen concentrations (1.26–2.13% DM). Nitrogen concentration was significantly lower in cones than in the remaining fractions of seed extraction residues, and it ranged from 0.40–0.44% DM (homogenous group *g* comprising larch cone stems and Scots pine cones) to 0.69% DM (spruce cones).

Chlorine concentration was highest in empty Scots pine seeds (0.055% DM), and lowest in Scots pine cones, Scots pine seed wings, and waste biomass from cleaning dewinged Scots pine seeds (0.010% DM) (Table 3). In the analyzed cones, chlorine content was lowest in Scots pine cones (0.010% DM) and highest in spruce cones (0.041% DM). The elemental composition of cones was similar to that reported in the literature. The examined cones were somewhat more abundant in carbon and hydrogen than cones harvested in other regions and seasons [46,51]. The sulfur and nitrogen content of the analyzed cones was somewhat higher than that reported in forest waste biomass [10,11,47]. The concentration of sulfur was highest in empty seeds, and similar observations were made in other studies of forest waste biomass [54].

### 3.5. General Characteristics of Solid Biofuels Derived from Seed Extraction Residues

The descriptive statistics of the examined fractions of Scots pine seed extraction residues are presented in Table 4.

**Table 4.** Thermophysical characteristics of solid biofuels derived from Scots pine seed extraction residues.

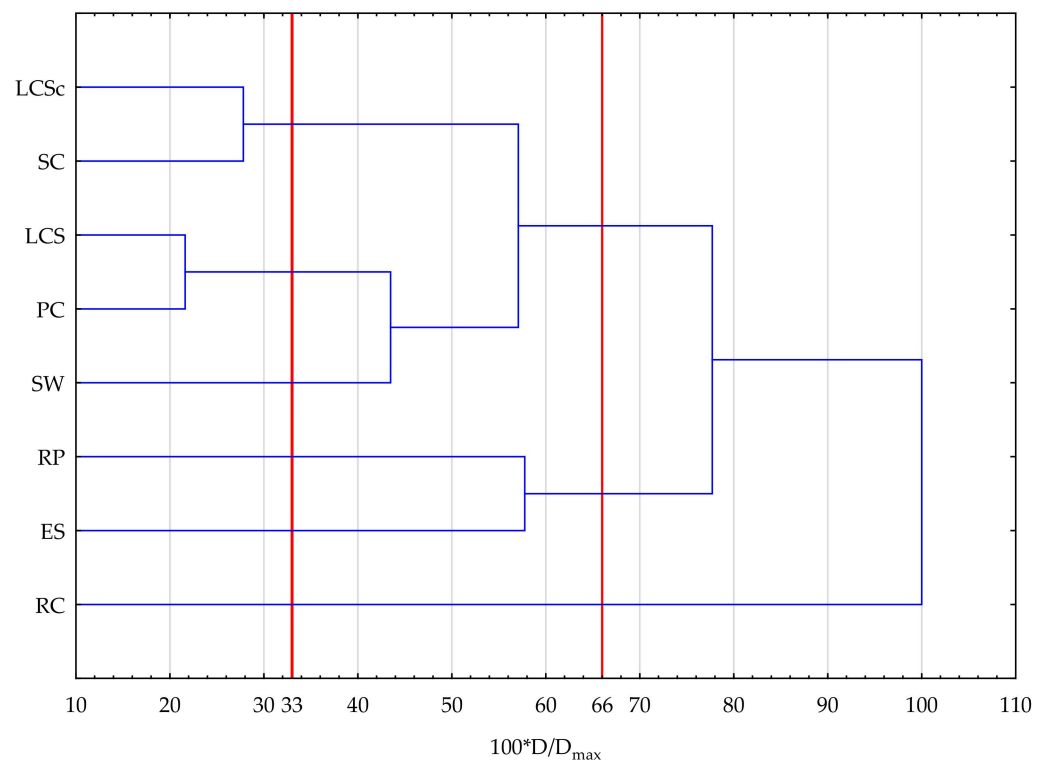
Parameter	Mean	Median	Minimum Value	Maximum Value	Lower Quartile	Upper Quartile	Standard Deviation	Coefficient of Variation (%)
BD (kg m <sup>-3</sup> )	269.13	251.38	88.90	433.61	215.54	362.04	119.91	44.55
MC (%)	8.99	8.98	7.15	10.16	8.53	9.87	0.98	10.89
Ash (% DM)	6.49	3.63	0.89	17.70	2.19	7.98	6.29	96.88
FC (% DM)	21.66	22.56	17.63	25.72	18.44	24.23	3.18	14.66
VM (% DM)	71.85	72.23	64.62	77.85	69.48	75.22	4.70	6.54
HHV (GJ Mg <sup>-1</sup> DM)	21.92	21.14	20.27	25.55	20.82	21.84	1.89	8.61
LHV (GJ Mg <sup>-1</sup> )	18.53	18.20	17.04	21.78	17.40	18.40	1.67	8.99
C (% DM)	56.41	54.63	53.11	64.38	54.38	55.51	4.14	7.35
H (% DM)	6.44	6.34	5.89	7.42	6.18	6.42	0.53	8.26
S (% DM)	0.10	0.09	0.03	0.21	0.07	0.12	0.06	60.42
N (% DM)	1.32	1.26	0.41	2.21	0.82	1.97	0.67	50.90
Cl (% DM)	0.02	0.01	0.01	0.06	0.01	0.03	0.02	80.42

Ash content was the most variable trait due to the very high content of minerals in waste biomass from seed precleaning. In this fraction, ash content varies depending on

the quality (purity) of processed cones. Even after precleaning, a certain amount of ash is retained on the surface of cones, and it is released when seeds are removed from cones after thermal processing. Ash is removed together with the extracted seeds, and it is transferred to the precleaned waste fraction.

Considerable variation was also noted in the chlorine, sulfur, and nitrogen content of seed extraction residues (coefficient of variation exceeded 50%). Sulfur concentration ranged from 0.03% to 0.21% DM, which points to a seven-fold difference between minimum and maximum values of this parameter. Nitrogen content ranged from 0.41% to 2.21% DM, whereas chlorine concentration ranged from 0.01% to 0.06%, and the minimum and maximum values of these parameters differed nearly six-fold.

The cluster analysis, based on the values of all parameters of the eight fractions of seed extraction residues, at the cut-off point of  $2/3 D_{\max}$  produced three clusters (Figure 4). The first cluster consisted of spent cones of all tree species (Scots pine, Norway spruce, and two fractions of European larch cones) and Scots pine seed wings. The second cluster was composed of waste biomass from precleaning winged Scots pine seeds and empty Scots pine seeds. The third cluster contained waste from cleaning dewinged Scots pine seeds. Six clusters were identified when a cut-off point of  $1/3 D_{\max}$  was applied to increase the accuracy of the cluster analysis. The first cluster comprised woody seed scales of larch cones and spruce cones. The second cluster contained larch cone stems and spent Scots pine cones. The remaining fractions of seed extraction residues (RP, SW, RC, ES) formed separate clusters.



**Figure 4.** The dendrogram of a hierarchical cluster analysis showing the similarities between fractions of seed extraction residues. Red vertical lines represent Sneath's criterion ( $2/3 D_{\max}$  and  $1/3 D_{\max}$ ). D—linage distance;  $D_{\max}$ —maximum linage distance.

Unlike energy from fossil fuels, energy from forest waste biomass contributes to decreasing the emissions of carbon dioxide and other greenhouse gases [55]. Direct combustion of forest waste biomass leads to the emissions of carbon dioxide, sulfur dioxide, and other substances and particulate matter [46], but their levels are lower than those noted during the combustion of fossil fuels [56,57]. The quality of forest waste biomass used as a biofuel source has to be closely monitored to improve the efficiency of energy genera-

tion [46]. An analysis of seed extraction residues revealed that they were highly diversified, but the majority of them were characterized by desirable thermophysical properties and elemental composition.

The use of forest waste biomass may be associated with adverse environmental impacts on biodiversity and local habitats [22]. However, cones are not harvested on a large scale. According to Aniszewska et al. [40], approximately 0.6–1.2 million tons of cones could be collected in Polish forests each year, provided that it would not negatively affect soil fertility or forest regeneration.

Due to its scale and specificity, the harvest of seed cones, described in this study, does not pose such a threat [2,58]. The analyzed seed extraction residues are a by-product of cone processing.

#### 4. Conclusions

This study was undertaken to evaluate the thermophysical properties and elemental composition of eight fractions of seed extraction residues obtained in a professional seed extraction plant. The type and composition of waste biomass is partly determined by tree species, processing line, and the applied seed processing method. Most Polish seed extraction plants operate similar types of equipment. The spent cone fraction is characterized by the largest mass and volume. Depending on tree species and the applied extraction method, empty cones remain intact, break into smaller fragments, or are mechanically crushed into differently sized fractions. Very large quantities of cone waste biomass are generated during seed extraction, and they have to be effectively managed.

The specific weight of all waste biomass fractions was relatively low, and they could be used on the local market. They could also be processed into pellets and briquettes to decrease their volume, thus permitting their wider use.

The thermophysical properties and elemental composition of seed extraction residues varied considerably. The fractions of empty cones of the examined tree species and Scots pine seed wings were characterized by a low moisture content, low concentrations of ash and sulfur, high content of fixed carbon and volatile matter, and high calorific value, which makes them suitable for energy generation, with a low risk of negative environmental impacts.

The remaining fractions, which have a high content of ash, nitrogen, and sulfur, should be used for other purposes, for example in the production of compost.

**Author Contributions:** Conceptualization, J.K. and Z.S.; methodology, J.K. and Z.S.; software, J.K.; validation, J.K. and Z.S.; formal analysis, J.K.; investigation, J.K. and Z.S.; resources, J.K. and Z.S.; data curation, J.K.; writing—original draft preparation, J.K. and Z.S.; writing—review and editing, J.K.; visualization, J.K.; supervision, J.K.; project administration, J.K.; funding acquisition, J.K. All authors have read and agreed to the published version of the manuscript.

**Funding:** The results presented in this paper were obtained as part of a comprehensive study financed by the University of Warmia and Mazury in Olsztyn, Faculty of Agriculture and Forestry, Department of Genetics, Plant Breeding and Bioresource Engineering (grant No. 30.610.007-110).

**Institutional Review Board Statement:** Not applicable.

**Informed Consent Statement:** Not applicable.

**Data Availability Statement:** Data are contained within the article.

**Acknowledgments:** We would like to thank the staff of the Department of Genetics, Plant Breeding and Bioresource Engineering and the Seed Extraction Plant for their technical support during the experiment.

**Conflicts of Interest:** The authors declare no conflicts of interest.

## References

1. Directive 2009/28/EC of the European Parliament and of the Council of 23 April 2009 on the Promotion of the Use of Energy from Renewable Sources and Amending and Subsequently Repealing Directives 2001/77/EC and 2003/30/EC. Official Journal of the European Union, Strasbourg, France. 2009. Available online: <https://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2009:140:0016:0062:en:PDF> (accessed on 10 January 2024).
2. Statistics Poland. *Energy from Renewable Sources in 2022*; Statistics Poland: Warsaw, Poland, 2023; p. 67.
3. Picchio, F.; Latterini, R.; Venanzi, W.; Stefanoni, A.; Suardi, D.; Tocci, L.; Pari, L. Pellet production from woody and non-woody feedstocks: A review on biomass quality evaluation. *Energies* **2020**, *13*, 2937. [\[CrossRef\]](#)
4. Pedišius, N.; Praspaliauskas, M.; Pedišius, J.; Dzenajavičienė, E.F. Analysis of Wood Chip Characteristics for Energy Production in Lithuania. *Energies* **2021**, *14*, 3931. [\[CrossRef\]](#)
5. Enström, J.; Eriksson, A.; Eliasson, L.; Larsson, A.; Olsson, L. Wood chip supply from forest to port of loading—A simulation study. *Biomass Bioenergy* **2021**, *152*, 106182. [\[CrossRef\]](#)
6. Stolarski, M.J.; Dudziec, P.; Krzyżaniak, M.; Olba-Zięty, E. Solid Biomass Energy Potential as a Development Opportunity for Rural Communities. *Energies* **2021**, *14*, 3398. [\[CrossRef\]](#)
7. Stolarski, M.J.; Stachowicz, P.; Dudziec, P. Wood pellet quality depending on dendromass species. *Renew. Energy* **2022**, *199*, 498–508. [\[CrossRef\]](#)
8. Stolarski, M.J.; Dudziec, P.; Olba-Zięty, E.; Stachowicz, P.; Krzyżaniak, M. Forest Dendromass as Energy Feedstock: Diversity of Properties and Composition Depending on Systematic Genus and Organ. *Energies* **2022**, *15*, 1442. [\[CrossRef\]](#)
9. Sherman, L.A.; Page-Dumroese, D.S.; Coleman, M.D. Idaho forest growth response to post-thinning energy biomass removal and complementary soil amendments. *GCB Bioenergy* **2017**, *10*, 246–261. [\[CrossRef\]](#)
10. Stolarski, J.; Wierzbicki, S.; Nitkiewicz, S.; Stolarski, M.J. Wood Chip Production Efficiency Depending on Chipper Type. *Energies* **2023**, *16*, 4894. [\[CrossRef\]](#)
11. Dudziec, P.; Stachowicz, P.; Stolarski, M.J. Diversity of properties of sawmill residues used as feedstock for energy generation. *Renewable Energy* **2023**, *202*, 822–833. [\[CrossRef\]](#)
12. Ibitoye, S.E.; Mahamood, R.M.; Jen, T.-C.; Loha, C.; Akinlabi, E.T. An overview of biomass solid fuels: Biomass sources, processing methods, and morphological and microstructural properties. *J. Bioresour. Bioprod.* **2023**, *8*, 333–360. [\[CrossRef\]](#)
13. Stafford, W.; De Lange, W.; Nahman, A.; Chuniwall, V.; Lekha, P.; Andrew, J.; Johakimu, J.; Sithole, B.; Trotter, D. Forestry biorefineries. *Renew. Energy* **2020**, *154*, 461–475. [\[CrossRef\]](#)
14. Costa, A.R.; Lourenço, A.; Patrício, H.; Quilhó, T.; Gominho, J. Valorization of Pine Nut Industry Residues on a Biorefinery Concept. Valorization of Pine Nut Industry Residues on a Biorefinery Concept. *Waste Biomass Valor* **2023**, *14*, 4081–4099. [\[CrossRef\]](#)
15. Wajs, A.; Urbańska, J.; Zaleskiewicz, E.; Bonikowski, R. Composition of Essential Oil from Seeds and Cones of *Abies alba*. *Nat. Prod. Commun.* **2010**, *5*, 1291–1294. [\[CrossRef\]](#)
16. Kar, T.; Kaygusuz, Ö.; Güney, M.Ş.; Cuce, E.; Keleş, S.; Shaik, S.; Owolabi, A.B.; Nsafon, B.E.K.; Ogunsua, J.M.; Huh, J.-S. Fast Pyrolysis of Tea Bush, Walnut Shell, and Pine Cone Mixture: Effect of Pyrolysis Parameters on Pyrolysis Crop Yields. *Sustainability* **2023**, *15*, 13718. [\[CrossRef\]](#)
17. Sahin, H.T.; Yalcin, O.U. Conifer Cones: An Alternative Raw Material for Industry. *Br. J. Pharm. Res.* **2017**, *17*, 3415. [\[CrossRef\]](#)
18. Statistics Poland. *Statistical Yearbook of Forestry*; Statistics Poland: Warsaw/Białystok, Poland, 2023; p. 348.
19. Fonder, W.; Matras, J.; Załęski, A. *Leśna Baza Nasienna w Polsce*; CILP: Warszawa, Poland, 2007; p. 302.
20. Statistics Poland. *Forestry in 2022*; Statistics Poland: Warsaw/Białystok, Poland, 2023; p. 5.
21. Aniszewska, M.; Kuszpit, D. Analysis of acquisition and potential usage of conifer cones from Polish seed extraction houses between 2009–2012. *Ann. Wars. Univ. Life Sci. SGGW Agric.* **2015**, *65*, 93–101.
22. Register of Issued Certificates of Origin for Forest Reproductive Material. Available online: <https://rejstry.bnl.gov.pl/registry/CERT> (accessed on 29 February 2024).
23. Titus, B.D.; Brown, K.; Helmissaari, H.S.; Vanguelova, E.; Stupak, I.; Evans, A.; Clarke, N.; Guidi, C.; Bruckman, V.J.; Varnagiryte-Kabasinskiene, I.; et al. Sustainable forest biomass: A review of current residue harvesting guidelines. *Energy Sustain. Soc.* **2021**, *11*, 10. [\[CrossRef\]](#)
24. Nicholls, D.L.; Monserud, R.A.; Dykstra, D.P. Biomass utilization for bioenergy in the western United States. *For. Prod. J.* **2008**, *58*, 6–16.
25. Brack, D. *The Impacts of the Demand for Woody Biomass for Power and Heat on Climate and Forests*; Chatham House: London, UK, 2017.
26. Saidur, R.; Abdelaziz, E.A.; Demirbas, A.; Hossain, M.S.; Mekhilef, S. A review on biomass as a fuel for boilers. *Renew. Sustain. Energy Rev.* **2011**, *15*, 2262–2289. [\[CrossRef\]](#)
27. Garcia-Maraver, A.; Zamorano, M.; Fernandes, U.; Rabaçal, M.; Costa, M. Relationship between fuel quality and gaseous and particulate matter emissions in a domestic pellet-fired boiler. *Fuel* **2014**, *119*, 141–152. [\[CrossRef\]](#)
28. Winter, F.; Wartha, C.; Hofbauer, H. NO and N<sub>2</sub>O formation during the combustion of wood, straw, malt waste and peat. *Bioresour Technol.* **1999**, *70*, 39–49. [\[CrossRef\]](#)
29. Saleh, S.B.; Flensburg, J.P.; Shoulaifar, T.K.; Sárossy, Z.; Hansen, B.B.; Egsgaard, H.; DeMartini, N.; Jensen, P.A.; Glarborg, P.; Dam-Johansen, K. Release of Chlorine and Sulfur during Biomass Torrefaction and Pyrolysis. *Energy Fuels* **2014**, *28*, 3738–3746. [\[CrossRef\]](#)
30. *PN-EN ISO 18134-1:2015-11*; Biofuels—Determination of Moisture Content—Dryer Method—Part 1: Total Moisture—Reference Method. Polish Standardization Committee: Warsaw, Poland, 2017.

31. PN-EN ISO 14780:2017-07; Solid Biofuels—Sample Preparation. Polish Standardization Committee: Warsaw, Poland, 2020.
32. PN-EN ISO 18125:2017-07; Solid Biofuels—Determination of Calorific Value. Polish Standardization Committee: Warsaw, Poland, 2017.
33. PN-EN ISO 18122:2016-01; Solid Biofuels—Determination of Ash Content. Polish Standardization Committee: Warsaw, Poland, 2016.
34. PN-EN ISO 16948:2015-07; Solid Biofuels—Determination of Total Content of Carbon, Hydrogen and Nitrogen. Polish Standardization Committee: Warsaw, Poland, 2015.
35. PN-EN ISO 16994:2016-10; Solid Biofuels—Determination of Total Content of Sulfur and Chlorine. Polish Standardization Committee: Warsaw, Poland, 2016.
36. PN-ISO 587:2000; Solid Fuels—Determination of Chlorine Content Using the Eschka Mixture (In Polish). Polish Standardization Committee: Warsaw, Poland, 2000.
37. Tyszkiewicz, S. *Nasiennictwo Leśne*; Instytut Badawczy Leśnictwa: Warszawa, Poland, 1949; p. 358.
38. Tylek, P. Selected physical features and sorting criteria for European larch seeds. *Sylvan* **2004**, *4*, 27–33. [[CrossRef](#)]
39. Aniszewska, M.; Gendek, A. Logistics of the supplies of selected forest tree species' cones. Part 1. Cone density and substitution coefficient. *Ann. Wars. Univ. Life Sci. SGGW Agric.* **2016**, *67*, 121–130.
40. Aniszewska, M.; Gendek, A.; Zychowicz, W. Analysis of Selected Physical Properties of Conifer Cones with Relevance to Energy Production Efficiency. *Forests* **2018**, *9*, 405. [[CrossRef](#)]
41. Spinelli, R.; De Francesco, F.; Eliasson, L.; Jessup, E.; Magagnotti, N. An agile chipper truck for space-constrained operations. *Biomass Bioenergy* **2015**, *81*, 137–143. [[CrossRef](#)]
42. Aniszewska, M. Analysis of opening cones of selected coniferous trees. *Ann. Wars. Univ. Life Sci. SGGW Agric.* **2010**, *55*, 57–64.
43. Stankov, S.; Tasheva, S.; Fidan, H.; Bozadzhiev, B.; Dimov, M.; Stoyanova, A. Investigation of chemical composition, basic energy indices, and thermodynamic properties of unripe and ripe black pine (*Pinus nigra* Arn.) cones. *AIP Conf. Proc.* **2023**, *2889*, 070001. [[CrossRef](#)]
44. García, R.; Gil, M.V.; Rubiera, F.; Pevida, C. Pelletization of wood and alternative residual biomass blends for producing industrial quality pellets. *Fuel* **2019**, *251*, 739–753. [[CrossRef](#)]
45. Stachowicz, P.; Stolarski, M.J. Short rotation woody crops and forest biomass sawdust mixture pellet quality. *Ind. Crop. Prod.* **2023**, *197*, 116604. [[CrossRef](#)]
46. Malaťák, J.; Gendek, A.; Aniszewska, M.; Velebila, J. Emissions from combustion of renewable solid biofuels from coniferous tree cones. *Fuel* **2020**, *276*, 118001. [[CrossRef](#)]
47. Stolarski, M.J.; Krzyżaniak, M.; Olba-Zięty, E.; Stolarski, J. Changes in Commercial Dendromass Properties Depending on Type and Acquisition Time. *Energies* **2023**, *16*, 7973. [[CrossRef](#)]
48. Gündüz, G.; Saraçoğlu, N.; Aydemir, D. Characterization and elemental analysis of wood pellets obtained from low-valued types of wood. *Energy Sources Part A Recovery Util. Environ. Eff.* **2016**, *38*, 2211–2216. [[CrossRef](#)]
49. Gendek, A.; Malaťák, J.; Velebil, J. Effect of harvest method and composition of wood chips on their caloric value and ash content. *Sylvan* **2018**, *162*, 248–257. [[CrossRef](#)]
50. Jia, Y.; Wang, Y.; Zhang, Q.; Rong, H.; Liu, Y.; Xiao, B.; Guo, D.; Laghari, M.; Ruan, R. Gas-carrying enhances the combustion temperature of the biomass particles. *Energy* **2022**, *239*, 121956. [[CrossRef](#)]
51. Patel, D.K.; Katiyar, R.; Dwivedi, P.; Rathore, A.K.; Singh, A. Co-pyrolysis of pine-cone and chicken feathers: A study to determine kinetic parameters, thermodynamic properties, and potential synergistic effects. *Energy Sources Part A Recovery Util. Environ. Eff.* **2024**, *46*, 1644–1657. [[CrossRef](#)]
52. Senelwa, K.; Sims, R.E.H. Fuel characteristics of short rotation forest biomass. *Biomass Bioenergy* **1999**, *17*, 127–140. [[CrossRef](#)]
53. Aniszewska, M.; Gendek, A.; Hýsek, Š.; Malaťák, J.; Velebil, J.; Tamelová, B. Changes in the Composition and Surface Properties of Torrefied Conifer Cones. *Materials* **2020**, *13*, 5660. [[CrossRef](#)]
54. Nurek, T.; Gendek, A.; Roman, K. Forest residues as a renewable source of energy: Elemental composition and physical properties. *BioResources* **2019**, *14*, 6–20. [[CrossRef](#)]
55. Pour, N.; Webley, P.A.; Cook, P.J. Opportunities for Application of BECCS in the Australian Power Sector. *Appl. Energy* **2018**, *224*, 615–635. [[CrossRef](#)]
56. Froese, R.E.; Shonnard, D.R.; Miller, C.A.; Koers, K.P.; Johnson, D.M. An Evaluation of Greenhouse Gas Mitigation Options for Coal-Fired Power Plants in the US Great Lakes States. *Biomass Bioenergy* **2010**, *34*, 251–262. [[CrossRef](#)]
57. Karaj, S.; Rehl, T.; Leis, H.; Müller, J. Analysis of Biomass Residues Potential for Electrical Energy Generation in Albania. *Renew. Sustain. Energy Rev.* **2010**, *14*, 493–499. [[CrossRef](#)]
58. Chałupka, W.; Barzdajn, W.; Blonkowski, S.; Burczyk, J.; Fonder, W.; Gładzki, T.; Gryzłó, Z.; Kacprzak, P.; Kowalczyk, J.; Koziół, C.; et al. *Program of Conserving Forest Genetic Resources and Breeding of Trees in Poland for the Years 2011–2035*; The State Forests Information Centre: Warsaw, Poland, 2011; p. 144.

**Disclaimer/Publisher's Note:** The statements, opinions and data contained in all publications are solely those of the individual author(s) and contributor(s) and not of MDPI and/or the editor(s). MDPI and/or the editor(s) disclaim responsibility for any injury to people or property resulting from any ideas, methods, instructions or products referred to in the content.