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Thermophysical Properties and Elemental Composition of Black Locust, Poplar and Willow Biomass

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Abstract: Biomass is currently the main renewable energy source (RES) in the EU, particularly in Poland. Solid biomass for energy purposes is primarily sourced from forests, the wood processing industry, and agriculture. A significant source of this energy feedstock could also be short-rotation woody crops (SRWCs), including black locust, poplar, and willow. Since numerous factors determine the SRWC biomass quality, the current study aimed at assessing biomass thermophysical properties and elemental composition depending on the plant species, soil enrichment procedure, and the plant harvest rotation over a consecutive 12-year period of cultivation. The characteristics under study, including the moisture content, ash content, volatile matter, fixed carbon, higher heating value (HHV), and the carbon, hydrogen, nitrogen, sulfur, and chlorine contents, were significantly differentiated by all the main factors, i.e., the SRWC species, the soil enrichment procedure, the harvest rotation, and the interactions between these factors. The SRWC species accounted for the highest percentage of the variation in the biomass moisture content, ash content, HHV, and nitrogen content, while the harvest rotation made the largest contribution to the variation in carbon, hydrogen, and chlorine contents. The black locust biomass was characterized by the significantly lowest moisture content (an average of 38.89%) and the highest sulfur content (an average of 0.033% DM), nitrogen content (an average of 0.91% DM), and chlorine content (an average of 0.032% DM). However, poplar was characterized by the highest HHV (an average of 19.84 GJ Mg^{-1} DM) and the highest moisture content (56.52% DM), carbon content (56.52% DM), and ash content (an average of 1.67% DM). Willow was characterized by the lowest ash content (an average of 1.67% DM), a medium moisture content, and the lowest nitrogen content (an average of 0.38% DM) and chlorine content (an average of 0.19% DM).

Keywords: black locust; poplar; willow; moisture content; ash content; higher heating value; sulfur content; nitrogen content

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1. Introduction

For many years, the European Union (EU) has been placing great emphasis on the use of renewable energy sources (RESs). As early as 2009, the EU set a target [1] which obliged EU Member States to ensure a certain share of renewable energy in final gross energy consumption by 2020. For all the EU Member States, the share of renewable energy in final gross energy consumption was set at 20%, while for Poland, the target was set at 15%. It should be stressed that the statistical data indicate that these indices were obtained both at the EU level (over 22%) and in Poland (over 16%) [2]. Therefore, this should be regarded as a positive aspect in favor of increasing the use of RESs. Nevertheless, due to the predicted further increase in final global energy consumption, with the negative environmental impact of fossil fuels, RESs serve a crucial role in the post-fossil fuel era [3–6]. Moreover, due to the current political and economic situation, dramatic increases in the prices of fossil energy feedstocks, and the restrictions on their availability, RESs will play a strategic role in energy supply.

Therefore, all RES types (solar, hydro, wind, geothermal, biomass) are very important for developing modern technologies and increasing their share in the overall energy mix. The structure of primary energy production from renewable sources in 2020 in the EU was dominated by solid biofuels (40.59%), followed by wind energy (14.56%), hydroelectric power (12.71%), solar energy (7.02%), liquid biofuels (6.63%), biogas (6.28%), heat pumps (6.61%), municipal waste (3.93%), geothermal energy (2.4%), and ocean wave energy (0.02%). In Poland, on the other hand, solid biofuels also came first in this structure, but their share was decidedly the highest (71.61%). These were followed by wind energy (10.85%), liquid biofuels (7.79%), biogas (2.58%), heat pumps (2.38%), solar energy (1.99%), hydroelectric power (1.46%), municipal waste (1.15%), and geothermal energy (0.20%) [2]. The above data indicate that biomass is currently the main RES at the EU level (57.1%), particularly in Poland (83.1%). It should also be added that biomass is a consistent source of energy in comparison with other RESs. The biomass potential increases if it is a waste in its initial form and, thanks to modern conversion technologies, it becomes a valuable energy feedstock [7,8]. Currently, biomass for energy purposes is primarily sourced from forests, the wood processing industry, agriculture, and the agro-food industry [9,10]. A significant biomass source may also be the perennial industrial crops (PICs), including short-rotation woody crops (SRWCs) such as black locust, poplar, and willow [11–14]. These perennial plants regrow after successive harvests and exhibit low demand for nutrients and a reduced need for agrotechnical intervention, which makes them contributors to a reduction in greenhouse gas emissions and an increase in soil carbon storage [15–17].

Woody biomass, including SRWCs, in the form of chips, pellets, or briquettes, is used to generate thermal energy and electricity [18–20] and gasification [21]. It needs to be stressed, however, that each woody biomass conversion technology requires a stable supply of a feedstock of adequate quality. In general, woody biomass is considered a good feedstock for thermal and thermochemical conversion, although ash and other minerals can pose problems due to the formation of corrosion, deposits, and slag. In contrast, in the biochemical conversion processes, ash can reduce the efficiency of biomass pre-treatment. Therefore, it is of great practical importance to characterize the biomass properties and identify the sources of its variation [22,23].

The SRWC biomass quality is determined by numerous factors, with the main ones including the type and species of the plant from which it is obtained. Other important factors include the environmental factors, the plant fertilization type and levels, and, for SRWCs, the harvest rotation. Therefore, the current study aimed to assess the thermophysical properties and the elemental composition of SRWC biomass depending on: (i) the plant species, (ii) the soil enrichment procedure, and (iii) the plant harvest rotation over a consecutive 12-year period of cultivation. In addition, based on the information collected, an additional aim was to quantify the relative contribution of the above-mentioned factors and their interactions to explain the variation in the biomass characteristics under study.

2. Materials and Methods

2.1. Field Experiment and the Subject of the Study

The study was based on a 12-year field experiment from 2010–2021 in north-eastern Poland (53°59′ N, 21°04′ E) on fields belonging to the University of Warmia and Mazury (UWM) in Olsztyn. The experiment was established on poor-quality soil, classified as Brunic Arenosol (Dystric), formed from loose sand. The main factors of the study included three SRWC species, i.e., willow (*Salix viminalis* L.; the Żubr cultivar, registered at the Research Centre for Cultivar Testing in Słupia Wielka, Poland), poplar (*Populus nigra* × P. *Maximowiczii* Henry cv. Max-5), and black locust (*Robinia pseudoacacia* L.). The initial planting density for all three plant species was the same at 11,111 plants per hectare. Another study factor was the soil enrichment procedure, which included the following variants (plots): control with no soil enrichment (C); the application of lignin (L); mineral fertilization (F); the application of mycorrhiza inoculation (M); lignin + mineral fertilization (LF); mycorrhiza + mineral fertilization (MF); lignin + mycorrhiza (LM); lignin + mycorrhiza + mineral fertilization

(LMF). Lignin (a residue from paper product production) was applied at 13.3 Mg ha^{-1} only once, shortly before the establishment of the experiment in the spring of 2010. Live mycorrhizal mycelium, different for each species, was applied only once in early September 2010 in a liquid suspension at 30–35 cm^3 for each plant. However, mineral fertilization (N: 90 kg ha^{-1} N; P: 30 kg ha^{-1} P_2O_5; K: 60 kg ha^{-1} K_2O) was applied three times before the beginning of the plant growing period in April 2011, 2014, and 2018. More information on this field experiment is provided in the studies [24,25], which present the selected results from the first period of its implementation. This manuscript, however, presents the results for the SRWC biomass quality from three consecutive four-year harvest rotations. Therefore, another factor of this study was the consecutive SRWC harvest rotation: the first rotation, during which the plants grew in the years 2010–2013 and were harvested in December 2013; the second rotation, during which the plants grew in the years 2014–2017, and were harvested in December 2017; and the third rotation, during which the plants grew in the years 2018–2021, and were harvested in December 2021. It should also be added that under the climatic conditions of Poland, December is a winter month; therefore, during each harvest, the plants were dormant and devoid of leaves, and in practice, only SRWC shoots, i.e., woody biomass, were harvested.

Therefore, the current study analyzed woody chips obtained from cutting down entire SRWC shoots using a Junkkari HJ 10 G woodchipper working with a New Holland tractor, while harvesting the plants in three consecutive rotations. In each harvest rotation, approx. 5 kg of chips were collected from each experimental plot, packed in plastic sacs, and transported to the Energy Feedstock Evaluation Laboratory, Department of Genetics, Plant Breeding and Bioresource Engineering, UWM, to determine their properties.

2.2. Laboratory Analyses

Laboratory samples of the SRWC chips were first separated, and their moisture content was determined at 105 °C by the gravimetric method with oven drying (PN-EN ISO 18134-1:2015). The chips were dried in an FD BINDER laboratory dryer. In the consecutive rotations, SRWC harvesting, the transport of hermetically packed chips, and the laboratory work were planned so that the moisture content could be determined on the second day following the plant harvest, which eliminated the potential of moisture loss. Another step of the laboratory work was grinding dry chips in an analytical mill (Retsch SM 200) using a 1 mm sieve to obtain a uniform biomass fraction for further analyses. The biomass samples prepared in this manner were stored in laboratory containers and were gradually used for further analyses.

The higher heating value (HHV) of the biomass was determined using an IKA C2000 bomb calorimeter and the dynamic method. It should be added that prior to the HHV determination, the biomass samples were again placed in a laboratory dryer at 105 °C, and analytical samples were collected from them to be placed in the calorimetric bomb. In order to determine the ash content at 550 °C, as well as the volatile matter (VM) and fixed carbon (FC) contents at 650 °C, an Eltra TGA-Thermostep thermogravimetric analyzer was used (PN-EN ISO 18122:2016-01 and PN-EN ISO 18123:2016-01). Using an Eltra CHS 500 automatic analyzer, the carbon (C), hydrogen (H), and sulfur (S) contents were also determined according to PN-EN ISO 16948:2015-07 and PN-EN ISO 16994:2016-10. Total nitrogen (N) content was determined by the Kjeldahl method using a K-435 mineralizer and a BUCHI B-324 distiller, and the total chlorine (Cl) content was determined according to standard PN-ISO 587:2000 using Eschka's mixture. All laboratory analyses were conducted in three replications.

2.3. Statistical Analysis

Before performing the statistical analyses, the normality of the characteristics under study was checked using the Shapiro–Wilk test. The statistical analyses for all ten biomass quality characteristics, i.e., the moisture content, ash content, volatile matter content, fixed carbon content, HHV, and carbon, hydrogen, nitrogen, sulfur, and chlorine contents were carried out based on the repeated measures ANOVA. In this analysis, the SRWC species (black locust, poplar, and willow) and the soil enrichment procedure (C, L, F, M, LF, MF, LM, LMF) were the fixed and grouping factors, while the three consecutive harvest rotations (2013, 2017, 2021) were the repeated measurement factors. Additionally, the percentage of all the effects under study in the total sum square (total SS) of a particular variance analysis was calculated. In this way, a measure of the proportion in the variation under study (understood as a percentage of the variance explained by each individual effect of the analysis model) was obtained. The arithmetical averages and the variation coefficients were calculated for the characteristics under study. Using the Tukey's significance (HSD) test, homogeneous groups were determined at a significance level of $p < 0.05$. Additionally, descriptive statistics were determined for the entire dataset and separately for each SRWC species under study, i.e., the average value, median, minimum value, maximum value, lower quartile, upper quartile, standard deviation, and variation coefficient. Moreover, agglomerative hierarchical clustering analysis was conducted for the SRWC biomass properties under study. Before performing the analyses, the input data were standardized in columns. Ward's method was selected as the agglomeration method, with the Euclidean distance as the distance measure. The clusters were separated using Sneath's criterion. Two cut-off lines were used on the dendrogram: the first was placed on 2/3 Dmax and the second on 1/3 Dmax, where Dmax denoted the maximum measure of distance D. All statistical analyses were conducted using STATISTICA 13 software (TIBCO Software Inc., Palo Alto, CA, USA, 2017).

3. Results and Discussion

3.1. Thermophysical Properties of SRWC Biomass

The thermophysical characteristics under study, including the moisture content, ash content, volatile matter content, fixed carbon content, and the HHV of the woody biomass were significantly differentiated by all the main factors, i.e., the SRWC species, the soil enrichment procedure, the harvest rotation, and the interactions between these factors (Table 1). The SRWC species accounted for the highest biomass moisture content variation (93.8% of the overall variance). The black locust biomass was characterized by the significantly lowest moisture content (an average of 38.89%, homogeneous group "c") (Table 2). The moisture contents in the willow and poplar biomasses were also significantly higher (by 27.9 and 45.3%, respectively). As regards the soil enrichment procedures, a statistical analysis showed significant differences, but all the variants under analysis fell into homogeneous groups from "a" through "ab" to "b", and the moisture content fell within a very narrow range of 48.11 to 48.70%. The negligibly greater differences in the biomass moisture content occurred for the first two consecutive harvest rotations (5.1% and 3.2%), whereas a significantly lower moisture content in the SRWCs was noted in the third rotation (an average of 47.08%). However, it must be added that 3–5% moisture content variations are well within the seasonal variation of a species. In general, the black locust biomass was characterized by lower moisture content in all the harvest rotations (homogeneous group "f–g") as compared to the willow biomass (homogeneous group "e–d") and the poplar biomass (homogeneous group "a–c"). A low moisture content in the black locust biomass (an average of 40%) was also noted under Italian climatic conditions [26]. Other studies have also demonstrated that the moisture content of black locust biomass during the harvest was usually lower (approx. 40%) compared to willow (approx. 50%) and poplar (approx. 60%) [27]. The moisture content of poplar biomass during the harvest is usually high and accounts, in general, for over 50%, and it can occasionally even reach over 60% [11,28–31]. However, willow biomass was generally characterized by a moisture content lower than that of poplar, with an average value of approx. 50% [30,32]. An even lower water content of willow biomass (an average of 48.9%) was determined in a study on 15 genotypes cultivated at two locations and harvested in two consecutive three-year harvest rotations [22]. The cited study also found that the genotype was by far the largest contributor to the variation of this characteristic (almost 81% of the overall variance), similar to the current study's results. It should be noted that the weather conditions during the plant harvest, and immediately before and after the harvest, also affect the biomass moisture content. Precipitation and high humidity during the harvest translate into increased moisture content of the obtained biomass. Therefore, the SRWC biomass moisture content in the literature can often vary significantly, even within the same species and cultivation technology. Nevertheless, of the three SRWC species analyzed in the current study, black locust always exhibited the lowest water content, and poplar always exhibited the highest.

Table 1. Statistics of *p* values from the analysis of variance of the repeated measurements and the percentage of the effects in the overall variance for the thermophysical characteristics of SRWC biomass.

Source of Variation	df	Moisture		Ash		Volatile Matter		Fixed Carbon		HHV	
		p Value	Share (%)	*p* Value	Share (%)	*p* Value	Share (%)	*p* Value	Share (%)	*p* Value	Share (%)
Species (A)	2	<0.001 *	93.8	<0.001 *	50.9	<0.001 *	5.2	0.010 *	2.1	<0.001 *	62.6
Soil enrichment procedure (B)	7	0.008 *	0.1	<0.001 *	2.3	<0.001 *	10.5	<0.001 *	10.6	0.017 *	1.0
A × B	14	0.002 *	0.1	0.009 *	2.4	0.034 *	5.5	0.047 *	5.5	0.007 *	1.9
Error 1	48		0.2		3.3		9.2		9.7		2.5
Harvest rotation (R)	2	<0.001 *	1.8	<0.001 *	10.2	<0.001 *	7.8	<0.001 *	13.4	<0.001 *	6.7
R × A	4	<0.001 *	3.3	<0.001 *	9.6	<0.001 *	15.5	<0.001 *	12.8	<0.001 *	16.6
R × B	14	0.084	0.1	<0.001 *	8.8	<0.001 *	16.3	<0.001 *	13.9	0.003 *	1.7
R × A × B	28	<0.001 *	0.3	<0.001 *	6.7	0.004 *	11.4	0.002 *	12.6	0.006 *	2.6
Error 2	96		0.4		5.8		18.6		19.6		4.4
Total	2		100.0		100.0		100.0		100.0		100.0

* Significant values ($p < 0.05$); Share (%) percentage share in the total sum of squares.

The ash contents, just like the moisture contents, were most strongly determined by the SRWC species (almost 51%), followed by the harvest rotation (10%) and, to a lesser extent, by the other factors and the interactions between them (Table 1). Of all the SRWC species under study, the lowest ash content, an average of 1.25% DM, was determined in willow biomass (Table 2). The ash contents in the black locust and the poplar biomasses were significantly higher (by 12.0% and 33.6%, respectively). All of the applied soil enrichment procedures contributed to an increase in the ash content in SRWC biomass compared to the control plots (homogeneous group "c"), although these differences were not always statistically significant. However, for the interaction of species (A) and soil enrichment procedure (B) more varied results were obtained. Slightly greater differences in the ash content occurred for the consecutive harvest rotations, as the value of this characteristic was significantly noted in the third rotation (an average of 1.33 DM), while in the second and the first rotation this value was higher by 12.0% and 13.5%, respectively. In general, poplar biomass was characterized by a higher ash content in all harvest rotations (homogeneous group "a–c") as compared to the willow biomass, for which the lowest value of this characteristic was noted in the third harvest rotation (1.14 DM, homogeneous group "f"). A low ash content (an average of 1.26% DM) was also noted in the biomass of 15 different willow genotypes cultivated on different sites in three-year rotations [22]. It was emphasized, however, that the differences in the ash content between the genotypes under study were great (up to 44%). A study conducted in the USA noted even greater variation in the ash content in willow biomass (1–3% DM) [23]. It should be added, however, that the study involved several locations and several dozen genotypes. Similar ash contents in willow biomass (1.9–3% DM) were determined in other studies [31,33]. For poplar, depending on the genotype, the ash content also exhibited a similar high variation (0.98–3.12% DM) [29–31]. In another study, the ash content in four-year poplar shoots averaged 1.4% DM, ranging from 1.0 to 1.6% DM [11]. For black locust, the ash content ranged from 0.17–2.2% DM [34] and even exceeded 3.3% DM [31]. Despite these large fluctuations in the SRWC ash content, it should be added that these values were still lower than the ash content of semi-woody biomass, straw, or palm kernel shells [27,35,36]. However, compared to a solid fossil fuel such as hard coal, the ash content of SRWC biomass can be several times lower [37]. This is clearly favorable, as it can translate into higher proportions of fuel energy use, and the ash remaining from biomass can be used to enrich the soil [38].

Table 2. Selected thermophysical properties of the short-rotation woody crop (SRWC) biomass depending on the species and the soil enrichment procedure in three consecutive four-year harvest rotations.

Source of Variation	Item	Moisture Content (%)	Ash Content (% DM)	Volatile Matter (% DM)	Fixed Carbon (% DM)	Higher Heating Value (GJ Mg^{-1} DM)
Species (A)	Black locust	38.89 ^{c} (6.5)	1.40 ^{b} (13.2)	77.86 ^{b} (1.4)	20.74 ^{a} (5.1)	19.46 ^{c} (0.8)
	Poplar	56.52 ^{a} (3.2)	1.67 ^{a} (9.7)	77.83 ^{b} (1.1)	20.50 ^{ab} (4.0)	19.84 ^{a} (0.5)
	Willow	49.75 ^{b} (2.0)	1.25 ^{c} (14.1)	78.38 ^{a} (1.6)	20.37 ^{b} (6.3)	19.63 ^{b} (0.5)
Soil enrichment procedure (B)	C	48.11 ^{b} (16.4)	1.39 ^{c} (14.1)	77.68 ^{bc} (1.7)	20.93 ^{ab} (6.5)	19.66 ^{ab} (1.1)
	L	48.51 ^{ab} (14.6)	1.47 ^{abc} (21.9)	78.34 ^{a} (1.1)	20.20 ^{c} (3.5)	19.62 ^{ab} (1.1)
	F	48.14 ^{b} (16.1)	1.50 ^{a} (14.9)	77.28 ^{c} (2.0)	21.22 ^{a} (6.9)	19.67 ^{a} (0.9)
	LF	48.46 ^{ab} (15.9)	1.40 ^{bc} (20.0)	78.45 ^{a} (0.7)	20.16 ^{c} (2.4)	19.65 ^{ab} (1.0)
	M	48.40 ^{ab} (16.5)	1.47 ^{ab} (15.8)	77.98 ^{ab} (1.3)	20.55 ^{bc} (4.6)	19.61 ^{b} (1.0)
	MF	48.55 ^{ab} (15.8)	1.45 ^{abc} (14.2)	77.99 ^{ab} (1.2)	20.56 ^{bc} (4.3)	19.64 ^{ab} (1.0)
	LM	48.18 ^{ab} (15.4)	1.44 ^{abc} (18.6)	78.18 ^{ab} (1.6)	20.38 ^{bc} (5.7)	19.63 ^{ab} (1.1)
	LMF	48.70 ^{a} (15.5)	1.41 ^{bc} (17.5)	78.31 ^{a} (1.1)	20.28 ^{c} (4.3)	19.66 ^{ab} (0.9)
Harvest rotation (R)	First (R1)	49.49 ^{a} (11.5)	1.51 ^{a} (18.4)	78.33 ^{a} (1.2)	20.16 ^{b} (3.6)	19.60 ^{b} (1.3)
	Second (R2)	48.58 ^{b} (18.6)	1.49 ^{a} (11.8)	78.15 ^{a} (0.5)	20.37 ^{b} (1.4)	19.62 ^{b} (0.9)
	Third (R3)	47.08 ^{c} (15.6)	1.33 ^{b} (18.4)	77.59 ^{b} (2.0)	21.08 ^{a} (7.4)	19.71 ^{a} (0.6)
A × B	Black locust C	38.17 ^{d} (8.1)	1.37 ^{bcd} (13.4)	77.58 ^{bcd} (1.7)	21.06 ^{abcd} (5.5)	19.43 ^{e} (1.0)
	Black locust L	39.63 ^{c} (4.7)	1.49 ^{bc} (19.5)	78.24 (0.9)	20.27 ^{bcd} (3.3)	19.41 ^{e} (1.0)
	Black locust F	38.59 ^{cd} (7.4)	1.47 ^{bc} (11.9)	77.42 ^{cd} (2.3)	21.11 ^{abc} (7.9)	19.52 ^{de} (0.5)
	Black locust LF	38.83 ^{cd} (6.3)	1.29 ^{bcd} (16.6)	78.60 ^{abc} (0.4)	20.11 ^{bcd} (1.8)	19.47 ^{de} (0.9)
	Black locust M	38.45 ^{cd} (7.9)	1.37 ^{bcd} (5.6)	77.63 ^{bcd} (0.8)	21.00 ^{abcd} (2.8)	19.42 ^{e} (0.8)
	Black locust MF	39.10 ^{cd} (6.6)	1.43 ^{bc} (6.7)	77.59 ^{bcd} (1.4)	20.99 ^{abcd} (4.9)	19.45 ^{e} (0.6)
	Black locust LM	38.87 ^{cd} (6.2)	1.40 ^{bc} (11.5)	77.63 ^{bcd} (1.9)	20.97 ^{abcd} (7.7)	19.48 ^{de} (1.0)
	Black locust LMF	39.44 ^{cd} (6.3)	1.36 ^{bcd} (13.5)	78.23 (0.4)	20.42 ^{abcd} (0.9)	19.51 ^{de} (0.8)
	Poplar C	56.13 ^{a} (3.5)	1.55 ^{ab} (5.9)	77.98 ^{abcd} (1.0)	20.47 ^{abcd} (3.4)	19.87 ^{a} (0.4)
	Poplar L	56.26 ^{a} (1.8)	1.70 ^{a} (14.8)	77.90 ^{abcd} (0.9)	20.41 ^{abcd} (2.4)	19.85 ^{a} (0.4)
	Poplar F	56.59 ^{a} (3.1)	1.73 ^{a} (7.4)	76.70 ^{d} (1.9)	21.57 ^{a} (7.2)	19.86 ^{a} (0.5)
	Poplar LF	56.60 ^{a} (4.2)	1.70 ^{a} (11.2)	78.11 ^{abc} (0.7)	20.19 ^{bcd} (2.0)	19.83 ^{a} (0.4)
	Poplar M	56.68 ^{a} (4.1)	1.71 ^{a} (4.8)	77.75 ^{abcd} (0.8)	20.54 ^{abcd} (3.2)	19.79 ^{ab} (0.5)
	Poplar MF	56.72 ^{a} (3.8)	1.66 ^{a} (7.3)	78.11 ^{abc} (0.5)	20.23 ^{bcd} (1.5)	19.85 ^{a} (0.3)
	Poplar LM	56.20 ^{a} (1.5)	1.70 ^{a} (10.9)	77.91 ^{abcd} (0.9)	20.39 ^{abcd} (2.9)	19.84 ^{a} (0.6)
	Poplar LMF	56.96 ^{a} (3.6)	1.66 ^{a} (10.5)	78.14 ^{abc} (0.7)	20.20 ^{bcd} (1.9)	19.80 ^{a} (0.5)
	Willow C	50.02 ^{b} (1.5)	1.25 ^{cd} (14.1)	77.48 ^{cd} (2.4)	21.27 ^{ab} (9.2)	19.66 ^{c} (0.3)
	Willow L	49.65 ^{b} (2.3)	1.21 ^{cd} (19.2)	78.87 ^{ab} (1.2)	19.92 ^{cd} (4.5)	19.59 ^{cd} (0.4)
	Willow F	49.26 ^{b} (2.0)	1.30 ^{bcd} (8.8)	77.72 ^{abcd} (1.7)	20.98 ^{abcd} (6.1)	19.62 ^{cd} (0.5)
	Willow LF	49.97 ^{b} (1.8)	1.19 ^{d} (7.8)	78.63 ^{abc} (0.8)	20.18 ^{bcd} (3.2)	19.66 ^{c} (0.6)
	Willow M	50.08 ^{b} (0.9)	1.34 ^{bcd} (19.8)	78.55 ^{abc} (1.7)	20.11 ^{bcd} (6.4)	19.63 ^{cd} (0.5)
	Willow MF	49.83 ^{b} (2.2)	1.26 ^{cd} (11.9)	78.28 ^{abc} (1.4)	20.47 ^{abcd} (5.1)	19.62 ^{cd} (0.5)
	Willow LM	49.46 ^{b} (2.4)	1.22 ^{cd} (16.5)	78.99 ^{a} (1.2)	19.80 ^{d} (4.1)	19.58 ^{cd} (0.6)
	Willow LMF	49.69 ^{b} (2.2)	1.21 ^{cd} (10.0)	78.57 ^{abc} (1.8)	20.22 ^{bcd} (7.3)	19.67 ^{bc} (0.6)
A × R	Black locust R1	42.13 ^{f} (1.1)	1.51 ^{c} (4.8)	78.05 ^{bcd} (0.7)	20.44 ^{c} (2.8)	19.33 ^{f} (0.4)
	Black locust R2	37.02 ^{g} (3.4)	1.39 ^{d} (12.7)	78.27 ^{b} (0.5)	20.34 ^{c} (1.4)	19.40 ^{e} (0.4)
	Black locust R3	37.51 ^{g} (3.2)	1.29 ^{e} (16.3)	77.26 ^{d} (2.1)	21.44 ^{a} (7.1)	19.65 ^{c} (0.6)
	Poplar R1	55.81 ^{b} (0.6)	1.83 ^{a} (6.0)	77.49 ^{cd} (0.4)	20.69 ^{bc} (1.6)	19.92 ^{a} (0.1)
	Poplar R2	58.75 ^{a} (1.8)	1.64 ^{b} (5.2)	78.03 ^{bcd} (0.5)	20.32 ^{c} (1.6)	19.79 ^{b} (0.3)
	Poplar R3	54.98 ^{c} (1.6)	1.55 ^{c} (9.4)	77.96 ^{bcd} (1.8)	20.49 ^{c} (6.5)	19.79 ^{b} (0.5)
	Willow R1	50.53 ^{d} (0.6)	1.18 ^{f} (3.3)	79.46 ^{a} (0.5)	19.36 ^{d} (2.0)	19.54 ^{d} (0.2)
	Willow R2	49.95 ^{d} (0.9)	1.42 ^{d} (9.2)	78.14 ^{bc} (0.4)	20.44 ^{c} (1.3)	19.66 ^{c} (0.4)
	Willow R3	48.75 ^{e} (2.0)	1.14 ^{f} (15)	77.56 ^{bcd} (2.2)	21.30 ^{ab} (7.9)	19.70 ^{c} (0.4)

Soil enrichment procedure: control plot with no soil enrichment (C), lignin (L), mineral fertilization (F), lignin + mineral fertilization (LF), mycorrhiza inoculation (M), mycorrhiza + mineral fertilization (MF), lignin + mycorrhiza (LM); lignin + mycorrhiza + mineral fertilization (LMF); a, b, c, etc.—homogenous groups for the main source of variation and their interaction separately for each attribute, no letter denotes an absence of significance; (the coefficients of variation—%).

The ash contents, just like the moisture contents, were most strongly determined by the SRWC species (almost 51%), followed by the harvest rotation (10%) and, to a lesser extent, by the other factors and the interactions between them (Table 1). Of all the SRWC species under study, the lowest ash content, an average of 1.25% DM, was determined in willow biomass (Table 2). The ash contents in the black locust and the poplar biomasses were significantly higher (by 12.0% and 33.6%, respectively). All of the applied soil enrichment procedures contributed to an increase in the ash content in SRWC biomass compared to the control plots (homogeneous group "c"), although these differences were not always statistically significant. However, for the interaction of species (A) and soil enrichment procedure (B) more varied results were obtained. Slightly greater differences in the ash content occurred for the consecutive harvest rotations, as the value of this characteristic was significantly noted in the third rotation (an average of 1.33 DM), while in the second and the first rotation this value was higher by 12.0% and 13.5%, respectively. In general, poplar biomass was characterized by a higher ash content in all harvest rotations (homogeneous group "a–c") as compared to the willow biomass, for which the lowest value of this characteristic was noted in the third harvest rotation (1.14 DM, homogeneous group "f"). A low ash content (an average of 1.26% DM) was also noted in the biomass of 15 different willow genotypes cultivated on different sites in three-year rotations [22]. It was emphasized, however, that the differences in the ash content between the genotypes under study were great (up to 44%). A study conducted in the USA noted even greater variation in the ash content in willow biomass (1–3% DM) [23]. It should be added, however, that the study involved several locations and several dozen genotypes. Similar ash contents in willow biomass (1.9–3% DM) were determined in other studies [31,33]. For poplar, depending on the genotype, the ash content also exhibited a similar high variation (0.98–3.12% DM) [29–31]. In another study, the ash content in four-year poplar shoots averaged 1.4% DM, ranging from 1.0 to 1.6% DM [11]. For black locust, the ash content ranged from 0.17–2.2% DM [34] and even exceeded 3.3% DM [31]. Despite these large fluctuations in the SRWC ash content, it should be added that these values were still lower than the ash content of semi-woody biomass, straw, or palm kernel shells [27,35,36]. However, compared to a solid fossil fuel such as hard coal, the ash content of SRWC biomass can be several times lower [37]. This is clearly favorable, as it can translate into higher proportions of fuel energy use, and the ash remaining from biomass can be used to enrich the soil [38].

When analyzing the volatile matter and fixed carbon contents, it was difficult to clearly indicate which factors most strongly determined these parameters. The main factors ranged between only 2.1% and 13.4%, while the interactions between them ranged from 5.5% to 16.3% (Table 1). Therefore, it is difficult to identify the factor with the strongest effect. Willow biomass was characterized by the highest volatile matter content (an average of 78.38% DM) and the lowest fixed carbon content (an average of 20.37% DM) (Table 2). On the other hand, the volatile matter contents in black locust and poplar biomass were only lower by 0.7%, and the fixed carbon content was higher by less than 1–2%. Moreover, the variation in these two parameters in terms of the soil enrichment procedures and the consecutive harvest rotations was small and ranged from 1% to 5%. The highest volatile matter content (an average of 79.46% DM) was noted in willow biomass in the first harvest rotation, while the highest fixed carbon content (an average of 21.44% DM) was noted in black locust biomass in the third harvest rotation. In another study, the fixed carbon and volatile matter contents in four-year poplar shoots were, on average, 18.6 and 79.4% DM, respectively [11], while in three-year willow shoots these values amounted to 19.4 and 79.4% DM, respectively [22]. Moreover, the cited study did not identify the factor with the clearly strongest effect on the fixed carbon and volatile matter contents, as they were determined by the harvest rotation, genotype, location, and the interactions between these factors. On the other hand, in a study conducted in Spain [31], the volatile matter contents in black locust, poplar, and willow biomass were generally higher than in the current study and amounted to 81.33, 82.37, and 83.59% DM, respectively. Nevertheless, of all the species in that study, similar to the current study, the highest value of this characteristic was noted for willow.

Higher heating value (HHV) was most strongly determined by the SRWC species (62.6%), followed by the interaction between the species and the harvest rotation (16.6%) and, to a lesser extent, by all the other factors and their interactions (Table 1). The poplar biomass was characterized by the significantly highest HHV with an average of 19.84 GJ Mg^{-1} DM (Table 2). The value of this characteristic for willow and black locust biomass was slightly (but significantly) lower (by 1.1% and 1.9%, respectively). As regards the soil enrichment procedures, the differences in the HHV between the plots under study were less than 0.5%. The situation was similar for consecutive harvest rotations, where a significantly higher HHV value was noted in the third rotation (an average of 19.71 GJ Mg^{-1} DM), while in the second and the first rotation this value was lower by 0.5% and 0.6%, respectively. The poplar biomass was characterized by higher HHV values in all harvest rotations (homogeneous group "a–b") as compared to willow (homogeneous group "c–d") and black locust (homogeneous group "c–f"). In another study, four-year poplar shoots were also characterized by a high HHV value (averaging 19.60 GJ Mg^{-1} DM), while in the second harvest rotation, for the UWM2 clone, it was as high as 19.9 GJ Mg^{-1} DM [11]. A lower HHV (an average of 19.53 GJ Mg^{-1} DM) was noted in a study involving 15 willow genotypes sourced from two locations in the consecutive three-year harvest rotations [22], which was consistent with the relationships demonstrated in the current study.

3.2. Elemental Composition of SRWC Biomass

The carbon (C), hydrogen (H), nitrogen (N), sulfur (S), and chlorine (Cl) contents in biomass were significantly differentiated by all the main factors, i.e., the SRWC species, soil enrichment procedure, and harvest rotation, as well as by most interactions between these factors (Table 3). The harvest rotation accounted for the greatest percentage of the C, H, and Cl contents in the variation (83.9%, 73.3%, and 28.3%, respectively, of the overall variation), which was somewhat surprising. However, regarding the N content, the SRWC species accounted for the decidedly highest percentage (81.3%) in the variation of this characteristic. The SRWC species also contributed to the variation in the S content (20.5%), but the interaction between the species, harvest rotation, and soil enrichment procedure contributed even more (22.4%), followed by the interaction between the species and the soil enrichment procedure (10.1%).

Table 3. Statistics of *p* values from the repeated measurement variance analysis and the percentage of the effects in the overall variance for the SRWC biomass elemental composition.

Source of Variation	df	C		H		N		S		Cl	
		p Value	Share (%)	*p* Value	Share (%)	*p* Value	Share (%)	*p* Value	Share (%)	*p* Value	Share (%)
Species (A)	2	<0.001 *	3.1	<0.001 *	0.8	<0.001 *	81.3	<0.001 *	20.5	<0.001 *	20.7
Soil enrichment procedure (B)	7	0.005 *	1.0	<0.001 *	2.3	<0.001 *	1.0	<0.001 *	5.4	<0.001 *	3.5
A × B	14	0.016 *	1.4	<0.001 *	3.6	<0.001 *	1.2	<0.001 *	10.1	<0.001 *	5.9
Error 1	48		2.0		1.2		0.9		7.9		2.0
Harvest rotation (R)	2	<0.001 *	83.9	<0.001 *	73.3	<0.001 *	2.1	<0.001 *	3.5	<0.001 *	28.3
R × A	4	0.111	0.3	<0.001 *	1.4	<0.001 *	3.9	<0.001 *	3.3	<0.001 *	22.5
R × B	14	0.030 *	1.2	<0.001 *	5.9	<0.001 *	3.9	<0.001 *	12.9	<0.001 *	4.0
R × A × B	28	0.003 *	2.7	<0.001 *	9.3	<0.001 *	4.2	<0.001 *	22.4	<0.001 *	9.1
Error 2	96		4.3		2.2		1.5		14.0		4.0
Total	2		100.0		100.0		100.0		100.0		100.0

* Significant values ($p < 0.05$); Share (%) percentage share in the total sum of squares.

Poplar and willow biomasses were characterized by a significantly higher C content (on average over 53.3% DM, homogeneous group "a") as compared to the black locust biomass (Table 4). For the soil enrichment procedures, the statistical analysis showed differences, but all the variants under analysis fell into homogeneous groups from "a" through "ab" to "b", and the C content fell within a range of 52.69–53.41% DM. Greater differences in the C content in SRWC biomass occurred for the consecutive harvest rotations, as a significantly higher value of this characteristic was noted in the second and the third rotation (over 54.4% DM), while in the first rotation this value was lower by approx. 7.5%. This could have been affected by varied interactions and environmental conditions in consecutive years of plant cultivation. In general, the black locust biomass was characterized by a lower C content in all the harvest rotations compared to the willow and the poplar biomasses. However, in another study [31], black locust biomass (51.51% DM) and poplar biomass (51.81% DM) were characterized by a higher C content compared to willow biomass (48.84% DM). A higher carbon content in willow biomass (amounting to an average of 52.90% DM) was determined for different genotypes, locations, and harvest rotations [22]. Moreover, the cited study found that the location was the greatest contributor to the variation in the C content, followed by the genotype, harvest rotation, and the interaction between these factors, which was also partially confirmed by the current study.

Willow biomass was characterized by a significantly higher H content of an average of 5.97% DM (Table 4). The value of this characteristic for the black locust and poplar biomass was slightly but significantly lower (by 0.5 and 1.2%, respectively). Of all the soil enrichment procedures applied, a significantly higher H content (an average of 6.02% DM) was noted for the control plot (C), while for the other soil enrichment variants the value of this characteristic was lower within a range of 1–3%. Greater differences in the H content in SRWC biomass occurred for the consecutive harvest rotations, as a significantly higher value of this characteristic was noted in the third rotation (over 6.31% DM), while in the first and the second rotation this value was lower (by 7.1% and 10.6%, respectively). In another study [31], black locust biomass (6.44% DM) and poplar biomass (6.39% DM) were characterized by a higher H content compared to willow biomass (6.18% DM). Moreover, the H content in SRWC biomass in the cited study was generally higher than the current study's results. A higher H content in willow biomass (6.23% DM) was also determined in another study [22]. Moreover, it was found that the content of this element was most strongly determined by the interaction between the location and the genotype, followed by the genotype and the location.

Black locust biomass was characterized by a significantly higher S content with an average of 0.033% DM (Table 4). The value of this characteristic for willow and poplar biomasses was significantly lower (by 21.2% and 24.2%, respectively). Of all the soil enrichment procedures applied, a significantly higher S content (an average of 0.032% DM) was noted for the plot on which mineral fertilization was applied (F), while for the other soil enrichment variants the value of this characteristic was lower within the range of 9–22%. However, for the interaction of species (A) and soil enrichment procedure (B) more varied results were obtained. In the second harvest rotation, the average sulfur content in SRWC biomass was 10% higher than in the first and third harvest rotations. It should be noted that black locust biomass was generally characterized by a higher S content in all harvest rotations (homogeneous group "a–ab") compared to poplar and willow. In a study conducted in Spain [31], black locust was characterized by a higher S content (0.05% DM) compared to poplar (0.04% DM) and willow (0.03% DM). In another study, the S content in willow biomass was mainly determined by the genotype (25%), location (18%), and the interaction between these two factors (21%) [22], which was similar to the results of the current study. However, the average content of this element in the cited study amounted to an average of 0.032% and was, therefore, 19% higher than the average value for the willow biomass in the current study. However, the value of this characteristic for the 15 willow genotypes under study (all harvested in two three-year rotations) varied and averaged between 0.026–0.037% DM.

Table 4. The elemental composition of short-rotation woody crop (SRWC) biomass depending on the species and the soil enrichment procedure in three consecutive four-year harvest rotations.

Source of Variation	Item	C (% DM)	H (% DM)	S (% DM)	N (% DM)	Cl (% DM)
Species (A)	Black locust	52.60 ^{b} (4.0)	5.94 ^{b} (6.1)	0.033 ^{a} (23.6)	0.91 ^{a} (19.8)	0.032 ^{a} (48.7)
	Poplar	53.46 ^{a} (3.7)	5.90 ^{c} (5.2)	0.025 ^{b} (26.5)	0.43 ^{b} (16.8)	0.027 ^{b} (22.7)
	Willow	53.25 ^{a} (3.9)	5.97 ^{a} (5.1)	0.026 ^{b} (26.1)	0.38 ^{c} (12.7)	0.019 ^{c} (42.6)
Soil enrichment procedure (B)	C	53.33 ^{a} (3.8)	6.02 ^{a} (4.0)	0.029 ^{abc} (28.3)	0.56 ^{bc} (41.7)	0.023 ^{c} (38.3)
	L	53.16 ^{ab} (4.0)	5.96 ^{ab} (5.3)	0.028 ^{abc} (31.2)	0.61 ^{a} (51.9)	0.025 ^{bc} (42.6)
	F	52.69 ^{b} (3.8)	5.96 ^{ab} (3.8)	0.032 ^{a} (31.5)	0.60 ^{ab} (44.8)	0.024 ^{c} (50.1)
	LF	52.93 ^{ab} (4.0)	5.93 ^{b} (5.7)	0.027 ^{bc} (18.9)	0.54 ^{c} (41.7)	0.029 ^{a} (43.2)
	M	53.17 ^{ab} (3.9)	5.92 ^{b} (6.2)	0.027 ^{bc} (19.9)	0.55 ^{bc} (49.2)	0.027 ^{ab} (49.1)
	MF	53.05 ^{ab} (3.9)	5.91 ^{b} (5.5)	0.027 ^{bc} (30.0)	0.59 ^{ab} (49.1)	0.025 ^{bc} (37.6)
	LM	53.41 ^{a} (4.3)	5.84 ^{c} (7.5)	0.025 ^{c} (24.5)	0.60 ^{ab} (44.1)	0.025 ^{bc} (44.7)
	LMF	53.08 ^{ab} (3.9)	5.96 ^{ab} (5.4)	0.029 ^{ab} (32.1)	0.55 ^{bc} (49.0)	0.029 ^{a} (56.2)
Harvest rotation (R)	First (R1)	50.42 ^{b} (1.2)	5.86 ^{b} (1.6)	0.027 ^{b} (29.5)	0.62 ^{a} (50.0)	0.025 ^{b} (20.1)
	Second (R2)	54.49 ^{a} (1.5)	5.64 ^{c} (4.6)	0.030 ^{a} (27.4)	0.58 ^{b} (44.8)	0.034 ^{a} (40.5)
	Third (R3)	54.40 ^{a} (1.9)	6.31 ^{a} (1.7)	0.027 ^{b} (26.8)	0.52 ^{c} (40.7)	0.019 ^{c} (53.7)
A × B	Black locust C	52.72 ^{abcd} (3.8)	6.05 ^{ab} (3.5)	0.036 ^{ab} (23.7)	0.85 ^{cd} (17.2)	0.023 ^{de} (45.1)
	Black locust L	52.38 ^{cd} (4.0)	5.85 ^{bcd} (7.2)	0.028 ^{bcd} (39.7)	0.98 ^{a} (28.9)	0.033 ^{bc} (23.4)
	Black locust F	52.08 ^{d} (4.2)	5.98 ^{abc} (3.6)	0.039 ^{a} (25.5)	0.94 ^{abc} (18.2)	0.030 ^{bcd} (53.8)
	Black locust LF	52.43 ^{cd} (4.3)	6.02 ^{abc} (4.6)	0.030 ^{abc} (15.7)	0.81 ^{d} (22.5)	0.036 ^{ab} (40.0)
	Black locust M	53.12 ^{abcd} (4.3)	5.88 ^{bc} (9.0)	0.031 ^{abc} (10.5)	0.90 ^{abc} (15.5)	0.032 ^{bc} (65.0)
	Black locust MF	52.76 ^{abcd} (4.4)	5.89 ^{bc} (7.3)	0.036 ^{ab} (21.0)	0.98 ^{a} (12.3)	0.032 ^{bc} (36.6)
	Black locust LM	52.75 ^{abcd} (4.4)	5.82 ^{bcd} (7.3)	0.031 ^{abc} (20.0)	0.95 ^{ab} (7.2)	0.031 ^{bcd} (50.9)
	Black locust LMF	52.58 ^{bcd} (4.1)	5.99 ^{abc} (5.8)	0.032 ^{bcd} (11.9)	0.87 ^{bcd} (26.6)	0.042 ^{a} (54.0)
	Poplar C	53.46 ^{abc} (3.7)	5.91 ^{bc} (4.4)	0.026 ^{bcd} (26.9)	0.41 ^{efg} (13.0)	0.026 ^{cd} (22.4)
	Poplar L	53.67 ^{ab} (3.3)	5.98 ^{abc} (3.3)	0.029 ^{bcd} (32.4)	0.48 ^{e} (21.7)	0.030 ^{bcd} (20.0)
	Poplar F	52.95 ^{abcd} (3.4)	5.90 ^{bc} (4.0)	0.030 ^{bcd} (32.4)	0.42 ^{efg} (10.7)	0.027 ^{cd} (22.5)
	Poplar LF	53.62 ^{ab} (4.0)	5.98 ^{abc} (3.7)	0.025 ^{bcd} (6.5)	0.44 ^{efg} (12.4)	0.030 ^{bcd} (30.9)
	Poplar M	53.12 ^{abcd} (4.2)	5.87 ^{bc} (5.2)	0.024 ^{bcd} (27.5)	0.39 ^{fg} (8.2)	0.028 ^{cd} (14.4)
	Poplar MF	53.32 ^{abc} (4.1)	5.95 ^{bc} (4.2)	0.021 ^{d} (8.9)	0.43 ^{efg} (7.5)	0.021 ^{de} (18.9)
	Poplar LM	53.68 ^{ab} (4.0)	5.73 ^{d} (9.6)	0.022 ^{cd} (17.5)	0.47 ^{ef} (24.7)	0.024 ^{cd} (10.7)
	Poplar LMF	53.83 ^{a} (4.1)	5.90 ^{bc} (6.0)	0.022 ^{cd} (12.9)	0.41 ^{efg} (15.9)	0.027 ^{cd} (20.4)
	Willow C	53.82 ^{a} (4.1)	6.10 ^{a} (3.8)	0.024 ^{bcd} (13.1)	0.41 ^{efg} (15.7)	0.019 ^{def} (47.1)
	Willow L	53.42 ^{abc} (4.6)	6.05 ^{ab} (4.7)	0.026 (19.3)	0.38 ^{fg} (11.9)	0.013 ^{def} (38.7)
	Willow F	53.06 ^{abcd} (3.9)	5.99 ^{abc} (4.2)	0.026 ^{bcd} (19.6)	0.42 ^{efg} (11.2)	0.016 ^{def} (50.9)
	Willow LF	52.75 ^{abcd} (3.9)	5.80 ^{cd} (8.1)	0.026 ^{bcd} (24.6)	0.37 ^{g} (6.7)	0.021 ^{de} (46.5)
	Willow M	53.28 ^{abc} (3.6)	5.99 ^{abc} (3.9)	0.025 ^{bcd} (8.3)	0.35 ^{g} (14)	0.022 ^{de} (35.1)
	Willow MF	53.08 ^{abcd} (3.6)	5.91 ^{bc} (5.3)	0.024 ^{bcd} (20.7)	0.37 ^{g} (14.1)	0.022 ^{de} (33.5)
	Willow LM	53.79 ^{a} (4.8)	5.95 ^{bc} (5.6)	0.022 ^{cd} (14.5)	0.38 ^{fg} (10.7)	0.020 ^{de} (41.6)
	Willow LMF	52.82 ^{abcd} (3.4)	5.99 ^{abc} (4.7)	0.033 ^{abc} (41.3)	0.36 ^{g} (5.1)	0.019 ^{def} (37.7)
A × R	Black locust R1	49.76 (0.5)	5.91 ^{c} (1.2)	0.034 ^{a} (27.8)	1.05 ^{a} (5.0)	0.030 ^{b} (16.4)
	Black locust R2	54.16 (1.4)	5.57 ^{f} (5.3)	0.033 ^{a} (15.9)	0.90 ^{b} (20.6)	0.049 ^{a} (24.7)
	Black locust R3	53.89 (1.3)	6.33 ^{a} (1.5)	0.031 ^{ab} (25.3)	0.78 ^{c} (20.9)	0.018 ^{e} (41.3)
	Poplar R1	50.96 (0.6)	5.78 ^{d} (1.4)	0.024 ^{d} (11.8)	0.44 ^{de} (5.0)	0.024 ^{d} (12.1)
	Poplar R2	54.75 (1.3)	5.67 ^{e} (4.6)	0.028 ^{bc} (28.5)	0.47 ^{d} (19.0)	0.028 ^{bc} (22.6)
	Poplar R3	54.65 (2.5)	6.25 ^{b} (2.0)	0.024 ^{d} (30.8)	0.39 ^{f} (17.7)	0.028 ^{bc} (25.1)
	Willow R1	50.53 (0.7)	5.90 ^{c} (1.1)	0.022 ^{d} (10.2)	0.37 ^{f} (11.9)	0.022 ^{d} (14.7)
	Willow R2	54.55 (1.7)	5.68 ^{e} (3.6)	0.029 ^{bc} (34.8)	0.38 ^{f} (11.4)	0.025 ^{cd} (22.2)
	Willow R3	54.67 (1.4)	6.34 ^{a} (1.1)	0.026 ^{cd} (13.2)	0.40 ^{ef} (13.7)	0.010 ^{f} (48.3)

Soil enrichment procedure: control plot with no soil enrichment (C), lignin (L), mineral fertilization (F), lignin + mineral fertilization (LF), mycorrhiza inoculation (M), mycorrhiza + mineral fertilization (MF), lignin + mycorrhiza (LM); lignin + mycorrhiza + mineral fertilization (LMF); a, b, c, etc.—homogenous groups for the main source of variation and their interaction separately for each attribute, no letter denotes an absence of significance; (the coefficients of variation—%).

Black locust biomass was also characterized by significantly higher N and Cl contents averaging 0.91% and 0.032% DM (Table 4). However, the lowest contents of these elements were noted for willow biomass, while medium contents were found for poplar biomass. The N content in poplar and willow biomass was lower by an average of 52.7% and 58.2%, respectively. However, these differences were smaller for the Cl content and amounted to 15.6% and 40.6%, respectively. The application of lignin for soil enrichment contributed to an increase in the N content in SRWC biomass (an average of 0.61% DM), while on the control plot this value was lower by 8%. It was also noted that the average nitrogen content in biomass was lower in the consecutive harvest rotations, since in the first harvest rotation it averaged 0.62% DM, while in the second and third rotations it was significantly lower (by 6.5% and 17.1%, respectively). It should be added that black locust biomass was generally characterized by a higher N content in all harvest rotations (homogeneous group "a–c") as compared to poplar (homogeneous group "d–f") and willow (homogeneous group "e–f"). Moreover, in a study conducted in a warmer climate [31], the black locust biomass was characterized by higher N and Cl contents of 0.63 and 0.02% DM, respectively, compared to willow and poplar. For willow biomass, the nitrogen content was lower by 27%, and for poplar, by as much as 75%. On the other hand, the lowest chlorine content in the cited study was noted for willow biomass. In another study [22], the average N content in willow biomass was 0.42% DM and was thus slightly higher than the value noted in the current study. However, it fell within a wider range of 0.36–0.51% DM. The average nitrogen content in poplar biomass (0.41–0.42% DM, i.e., a value similar to that obtained in the current study) was obtained in other studies [11,39].

3.3. Practical Implication of the Study

When SRWCs are used for energy purposes, the properties of this biomass type are very often generalized, and the biomass is regarded as a relatively homogeneous energy feedstock with similar parameters. Table 5 presents descriptive characteristics of the entire dataset for three SRWC species cultivated in eight soil enrichment variants and harvested in three consecutive four-year rotations, therefore for a total of 216 plots. Based on the presented data, the lowest variation expressed by the variation coefficient (below 6%) was noted for such characteristics as the HHV, volatile matter, fixed carbon, and the C and H contents. However, the differences between the minimum and the maximum value for these parameters ranged from 5% to 35%. Even lower were the differences (1–8%) between the values from the upper and lower quartile for these parameters. On the other hand, the biomass moisture and ash contents were characterized by greater variation expressed by the variation coefficient (a range of 15–17%). For example, the SRWC biomass moisture content during the winter harvest was 48.38%. However, the minimum value for this parameter was only 35.05%, while the maximum value was as much as 60.03%, i.e., the difference between the minimum and the maximum value was 71%. Even greater variation was noted for the ash content, where the difference between the minimum and the maximum value was 135%. The differences between the values from the upper and lower quartile for these two parameters were lower and amounted to approx. 33%. Despite this, the contents of sulfur (28%), nitrogen, and chlorine (46% each) were characterized by decidedly higher variation coefficient values. Obviously, for these three elements, the differences between the minimum and the maximum value were greater and amounted to 310% for S, 352% for N, and 1739% for Cl, respectively. On the other hand, the differences between the lower and the upper quartile were 40%, 113%, and 52%, respectively.

Table 5. Selected statistical analysis indicators for the SRWC characteristics under study.

Attribute	Mean	Median	Minimum Value	Maximum Value	Lower Quartile	Upper Quartile	Standard Deviation	Coefficient of Variation
Moisture (%)	48.38	50.04	35.05	60.03	41.94	55.44	7.52	15.54
Ash (% DM)	1.44	1.44	0.86	2.02	1.22	1.63	0.25	17.25
HHV (GJ Mg^{-1} DM)	19.64	19.68	19.18	20.05	19.50	19.77	0.19	0.99
Volatile matter (% DM)	78.02	78.15	72.92	80.16	77.66	78.58	1.12	1.44
Fixed carbon (% DM)	20.53	20.38	18.69	25.26	20.04	20.78	1.07	5.23
C (% DM)	53.10	53.84	49.21	56.86	50.90	54.71	2.08	3.92
H (% DM)	5.94	5.88	5.04	6.55	5.78	6.25	0.33	5.49
S (% DM)	0.03	0.03	0.01	0.06	0.02	0.03	0.01	28.26
N (% DM)	0.57	0.44	0.30	1.36	0.38	0.80	0.27	46.23
Cl (% DM)	0.03	0.02	0.00	0.07	0.02	0.03	0.01	46.45

Number of observations for all attributes n = 216.

Given this variation, similarities were sought between the plots studied over the 12-year period. Therefore, Figure 1 shows a dendrogram of a hierarchical cluster analysis illustrating the mutual similarities of the biomass of the species and soil enrichment procedures under study in terms of its parameters as a solid biofuel. This analysis showed that in terms of the biomass characteristics under analysis, when cutting off 2/3 Dmax, two clusters were formed (Figure 1a). One cluster contained the fixed carbon, S, N, and Cl contents, while the second cluster contained the other six parameters under analysis. However, when cutting off 1/3 Dmax, up to six clusters were formed. The fixed carbon content separated from S, N, and Cl, which resulted in the formation of two independent clusters. Consequently, as many as four parameters (fixed carbon, volatile matter, H, and ash contents) constituted separate clusters, while the sixth cluster comprised the moisture content, HHV, and C content. As regards the analysis of the similarities between the SRWC species and the soil enrichment procedures under study, it was found that cutting off 2/3 Dmax formed two clusters (Figure 1b). Black locust, irrespective of the soil enrichment procedure, formed its own cluster, while poplar and willow formed another common cluster. On the other hand, when cutting off 1/3 Dmax, four clusters were formed. Black locust continued to represent a single separate cluster. Moreover, three separate clusters were formed. One of them contained most soil enrichment variants in poplar cultivation, except poplar F. The second cluster contained poplar and willow in variants F and willow in variant C, while the third cluster included all the remaining soil enrichment variants in willow cultivation.

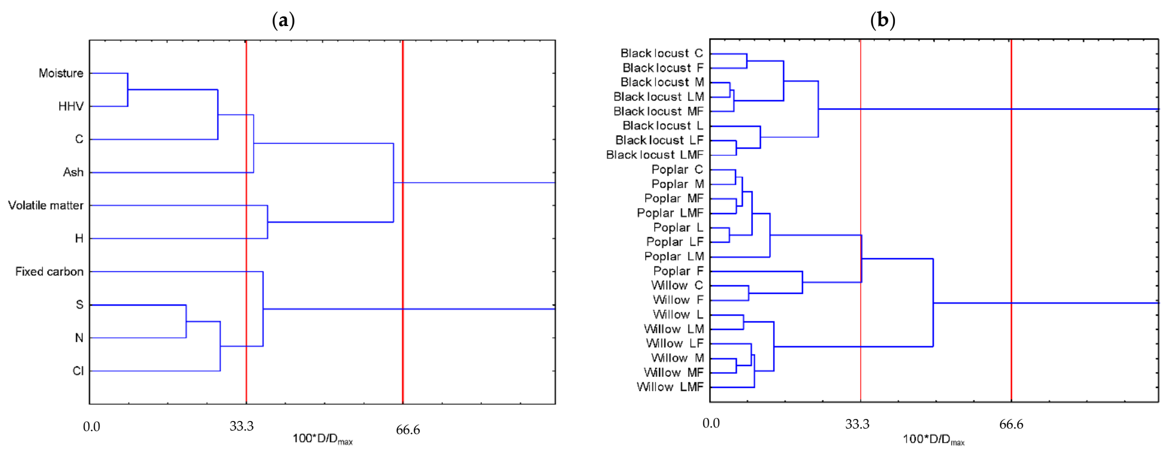

Figure 1. A dendrogram of a hierarchical cluster analysis illustrating the mutual similarities of the characteristics under study (**a**) and the SRWC species and the soil enrichment procedures (**b**). The red vertical line marks the Sneath criterion, (2/3 D_{max}) and (1/3 D_{max}). D—linkage distance; D_{max}—maximum linkage distance.

Black locust is a species that fixes free nitrogen from the air and tolerates unfavorable habitat conditions very well, including the possibility of cultivation on land after open-cast mine reclamation [40]. This feature is undoubtedly a great advantage of this plant, but it can increase the nitrogen content in biomass. However, the distinctiveness of black locust biomass from willow and poplar biomass is also clearly illustrated by selected descriptive statistics (Tables 6–8). As regards the biomass moisture content, the median for the black locust biomass was 38%, and the maximum value was just under 43%, which is favorable in terms of the energy use of this species. However, as regards poplar and willow biomass, the moisture content median was decidedly higher (56 and 50%, respectively) than the maximum value for black locust. Moreover, the black locust biomass N content median (0.93% DM) was higher than the maximum contents of this element in willow and poplar biomass (0.52 and 0.63% DM, respectively). The median and the maximum S and Cl contents in black locust biomass were also higher than the analogous values in willow and poplar biomass. The higher N, S, and Cl contents in black locust biomass are unfavorable in terms of the use of this species for energy purposes. However, it does not mean that biomass from black locust is some kind of problem for the energy industry; rather, it indicates that this biomass is not optimum. The C content and HHV of black locust biomass were lower than the analogous values for willow and poplar biomass, even though these differences were not as great as for the above-described parameters. On the other hand, as regards SRWCs, poplar was characterized by the highest maximum values of biomass moisture, ash, and C contents, as well as the HHV. Willow was characterized by the lowest maximum ash, S, N, and Cl content values.

Table 6. Selected statistical analysis indicators for the black locust characteristics under study.

Attribute	Median	Minimum Value	Maximum Value	Lower Quartile	Upper Quartile	Standard Deviation
Moisture (%)	38.21	35.05	42.97	36.63	41.94	2.53
Ash (% DM)	1.42	0.93	1.82	1.26	1.51	0.19
HHV (GJ Mg^{-1} DM)	19.41	19.18	19.79	19.35	19.61	0.16
Volatile matter (% DM)	78.11	72.92	79.35	77.69	78.43	1.10
Fixed carbon (% DM)	20.54	19.03	25.26	20.24	20.85	1.06
C (% DM)	53.48	49.21	55.63	49.90	54.35	2.12
H (% DM)	5.92	5.15	6.55	5.80	6.27	0.36
S (% DM)	0.032	0.017	0.055	0.028	0.036	0.008
N (% DM)	0.93	0.52	1.36	0.80	1.04	0.18
Cl (% DM)	0.030	0.004	0.074	0.022	0.042	0.016

Number of observations for all attributes $n = 72$.

Table 7. Selected statistical analysis indicators for the poplar characteristics under study.

Attribute	Median	Minimum Value	Maximum Value	Lower Quartile	Upper Quartile	Standard Deviation
Moisture (%)	55.96	53.66	60.03	55.44	57.83	1.82
Ash (% DM)	1.68	1.30	2.02	1.57	1.79	0.16
HHV (GJ Mg^{-1} DM)	19.84	19.67	20.05	19.76	19.91	0.09
Volatile matter (% DM)	77.92	74.39	79.09	77.43	78.34	0.87
Fixed carbon (% DM)	20.36	19.54	23.85	20.08	20.72	0.82
C (% DM)	54.00	50.32	56.86	51.16	55.13	1.98
H (% DM)	5.83	5.04	6.47	5.74	6.15	0.31
S (% DM)	0.024	0.013	0.054	0.021	0.027	0.007
N (% DM)	0.43	0.30	0.63	0.39	0.46	0.07
Cl (% DM)	0.026	0.012	0.043	0.023	0.030	0.006

Number of observations for all attributes $n = 72$.

Table 8. Selected statistical analysis indicators for the willow characteristics under study.

Attribute	Median	Minimum Value	Maximum Value	Lower Quartile	Upper Quartile	Standard Deviation
Moisture (%)	50.04	47.47	50.99	49.32	50.48	0.98
Ash (% DM)	1.20	0.86	1.72	1.15	1.38	0.18
HHV (GJ Mg^{-1} DM)	19.63	19.44	19.79	19.54	19.72	0.10
Volatile matter (% DM)	78.51	74.42	80.16	77.97	79.35	1.29
Fixed carbon (% DM)	20.16	18.69	24.53	19.50	20.64	1.28
C (% DM)	54.14	49.89	56.03	50.83	54.93	2.07
H (% DM)	5.89	5.19	6.48	5.81	6.29	0.31
S (% DM)	0.024	0.016	0.052	0.022	0.027	0.007
N (% DM)	0.37	0.30	0.52	0.34	0.41	0.05
Cl (% DM)	0.021	0.004	0.035	0.013	0.025	0.008

Number of observations for all attributes n = 72.

4. Conclusions

This study determined the variation of SRWC biomass. Multidimensional analyses were conducted based on the large number of biomass samples collected from three SRWC species cultivated in eight soil enrichment variants and harvested in three consecutive four-year harvest rotations. This study determined the effect of the species, soil enrichment procedure, and harvest rotation, as well as the interactions between these factors on the variation in the thermophysical properties and the elemental composition of SRWC biomass. It was found that the SRWC species determined the moisture, ash, nitrogen contents, and higher heating value in the biomass to the greatest extent. In terms of the SRWC biomass moisture content during the harvest, black locust was characterized by the lowest (i.e., the most favorable values from an energy perspective) values, with the poplar biomass exhibiting the worst results. The poplar biomass was characterized by the highest HHV value, which is beneficial from an energy perspective, but it contained the most ash (which is undesirable). Willow was characterized by the lowest ash content, with a medium moisture content among all the SRWC species under study. This study also found that the harvest rotation largely affected the C, H, and Cl contents in biomass, and the S content was mainly determined by the interaction between the three main factors (the species, soil enrichment procedure, and harvest rotation) and by the plant species itself. This also could have been affected by varied interactions and environmental conditions in subsequent years of plant cultivation. Therefore, in practice, greater variation should be expected in the content of individual elements in SRWC biomass sourced from plantations located under different habitat conditions when applying various fertilization types and levels, despite the cultivation of the same SRWC species at the same locations. Therefore, it can be concluded that there are possibilities for modifying the SRWC biomass composition by selecting species suitable for cultivation and plantation management, which can be used to optimize the quality of woody biomass sourced from field plantations for selected conversion technologies. However, to identify specific applications, it is necessary for SRWC producers and final biomass users to cooperate and exchange information, which, in the current dynamic and difficult political and economic situation, is becoming particularly important, if only from the perspective of partially increasing energy security through the supply of adequate quality SRWC biomass.

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