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Biochar from Grapevine-Pruning Residues Is Affected by Grapevine Rootstock and Pyrolysis Temperature

Dominik Anđelini [1], Danko Cvitan [1,*], Melissa Prelac [1], Igor Pasković [1], Marko Černe [1], Ivan Nemet [2], Nikola Major [1], Smiljana Goreta Ban [1], Zoran Užila [1], Tea Zubin Ferri [3], Branka Njegić Džakula [4], Marko Petek [5], Dean Ban [1] and Igor Palčić [1]

1 Institute of Agriculture and Turism, Karla Huguesa 8, 52440 Poreč, Croatia
2 Faculty of Science, Department of Chemistry, Horvatovac 102a, 10000 Zagreb, Croatia
3 MAR LAB d.o.o., Vukovarska Ulica 22, 52100 Pula, Croatia
4 Ruđer Bošković Institute, Bijenička Cesta 54, 10000 Zagreb, Croatia
5 Department of Plant Nutrition, University of Zagreb Faculty of Agriculture, Svetošimunska Cesta 25, 10000 Zagreb, Croatia
* Correspondence: danko@iptpo.hr

Abstract: In recent years, the production and application of biochar as a soil amendment produced from pruning residues has gained attention worldwide. Since the effect of grapevine rootstock type on grapevine-pruning residues used as feedstock for biochar production had not yet been researched, the present research was performed. Two grapevine rootstocks, different in vigor, were selected, with the hypothesis that they would affect their chemical composition and, consequently, the composition of the produced biochar. In this work, grapevine-pruning residues of the indigenous variety "Istrian Malvasia" (*Vitis vinifera* L.) grafted on 420A and SO4 rootstocks were analyzed and used for biochar production under three peak temperature programs (400 °C, 500 °C, and 600 °C). Higher pyrolysis temperature decreased yield but increased EC, ash, and TC content, as well as the content of most of the studied elements. On the other hand, grapevine rootstock type affected biochar EC, ash content, and specific surface area. Results showed that a more vigorous rootstock affects the produced biochar qualities by enhancing the above-mentioned properties. The present research showed that biochar produced from grapevine-pruning residues, especially at 500 °C or 600 °C, could be a valuable tool for the valorization of this biomass as a soil amendment.

Keywords: canes; grapevine; elemental composition; slow pyrolysis

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1. Introduction

Grapevine is the most widespread fruit species in the world, which, with its total production of almost 7.4 million hectares in the global area, surpasses all other fruit species [1,2]. The grapevine total production area in Croatia accounts for 19 thousand hectares [3]. The region of Istria in Croatia is well known for wine production. The main white grape variety is "Istrian Malvasia", mostly grafted on *V. berlandieri* × *V. riparia* rootstocks 420A Millardet et de Grasset, Sélection Oppenheim 4 (SO4), and Kober 5BB [4]. Istrian producers are progressively implementing new vineyard management practices with the purpose of obtaining better quality the grapes with lower negative environmental impact. Climate change represents the dominant challenge for viticulture in the upcoming decades [5–8]. Global warming is one of the major threats, especially because the increase in air temperature is causing drought and water scarcity [8], ultimately affecting grape quality. The rootstock has an important role in fighting drought stress and plays an important role in increasing the production and quality of grapes [9] through the partitioning of biomass between root, shoot, trunk, and fruit [10], which has an impact on vine growth, productivity, and the vegetative vigor of scions, thereby improving or reducing yield [9,11]. Moreover, natural fertilizers and soil conditioners are more and more in the use.

Among viticultural practices, vineyard canopy management has a particular relevance due to the impact on grapevine vegetative and generative development [12]. Canopy management practices include the yearly activity of pruning, causing vineyard wastes that can also contribute noticeably to water and soil pollution around the world [13]. Owing to the fact that grapevine produces an estimated 2–5 tons of grapevine-pruning residues per hectare per year, depending upon plantation density, climate, and the vigor of the grape variety [14], it can be easily calculated that the amount generated yearly equals 14.8–37 millions of tons globally, and 38–95 thousands of tons in Croatia. Likewise, in the production, several grapevine-pruning residues waste management practices can be found. Grapevine-pruning residues can be used as a source of polyphenols [15], lignin, and bio-ethanol [16], as a winemaking additive [17], material for particleboard and paper production [18,19], compound source for cosmetic industries [20], etc. Mostly, grapevine-pruning residues are burned or chopped and mulched and those practices can have some negative environmental effects. Burning of pruning residues is seen as controversial regarding its contribution to greenhouse gas emissions, while chipping pruning residues and using it as mulch is seen as highly risky regarding pest and disease control [21]. As an alternative, the pyrolysis of organic residues and the utilization of its solid product, known as biochar, as soil amendment is attracting increasing attention [22]. Biochar is a carbon-rich substance that is produced via the pyrolysis of different organic materials in an oxygen-limited environment [23]. The interest in biochar soil application is the result of the demonstrated increase in soil water and nutrient retention [22], and the application of biochar has emerged as a very promising method for solving the multiple issues simultaneously [24]. Biochar can be applied in temperate soils, where drought-like conditions are prevalent, to increase the water holding capacity and bulk density of the soil [25]. Highly significant correlations were found between the sorption capacity of adsorbents and their organic C content, thus confirming the prominent role of organic matter in the interaction and retention of this compound [13]. Biochar has different physicochemical properties depending upon the type of biomass and the pyrolysis temperature used for biochar production [26,27]. A positive correlation between pyrolysis temperature and specific surface area (SSA) is known for most biochars, as more pores were generated at higher pyrolysis temperatures [28], implying a lighter biochar per volume unit. At very high pyrolysis temperatures (800–1000 °C), porosity may fall again. Micropores (0.05–0.0001 µm) make up the majority of biochar's pore structure (>80% of total pore vol.), which indicates, in the first instance, a high water adsorption capacity. The higher pyrolysis temperature results in higher ash content, pH, electrical conductivity (EC), basic functional groups, carbon stability, and total content of C, N, P, K, Ca, and Mg [29]. The higher pyrolysis temperature decreases biochar yield, total content of O, H, and S, the unstable form of organic C, and acidic functional groups [29]. The data of Fourier transformation infrared (FTIR) analysis indicated an increase in aromaticity and a decrease in polarity of biochar produced at a high temperature [29]. The reported results of infrared spectra analysis revealed that the functional groups in biochar decreased along with the decrease in pyrolysis temperature, while the carbonization had an opposite trend [30]. Biochars derived at low temperatures (<400 °C) are characterized by high-energy content and high volatile matter which contains easily decomposable substrates [31]. The composition and proportions of cellulose, hemicellulose, lignin, and extractives vary depending on the type of biomass used for biochar production. The pyrolysis of these components results initially in the thermal degradation of hemicelluloses and celluloses at 200–255 °C and 235–345 °C, respectively, and finally between 275 °C and 495 °C for lignin.

Previous research on biochar produced from grapevine-pruning residues did not present data about grapevine rootstock's effect on biochar properties. In this research, the grapevine-pruning residues used were from *Vitis vinifera* L. variety "Istrian Malvasia", grafted on two different rootstocks (420A and SO4), which are most common in the Istria region. These two rootstocks were selected due to their different vigor levels and their capacity for producing different weights and chemical compositions of pruning residues [32].

The hypothesis was that a more vigorous rootstock—in this case, SO4—would uptake more nutrients from the soil and, consequently, produce biochar with better properties for application as a soil amendment. Furthermore, the aim of this research was to investigate the effects of different pyrolysis temperatures on physicochemical characteristics of the biochar produced from grapevine-pruning residues. The second hypothesis was that a higher pyrolysis peak temperature will produce biochar with higher ash and nutrient content, pH, and specific surface area.

2. Materials and Methods

2.1. Pyrolysis Conditions

The biochars produced in this research were obtained through pyrolysis at three different temperatures (400 °C, 500 °C, and 600 °C) from grapevine-pruning residues. These temperatures were selected according to previous research on grapevine-pruning residues [33] with temperature peak differences big enough to expect a significant effect of pyrolysis temperature. The experiment was performed on pruning residues of the "Istrian Malvasia" (*Vitis vinifera* L.) variety, the most widespread and economically important native white grape cultivar in Croatia, grafted on two different rootstocks; 420A (Millardet et de Grasset; *V. berlandieri* × *V. riparia*) and SO4 (Selection Oppenheim; *V. berlandieri* × *V. riparia*). The pruning residues were collected from the experimental vineyard of the Institute of Agriculture and Tourism (Poreč, Istria, Croatia (lat. 45°13′22″ N; long. 13°36′02″ E; 15 m asl)). Biochar was produced using a muffle furnace (Nabertherm Muffle Furnace L9/11/B410, Nabertherm GmbH, Lilienthal, Germany). Grapevine-pruning residues were placed in ceramic crucibles with ceramic covers to minimalize the oxygen contact. The following peak temperature programs were used: after a 10 °C/min ramp up, the maximum temperatures were reached—(a) 400 °C; (b) 500 °C, and (c) 600 °C—and held for one hour. Subsequently, the samples were left to cool to room temperature. Biochar samples were produced in three replicates and analyzed separately.

2.2. Biochar Characterisation

Biochar yield was determined gravimetrically and calculated using the following equation [34]:

$$Yield(\%) = \frac{m\mathrm{BC}}{m\mathrm{PR}} \times 100 \quad (1)$$

where *Yield* (%) equals the mass yield of biochar, expressed as a percentage (%), mBC equals mass of biochar, expressed in kg, and mPR equals mass of pruning residues, expressed in kg. To calculate the exact ash percentage, the following equation was used:

$$Ash(\%) = \frac{m\mathrm{ASH}}{m\mathrm{SAMPLE}} \times 100$$

where *Ash* (%) equals the mass content of ash, expressed as a percentage (%), mASH equals mass of produced ash, expressed in g, and mSAMPLE equals the mass of sample (grapevine-pruning residues or biochar), expressed in g. The grapevine-pruning residues were milled through a 0.2 mm sieve using electrical centrifugal mill (ZM200, Retsch GmbH, Haan, Germany). The biochar samples were ground to powder and homogenized in a mortar before analysis.

Grapevine-pruning residues and biochar ash content were determined by mineralizing 1 g of the sample in ceramic crucibles using a muffle furnace (Nabertherm Muffle Furnace L9/11/B410, Nabertherm GmbH, Lilienthal, Germany) with the following temperature program: 0–105 °C ramp 20 min, 105–750 °C ramp for 5 h.

The grapevine-pruning residues' pH analysis was conducted using 5 mL of air-dried sample and 25 mL deionized water (1:5; v/v). The biochar pH analysis was conducted according to DIN ISO 10390. Briefly, 5 mL of air-dried biochar was mixed with 25 mL (ratio 1:5; v/v) of 0.01 M $CaCl_2$ and rotated for 1 h. The pH value of the mixture was measured using a pH meter (inoLab Multi 9310 IDS, Xylem Inc., Washington, WA, USA).

Electrical conductivity (EC) of grapevine-pruning residues and biochar was measured by mixing 1 g of air-dried sample with 25 mL of deionized water and rotating for 1 h (ratio of 1:25; m/v). The EC was measured from the obtained suspension using an EC meter (FiveGo F3, Mettler Toledo AG, Columbus, OH, USA).

The total carbon (TC) content of grapevine-pruning residues and biochar samples was detected by burning 50 mg of grounded sample in the Solid Sample Combustion Unit (SSM-5000A) on TOC-L analyzer (Shimadzu Corporation, Kyoto, Japan).

Nitrogen (N) content of grapevine-pruning residues and biochar was determined by the Kjeldahl method [35]. For digestion, 1 g of grounded sample, 12 mL of H_2SO_4, and 2 KJTabs™ tablets were used and a 1 h digestion at 420 °C program was performed. After digestion and cooling, 30 mL of H_3BO_4 and 50 mL of NaOH were used for distillation on UDK 149 Nitrogen Analyzer (VELP Scientifica Srl., Usmate Velate, Italy). Titration was performed with 0.1N HCl.

The grapevine-pruning residues and biochar elemental analysis was performed by ICP-OES (Shimadzu Corporation, Kyoto, Japan) after microwave digestion (Ethos UP, Millestone Srl, Milan, Italy). Microwave digestion of the samples was performed using 6 mL of HNO_3 and 2 mL of H_2O_2 added to 250 mg of the sample. The program was set for a 25 min ramp to 200 °C and held for 15 min. Microwave digestion of biochar was performed in two steps. In the first step, 6 mL of HNO_3, 2 mL of H_2O_2, and 0.4 mL of HF were added to the 200 mg of grounded biochar. The program was set for 15 min temperature ramp up to 190 °C, holding that temperature for 20 min. After cooling, in the second step, 5 mL of 4% H_3BO_3 were added. Temperature ramp lasted eight minutes up to 160 °C and was held for seven minutes.

Specific surface area (SSA) was determined by nitrogen adsorption under the liquid nitrogen temperature of −196 °C according to the Brunauer–Emmett–Teller (BET) method [36]. Gemini 2380 Surface Area Analyzer (Micromeritics, Norcross, GA, USA) was used for the analysis of previously grounded and dried grapevine-pruning residues and biochar.

The surface morphology was observed with a scanning electron microscope combined with a field emission gun (QUANTA 250 FEG—SEM, FEI Company, Hillsboro, OR, USA).

For the Fourier transform infrared spectroscopy, grapevine-pruning residues and biochar samples were grounded to powder in a mortar and mixed thoroughly with potassium bromide (KBr) with a 1/150 mass ratio in order to make pressed pellets. The spectra were recorded in the wave number range of 400–4000 cm^{-1} at a resolution of 4.0 cm^{-1} using the FTIR spectrometer (Shimadzu IRTracer-100, Shimadzu Corporation, Kyoto, Japan).

2.3. Statistical Analysis

Statistical analyses on the obtained results were performed with the software Statistica 12 (Tibco, Inc., Palo Alto, CA, USA). The data were processed by a *t*-test (grapevine-pruning residues) or factorial (biochar) analysis of variance (ANOVA) to evaluate the influence of the individual factors "rootstock" and "pyrolysis temperature" and their possible interaction. A post hoc Fischer's LSD test with 95% confidence level was used for pairwise evaluation between treatments.

3. Results

3.1. Characterisation of Grapevine-Pruning Residues

The physicochemical properties of the analyzed grapevine-pruning residues are presented in Table 1. Ash content values were significantly different with the SO4 rootstock value (3.71%), being higher compared to the 420A rootstock (3.27%). On the other hand, the measured pH value and EC were found to be significantly higher in the 420A rootstock compared to the SO4 rootstock (Table 1). Total carbon content in pruning residues was not significantly different between rootstocks and the obtained values were 44.4% and 44.5% for the SO4 and 420A rootstocks, respectively.

Table 1. Physicochemical properties of grapevine-pruning residues.

Parameter	Unit	Rootstock		Significance
		SO4	**420A**	
ash	%	3.71 ± 0.01	3.27 ± 0.01	***
pH	/	5.21 ± 0.02	5.34 ± 0.02	**
EC	µS/cm	2240 ± 70.0	2456 ± 8.82	*
TC	%	44.4 ± 0.32	44.5 ± 0.42	n.s.
N	%	0.61 ± 0.05	0.61 ± 0.06	n.s.
P	g/kg	0.68 ± 0.04	0.63 ± 0.02	n.s.
K	g/kg	9.14 ± 0.63	10.9 ± 1.12	n.s.
S	g/kg	0.35 ± 0.02	0.29 ± 0.02	n.s.
Ca	g/kg	5.91 ± 0.95	5.19 ± 0.57	n.s.
Mg	g/kg	0.95 ± 0.10	0.85 ± 0.05	n.s.
Na	g/kg	34.2 ± 2.48	41.5 ± 6.04	n.s.
As	mg/kg	n.d.	n.d.	/
Cu	mg/kg	3.71 ± 0.95	3.37 ± 0.57	n.s.
Fe	mg/kg	92.8 ± 41.5	27.7 ± 3.76	n.s.
Mn	mg/kg	25.1 ± 3.83	12.1 ± 1.84	*
Mo	mg/kg	0.19 ± 0.01	0.17 ± 0.02	n.s.
Ni	mg/kg	0.24 ± 0.10	0.04 ± 0.02	n.s.
Pb	mg/kg	n.d.	n.d.	/
Se	mg/kg	0.02 ± 0.24	0.06 ± 0.23	n.s.
Zn	mg/kg	8.64 ± 3.13	4.66 ± 1.44	n.s.
SSA	m^2g^{-1}	1.02 ± 0.01	1.05 ± 0.01	n.s.

Data were subjected to *t*-test *, **, ***, n.s. indicate significant differences at $p < 0.05$, 0.01, 0.001, or not significant, respectively.

The elemental composition of the grapevine-pruning residues is shown in Table 1. There was no significant effect of rootstock type on pruning residues' elemental composition, except for manganese (Mn) content.

Specific surface area values were also not significantly different between the investigated rootstocks.

3.2. Biochar Characterisation

The investigated physicochemical properties of the biochar produced from grapevine-pruning residues of the "Istrian Malvasia" variety, grafted on two different rootstocks and under different peak temperature programs, are presented in Table 2. Temperature had a strong effect and showed significant differences for all researched parameters. The yield of produced biochar decreased with the rise of the temperature program, from 34.0% at 400 °C to 28.7% at 600 °C. On the other hand, the type of rootstock did not show significant effect on biochar yield. There was also no significant interaction effect between rootstock type and pyrolysis temperature on the biochar yield. A higher pyrolysis peak temperature significantly increased the ash content from 8.36% (400 °C) to 9.52% (600 °C). Rootstock type, pyrolysis temperature, and the interaction of these factors showed a significant effect on pH values, which were in a range from 9.00 to 9.79. The pH value was the highest at the temperature 500 °C, lower at 600 °C, and the lowest on 400 °C. On a particular pyrolysis temperature, pH value was the same between two rootstocks, except on the 600 °C program where SO4 showed a higher value. Biochar EC values ranged from 792 to 2318 μS/cm and temperature had a strong effect on it. A significant increase in EC value was reported under the 600 °C pyrolysis program. Biochar ash content was significantly affected by both grapevine rootstock type and pyrolysis peak temperature. Rootstock SO4 showed significantly higher ash content compared to rootstock 420A. Following pyrolysis peak temperature increase, biochar ash content increased from 8.36% at 400 °C to 9.52% at 600 °C. The strong effect of temperature was also noted for TC content, where the TC content of biochar significantly increased from 73.1% at 400 °C to 75.8% at 500 °C, and finally to 77.4% at 600 °C. The rootstock choice did not affect the TC content.

Table 2. Properties of biochar produced from grapevine pruning residues of Istrian Malvasia grafted on two different rootstocks after pyrolyzation at three different temperatures.

	Yield	**pH**	**EC**	**Ash**	**TC**
	%		**μS/cm**	**%**	**%**
Rootstock					
SO4	30.6 ± 0.01	9.53 ± 0.13 a	1727 ± 257 a	9.51 ± 0.01 a	75.60 ± 0.64
420A	31.1 ± 0.01	9.38 ± 0.12 b	1094 ± 233 b	8.29 ± 0.01 b	75.29 ± 0.73
p value	n.s.	**	***	***	n.s.
Temperature					
400 °C	34.0 ± 0.01 a	9.79 ± 0.05 a	792 ± 65.9 c	8.36 ± 0.01 c	73.1 ± 0.43 c
500 °C	29.9 ± 0.01 b	9.00 ± 0.03 c	1123 ± 249 b	8.81 ± 0.01 b	75.8 ± 0.26 b
600 °C	28.7 ± 0.01 c	9.58 ± 0.10 b	2318 ± 158 a	9.52 ± 0.01 a	77.4 ± 0.23 a
p value	***	***	***	***	***
Rootstock × Temperature			400 °C		
SO4	33.9 ± 0.01	9.78 ± 0.46 a	921 ± 80.9	9.03 ± 0.01	73.5 ± 0.65
420A	33.9 ± 0.1	9.79 ± 0.19 a	663 ± 93.5	7.69 ± 0.01	72.7 ± 1.39
			500 °C		
SO4	29.6 ± 0.01	9.02 ± 0.95 c	1595 ± 196	9.49 ± 0.01	75.6 ± 0.68
420A	30.1 ± 0.01	8.97 ± 0.05 c	651 ± 470	8.13 ± 0.01	76.0 ± 0.65
			600 °C		
SO4	28.3 ± 0.01	9.79 ± 0.92 a	2666 ± 55.1	10.0 ± 0.01	77.7 ± 0.71
420A	29.1 ± 0.01	9.37 ± 0.70 b	1969 ± 95.9	9.04 ± 0.01	77.2 ± 0.37
p value	n.s.	**	n.s.	n.s.	n.s.

Data were subjected to factorial ANOVA: *, **, ***, n.s. indicate significant differences at $p < 0.05$, 0.01, 0.001, or not significant. Within rootstock and pyrolysis temperature, different letters indicate significant differences (LSD test, $p < 0.05$).

The most abundant elements present in biochar produced from grapevine pruning residues are shown in Table 3. Rootstock type had a significant effect only on S content. Pyrolysis temperature had a significant effect on the content of all the most abundant elements except N, while a significant interaction was observed between rootstock and temperature for P content, where the differences between the researched rootstocks were observed only at a temperature of 400 °C. The rootstock SO4 showed significantly lower P content compared to the other temperatures, while, for 420A rootstock, the highest *p* values were observed at 600 °C.

Table 3. Content of the most abundant elements in biochar produced from Istrian Malvasia grafted on different rootstocks after pyrolyzation at different temperatures.

	N	P	K	S	Ca	Mg	Na
	%	g/kg	g/kg	g/kg	g/kg	g/kg	g/kg
Rootstock							
SO4	1.07 ± 0.01	24.3 ± 1.02	43.8 ± 4.81	14.8 ± 0.53 a	224 ± 12.4	31.0 ± 1.46	15.0 ± 1.80
420A	1.04 ± 0.05	25.1 ± 0.69	42.1 ± 4.45	13.1 ± 0.37 b	212 ± 9.36	33.1 ± 1.23	15.3 ± 1.58
p-value	n.s.	n.s.	n.s.	***	n.s.	n.s.	n.s.
Temperature							
400 °C	1.06 ± 0.01	22.0 ± 0.82 b	28.6 ± 0.43 b	12.6 ± 0.32 b	182 ± 3.79 b	27.7± 0.96 b	9.65 ± 0.47 b
500 °C	1.03 ± 0.03	25.3 ± 0.72 a	45.9 ± 5.76 a	14.2 ± 0.68 a	227 ± 9.26 a	33.4 ± 0.82 a	16.4 ± 1.45 a
600 °C	1.07 ± 0.07	26.8 ± 0.36 a	54.3 ± 1.33 a	15.1 ± 0.49 a	246 ± 8.13 a	35.1 ± 1.34 a	19.4 ± 1.24 a
p-value	n.s.	***	***	***	***	***	***
Rootstock × Temperature							
				400 °C			
SO4	1.07 ± 0.01	20.5 ± 0.87 d	28.7 ± 1.17	12.9 ± 0.86	182 ± 12.5	26.4 ± 1.82	9.43 ± 0.92
420A	1.04 ± 0.01	23.5 ± 1.51 c	28.6 ± 1.16	12.24 ± 0.69	181 ± 7.57	29.1 ± 2.29	9.88 ± 1.51
				500 °C			
SO4	1.08 ± 0.01	26.1 ± 1.74 ab	46.2 ± 15.3	15.7 ± 0.39	242 ± 23.7	33.2 ± 2.95	16.4 ± 4.36
420A	0.98 ± 0.08	24.4 ± 1.56 bc	45.7 ± 16.2	12.7 ± 0.53	211 ± 1.94	33.5 ± 1.10	16.4 ± 3.54
				600 °C			
SO4	1.05 ± 0.05	26.4 ± 0.46 ab	56.4 ± 2.82	15.9 ± 1.06	248 ± 23.7	33.5 ± 4.03	19.3 ± 4.41
420A	1.09 ± 0.27	27.3 ± 1.06 a	52.1 ± 2.08	14.3 ± 0.79	244 ± 12.0	36.8 ± 1.57	19.4 ± 1.86
p-value	n.s.	*	n.s.	n.s.	n.s.	n.s.	n.s.

Data were subjected to factorial ANOVA: *, **, ***, n.s. indicate significant differences at $p < 0.05$, 0.01, 0.001, or not significant. Within rootstock and pyrolysis temperature, different letters indicate significant differences (LSD test, $p < 0.05$).

Rootstock type did not significantly affect the content of less abundant elements present in biochar, except for Mn where the 420A rootstock had a significantly higher value (3.01 mg/kg) compared to the SO4 rootstock (6.99 mg/kg), which is more than double the difference (Table 4). The pyrolysis temperature program had an influence only on Cu and Ni content, which is also shown in Table 4. Both the Cu and Ni content increased with temperature, where the biochar produced at 600 °C had significantly higher values compared to 400 °C in the case of Cu and even 500 °C in the case of Ni. Interaction between rootstock and temperature had a significant influence on Mo and Zn content. The content of Mo and Zn was the highest in biochar from rootstock SO4, pyrolyzed at 500 °C.

When looking at specific surface area results, it is noticeable that rootstock type, pyrolysis temperature, and the interaction between them were highly significant (Table 5). Biochar samples produced from the grapevine-pruning residues of the more vigorous rootstock SO4 had a higher specific surface area when the biochar was produced at lower peak temperature (400 °C).

Scanning electron microscope (SEM) pictures of grapevine pruning residues and produced biochar are shown in Figure 1. That biochar had a higher number of pores compared with grapevine pruning residues is visible. When we take into account the influence of pyrolysis temperature, it can be seen that biochars produced at higher temperatures showed finer pores and a more hexagonal shape compared with the ones produced at lower temperatures.

Table 4. Content of the less abundant elements in biochar produced from Istrian Malvasia grafted on different rootstocks after pyrolyzation at different temperatures.

	As	Cu	Fe	Mn	Mo	Ni	Pb	Se	Zn
	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg
Rootstock									
SO4	0.10 ± 0.03	5.05 ± 0.15	6.38 ± 0.37	6.99 ± 0.26 a	0.10 ± 0.01	0.14 ± 0.03	0.65 ± 0.08	0.07 ± 0.02	3.82 ± 0.42
420A	0.33 ± 0.14	5.15 ± 0.44	6.74 ± 0.47	3.01 ± 0.08 b	0.08 ± 0.01	0.12 ± 0.02	0.65 ± 0.08	0.12 ± 0.07	3.99 ± 0.16
p-value	n.s.	n.s.	n.s.	***	n.s.	n.s.	n.s.	n.s.	n.s.
Temperature									
400 °C	0.08 ± 0.04	4.36 ± 0.34 b	6.60 ± 0.84	4.89 ± 0.88	0.09 ± 0.01	0.05 ± 0.01 c	0.49 ± 0.04	0.05 ± 0.01	3.59 ± 0.31
500 °C	0.19 ± 0.14	5.16 ± 0.27 ab	6.34 ± 0.40	4.94 ± 0.97	0.10 ± 0.02	0.12 ± 0.02 b	0.65 ± 0.11	0.05 ± 0.01	4.48 ± 0.38
600 °C	0.36 ± 0.17	5.77 ± 0.37 a	6.74 ± 0.09	5.16 ± 0.91	0.08 ± 0.01	0.23 ± 0.03 a	0.80 ± 0.08	0.19 ± 0.10	3.63 ± 0.38
p-value	n.s.	**	n.s.	n.s.	n.s.	***	n.s.	n.s.	n.s.
Rootstock × temperature					400 °C				
SO4	0.12 ± 0.12	4.73 ± 0.29	5.61 ± 1.37	6.76 ± 1.02	0.08 ± 0.02 b	0.05 ± 0.00	0.49 ± 0.09	0.05 ± 0.00	3.10 ± 0.28 b
420A	0.05 ± 0.04	3.98 ± 1.11	7.58 ± 2.39	3.03 ± 0.29	0.09 ± 0.01 ab	0.05 ± 0.00	0.48 ± 0.12	0.05 ± 0.00	4.08 ± 0.77 ab
					500 °C				
SO4	0.05 ± 0.00	5.30 ± 0.98	6.69 ± 1.29	7.06 ± 0.83	0.13 ± 0.04 a	0.11 ± 0.04	0.67 ± 0.27	0.05 ± 0.00	5.16 ± 0.89 a
420A	0.23 ± 0.48	5.02 ± 1.02	5.99 ± 0.64	2.82 ± 0.21	0.07 ± 0.01 b	0.12 ± 0.05	0.64 ± 0.34	0.05 ± 0.00	3.80 ± 0.18 b
					600 °C				
SO4	0.11 ± 0.13	5.11 ± 0.69	6.84 ± 0.29	7.14 ± 0.80	0.08 ± 0.03 b	0.26 ± 0.07	0.78 ± 0.26	0.12 ± 0.13	3.19 ± 1.14 b
420A	0.61 ± 0.45	6.43 ± 0.55	6.64 ± 0.12	3.19 ± 0.15	0.08 ± 0.02 b	0.19 ± 0.02	0.82 ± 0.14	0.26 ± 0.36	4.07 ± 0.51 ab
p-value	n.s.	n.s.	n.s.	n.s.	*	n.s.	n.s.	n.s.	*

Data were subjected to factorial ANOVA: *, **, ***, n.s. indicate significant differences at $p < 0.05$, 0.01, 0.001, or not significant. Within rootstock and pyrolysis temperature, different letters indicate significant differences (LSD test, $p < 0.05$).

Table 5. Specific surface area of biochar samples in regard to pyrolysis temperature and rootstock.

	SSA (m^2g^{-1})
Rootstock	
SO4	1.60 ± 0.20 a
420A	1.36 ± 0.10 b
p-value	***
Temperature	
400 °C	2.07 ± 0.14 a
500 °C	1.14 ± 0.02 b
600 °C	1.25 ± 0.02 b
p-value	***
Rootstock × Temperature	400 °C
SO4	2.38 ± 0.02 a
420A	1.76 ± 0.02 b
	500 °C
SO4	1.15 ± 0.05 e
420A	1.12 ± 0.03 e
	600 °C
SO4	1.28 ± 0.01 c
420A	1.21 ± 0.01 d
p-value	***

Data were subjected to factorial ANOVA: *, **, ***, n.s. indicate significant differences at $p < 0.05$, 0.01, 0.001, or not significant. Within rootstock and pyrolysis temperature, different letters indicate significant differences (LSD test, $p < 0.05$).

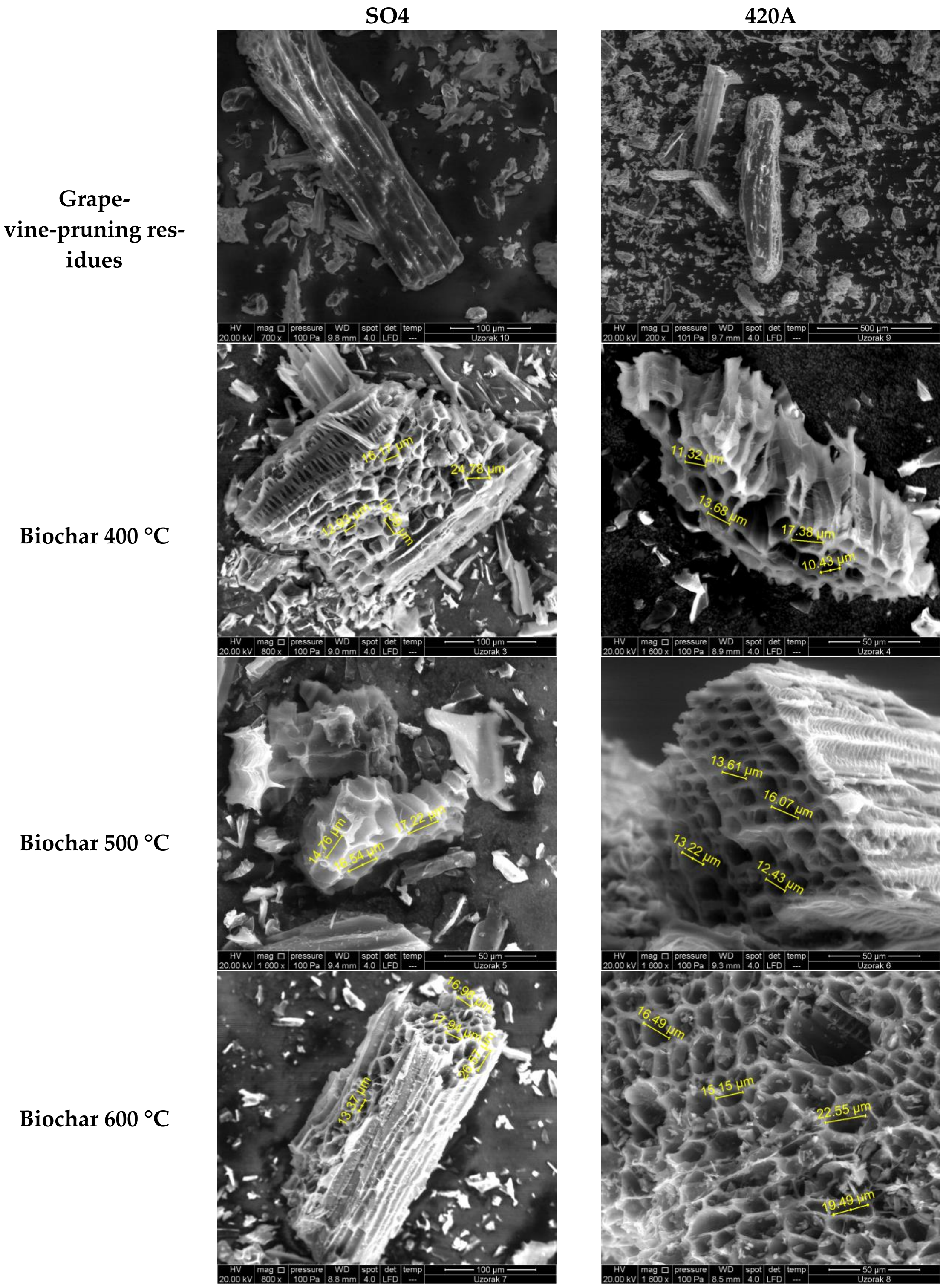

Figure 1. Scanning electron microscope (SEM) pictures of grapevine-pruning residues of Istrian Malvasia grafted on two different rootstocks (420A and SO4) and biochar produced at different pyrolysis temperatures (400 °C, 500 °C and 600 °C).

The obtained FTIR spectra showed the expected peaks and shapes typical for amorphous carbon as observed for other biochars produced from lignin-rich feedstocks. Nevertheless, some differences in the composition of the biochars could be noted—the biochar produced from SO4 rootstock at peak temperature of 400 °C presented different spectra compared to the others (Figure 2).

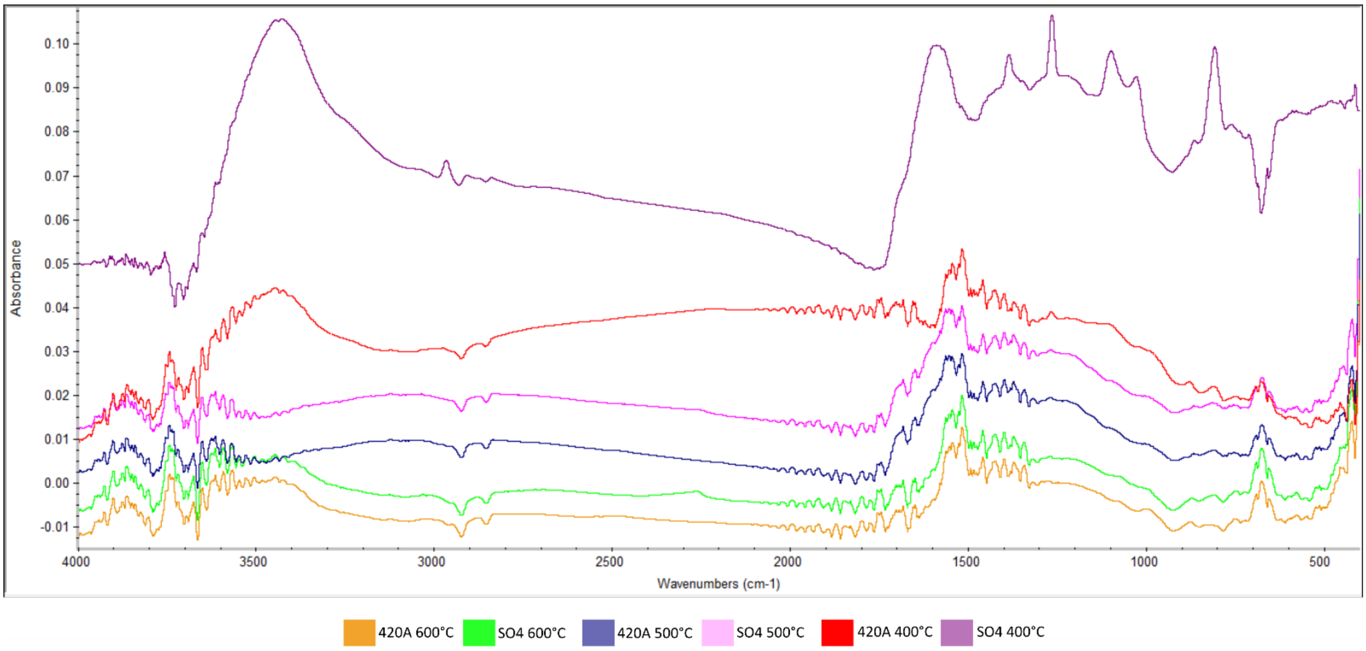

Figure 2. FTIR spectra of biochar produced from the grapevine-pruning residues of Istrian Malvasia grafted on two different rootstocks (420A and SO4) at different temperature programs (400 °C, 500 °C and 600 °C).

4. Discussion

Biomass is transformed, through pyrolysis and carbonization, into biochar, a carbon-rich microporous material which has a well-developed porous structure [37]. Biochar has different physicochemical properties depending upon the type of biomass used and the pyrolysis temperature used for biochar production [27], which this study also confirmed. Temperature is one of the most influential parameters for the final biochar yield during the pyrolysis process [38]. Therefore, some authors [29] studied the effect of pyrolysis temperature on *Conocarpus erectus* L. biochar yield and reported that as pyrolysis temperature increases, the yield decreases. In our study, it was confirmed that the biochar yield decreased from 34.0% to 28.7% with the increase in temperature from 400 °C to 600 °C. It confirmed results reported by Reza et al. [39] where biochar yield decreased from 35% at 400 °C to 23% at 600 °C. The decrease in the biochar yield with increasing temperature could be due either to the greater primary decomposition of the wood at higher temperatures or to the secondary decomposition of the biochar [40]. Likewise, lower yield of biochar at higher temperature (600 °C) is correlated with the emission of more gasses such as CH_4, CO, and CO_2 [41]. Brewer et al. [42] also reported that lower biochar yield at higher pyrolysis temperature could mainly be attributed to the rising volatile matter amount.

This research showed that when a particular pyrolysis temperature was applied, pH value was not statistically different between rootstocks SO4 and 420A, except at 600 °C peak temperature, where SO4 had a higher pH value. Considering pyrolysis peak temperature, biochar pH value was the highest at 400 °C, while the pH value at 600 °C was higher compared to the pH value at 500 °C. These results are not confirmed by other authors, where the pH value increased following peak temperature increase [43]. The reported grapevine-pruning residues' pH was acidic, while the produced biochar had an alkaline pH, which can be important when implementing biochar as a soil amendment or in some other application where pH value is important.

The EC value of biochar did not follow the same pattern as pH values; it increased following peak temperature increase. The peak temperature of 600 °C increased the EC value compared to both 400 °C and 500 °C, probably due to the lower biochar yield at 600 °C and thus the higher concentration effect regarding ion quantity. Regarding the grapevine rootstock effect, SO4 showed higher values compared to 420A rootstock, probably due to the fact that it is more vigorous and can uptake more ions. The availability of soluble nutrient ions such as NO_3^-, K^+, and Ca^{2+} could be directly related to the soluble salt content and the EC of biochar when applied to soil [44]. With a potential increase in the dose of biochar as a soil amendment, the EC of the soil also increases [45], and this is the reason why monitoring the EC value of biochar is very important.

Biochar ash content increased with higher pyrolysis temperature. These results confirmed results reported by different authors [43,46,47]. The higher biochar ash content at higher pyrolysis temperatures could be explained by the degradation of organic material and the volatilization of C, H, O, and volatile solids [48]. Ash content was also significantly affected by grapevine rootstock type, where the more vigorous SO4 rootstock showed higher values compared to the less vigorous 420A rootstock. It was probably due to the higher uptake of elements, leading to the higher content of elements in grapevine-pruning residues and, consequently, higher biochar ash content.

Increasing pyrolysis peak temperature significantly increased biochar TC content, probably due to volatilization losses of other elements, especially H and O, and confirmed results reported by Ippolito et al. [46]. The average TC content in grapevine pruning residues was 44.5%, which is similar to other studies [49–51], where authors reported values from 44.1% to 47.8%. In biochar, the average TC content was 75.4%, which agrees with the results of other studies (73.5%) [50], while a recent study reported even lower TC content (69.4%) [52]. Biochar TC content was not affected by rootstock type, showing a predominant effect of grapevine-pruning residues as feedstock [53]. Biochar total carbon is made up of easily degradable organic carbon compounds and very stable, polycondensed, aromatic carbon structures (black carbon). Black carbon content is an important criterion for characterizing biochar, and it also reflects the biochar's stability as a soil amendment [54].

It is widely accepted that the essential or nutritious ash-forming elements for plants and animals can be macronutrients such as K, Ca, Mg, P, and S, and micronutrients such as Fe, Mn, and Cl. The elements Al and Na are normally non-essential for plant growth [55]. Mostly, there was no significant difference in the elemental composition of grapevine-pruning residues, except for Mn. However, these results are expressed based on dry weight and were probably affected by the dilution effect where rootstock SO4 produced around 20% more grapevine-pruning biomass compared to 420A (data not shown). On the other hand, the results of biochar elemental composition were different, mostly referring to macroelements and other elements present in higher amounts. For P, K, S, Ca, Mg, and Na, the temperature of 400 °C showed lower biochar content values, while the highest contents were reported at 600 °C or were comparable at 500 °C and 600 °C. Biochar yield after the pyrolysis process was around 30%, but just a few of the analyzed elements were concentrated three times (as would be expected) in biochar compared with the initial feedstock: grapevine-pruning residues. Elements such as Cu and Se were less than 1 time higher in biochar. The content of K was 4–5 times higher, but S, P, Mg, and Ca contents were from 32 to 45 times higher compared with grapevine-pruning residues. The contents of elements such as Fe, Mn, Mo, Zn, and Na were 1–14.5 times lower in biochar compared with the pruning residues, suggesting that some losses occur during the pyrolysis process. All the analyzed biochar contained higher Ca and Mg content compared to the initial feedstock, which was probably because these nutrients volatilize only at temperatures higher than 1000 °C [56]. Other authors [57] assume that such a high concentration of Ca might be due to the bioconversion of organic materials into biogas, resulting in a predictable liberation of Ca, which interacts with CO_3^- or PO_4^{-3} and precipitates. The rising biochar Ca quantity was 37.9–40.8 times higher compared with grapevine-pruning residues. The content of P during the pyrolysis process increased 35.7–39.8 times compared with the initial feedstock.

Some authors claim that elements such as Ca and P could be present in oxide or calcite forms, which can be volatilized or calcined at the high pyrolysis temperature, thus causing the compositional and configurationally differences of the produced biochar [56]. The total Mn content of biochar decreased 3.5–4.1 times compared with grapevine-pruning residues. The content of Mn, together with other heavy metals such as Cu, Ni, and Pb, was far below the maximum allowed values declared by the Commission of the European Union [58] for biochar application as a soil amendment.

Specific surface area (SSA) data shows that biochar samples produced from pruning residues of the more vigorous rootstock SO4 had higher specific surface area regardless of the fact that the significant difference in the SSA of grapevine-pruning residues from different rootstocks was not noticed. Although some authors [28] reported that higher pyrolysis temperature increase specific surface area, from all observed pyrolysis temperatures in this research, the lowest (400 °C) caused the highest specific surface area. Measured SSA values are in accordance with de la Rosa et al. [59], showing values lower than 5 m^2g^{-1}. The specific surface area is often associated with sorption and retention properties for nutrients and contaminants [60] and is thus important for biochar's potential use as a soil amendment.

The SEM images of grapevine-pruning residues showed similarity in their surface morphology, having a non-porous structure. On the other hand, biochar particles showed different sizes and shapes that may be attributed to the sample grinding. During pyrolysis, pores are expanded by the high pressure generated by the rapid evolution of volatiles [61]. As reported by de la Rosa et al. [59], grapevine-pruning biochar showed good anatomical preservation of the initial feedstock. Xylem vessels with scalariform perforation plates were also observed. Differences in the structure of biochars produced at different temperatures were observed. Results followed the trend reported by Kim et al. [62], showing that peak temperature increase causes a porosity increase due to aromatic arrangements. In the present research, biochars mostly contained pores with diameters ranging from 10.43 μm to 26.57 μm, which could be characterized as micropores (5–30 μm). Micropores usually store plant available water which could be progressively released and help alleviate drought stress [63]. In this experiment, a few small holes and cracks were present in the biochar, especially in biochar produced at higher temperatures, due to the generation of volatile substances during the process of carbonization [64], and they can be characterized as macropores (>75 μm), confirming results reported by Marshall et al. [65]. Macropores usually contain air and improve soil water–air ratio.

FTIR spectra show that biochar produced at lower temperatures (400 °C) and from more vigorous rootstock (SO4) showed some signals in the interval 1600–400 cm^{-1}, suggesting that some lignin structures were still present [66]. Thus, the FTIR specific spectra associated with lignin content can be a fast method to confirm the quality of the pyrolysis process. At a higher temperature of 500 °C, those signals are lost, indicating complete pyrolysis of the grapevine-pruning residues. No substantial differences could be observed among samples produced at 500 °C and 600 °C, indicating that complete pyrolysis was obtained already at 500 °C. Grapevine rootstock effect was not visible on other spectra at all applied temperature peaks. All spectra showed peaks around 3600 and 3350 cm^{-1}, attributed to O–H vibrations [67]. Peaks around 3000–3100 cm^{-1} can be attributed to aromatic C–H groups. Peaks around 1690 cm^{-1} are usually assigned to aromatic C=C bending and C=C alkene stretching [59]. Our results confirmed results reported by Gamiz et al. [68], suggesting a decrease in O–H stretching around 3400 cm^{-1} following peak temperature increase due to a loss of hydration and the C–H stretching of aliphatic vibration groups.

The grapevine rootstock effect was different throughout the researched biochar parameters. Even though some authors reported that type of feedstock biomass affects the biochar yield [69,70] in the present research, it was confirmed only at first level, considering grapevine-pruning residues as feedstock; it was not confirmed on second level, considering grapevine rootstock effects. Grapevine-pruning residues from rootstock 420A had a significantly higher pH value compared to SO4, while pH values of the biochar produced from these rootstocks showed an opposite trend. The effect of grapevine rootstock type was the same on the ash content and EC in grapevine-pruning residues, while in biochar samples, rootstock SO4 showed higher values for both parameters. The composition of biochar ash is usually directly related to the biomass used as feedstock because the original constituents in biomass are the precursors of the newly formed components in biochar ash [61].

5. Conclusions

The results of this experiment indicated that increasing the pyrolysis peak temperature resulted in higher biochar EC, ash, and TC content, as well as a higher concentration of most of the studied elements (C, P, K, S, Ca, Mg, Na, Cu, Mn, As, Ni, Pb, Se). Biochar yield and specific surface area decreased along with the increase in pyrolysis peak temperature. Biochars produced from grapevine-pruning residues at 400 °C had the highest biochar yield, the highest pH value, and the highest SSA value. On the other hand, biochars produced at 400 °C had the lowest TC content and the lowest content of most of the studied elements compared to the other peak temperatures, which is of practical importance in terms of carbon sequestration and biochar's quality as a soil amendment. Biochars produced at a peak temperature of 500 °C had the lowest pH and EC value. Pyrolysis peak temperature of 600 °C produced biochars with the highest EC, TC, and ash content, as well as porosity visible on SEM images. Biochars produced from grapevine-pruning residues at peak temperatures of 500 and 600 °C showed better physicochemical characteristics for biochar application as a soil amendment due to the highest content of most of the studied elements. An additional positive effect of this valorization method is the fact that it turns acidic grapevine-pruning residues into alkaline biochar, suitable especially for application in soils with lower pH. The grapevine rootstock type affected biochar EC, ash content, and SSA values; the higher vigor of SO4 rootstock was reflected in the higher values of the researched parameters. It suggests that the application of biochar from grapevine-pruning residues should be performed in the same vineyard from where the residues come from, since a more vigorous rootstock would produce 'richer' biochar and, at the same time, would present higher grapevine growth requirements. Grapevine-pruning-residues-derived biochar produced on site could be a valuable tool for both the valorization of this valuable biomass and the preservation of soil quality. Future research should focus on the cultivar's effect on grapevine-pruning-residues-derived biochar's physiochemical properties and its potential as a soil amendment.

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Abbreviations

The following abbreviations are used in this manuscript (in appearing order):

SSA	Specific Surface Area
EC	Electrical conductivity
FTIR	Fourier transform infrared spectroscopy
TC	Total carbon
ICP-OES	Inductively coupled plasma—optical emission spectrometry
BET	Brunauer–Emmett–Teller method
SEM	Scanning Electron Microscope
ANOVA	Analysis of variance
N	Nitrogen
P	Phosphorus
K	Potassium
Ca	Calcium
S	Sulfur
Mg	Magnesium
Na	Sodium
As	Arsenic
Fe	Iron
Ni	Nickel
Zn	Zinc
Cu	Copper
Mo	Molybdenum
Pb	Lead
Se	Selenium
Mn	Manganese

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