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Effect of Ash from *Salix viminalis* on the Biomass and Heating Value of *Zea mays* and on the Biochemical and Physicochemical Properties of Soils

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Abstract: Wood ash is sometimes used as an alternative to mineral fertilizers; however, there is still a paucity of reliable data concerning its effect on plants—and on biological properties of soil. The present study aimed to determine the possible extent of soil pollution with ash from *Salix viminalis* that does not disturb the growth of *Zea mays* L., intended for energetic purposes, in order to identify how the increasing ash doses affect biochemical and physicochemical properties of soil and to finally to establish the neutralizing effects of soil additives, i.e., compost and HumiAgra preparation, on this soil pollutant. The study demonstrated that the heating value of *Zea mays* L. was stable and not modified by the excess content of ash from *Salix viminalis* in the soil. This finding points to the feasibility of *Zea mays* L. cultivation on soils contaminated with ash from *Salix viminalis* and its use in bio-power engineering. The biomass of the aboveground parts of *Zea mays* L. was significantly reduced after soil contamination with *Salix viminalis* ash dose of 20 g kg^{-1} d.m. soil, whereas the smaller ash doses tested (5–10 g kg^{-1} d.m. soil) did not impair either the growth or the development of *Zea mays* L. The ash inhibited activities of all analyzed soil enzymes but increased soil pH and sorption capacity. Fertilization with compost proved more effective in neutralizing the adverse effect of ash on enzymatic activity of the soil.

Keywords: ash; soil; plant; soil enzymes; heat of combustion; heating value

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1. Introduction

Zea mays L. belongs to the *Poaceae* family. Due to its high yield potential and nutritional value, it is cultivated in many regions across the world for food and feed production purposes [1,2]. It is also commonly used as a main energy crop for biogas production. In a major part of Europe, it is treated as a "green energy" for biogas plants [3,4]. In addition, it may be grown for energetic purposes [5] on marginal soil and under stress-triggering conditions [6]. These stress conditions may be induced by excessive soil fertilization with wood ash obtained from, e.g., osier (*Salix viminalis*). *S. viminalis* is a fast-growing species with few soil requirements [7]. It may be cultivated in areas of low agricultural productivity and wastelands [5]. Finally, osier (*Salix viminalis*) has been reported to ensure the best performance among the energy crops [8] and has also been found effective in soil remediation [9].

Wood ash is a product of the combustion of wood materials containing organic and inorganic compounds [10,11]. Ash from biomass has strongly alkaline pH values (pH > 12.5) and is enriched with specified forms of minerals (like quartz and calcite) and nutrients (P, K, Ca, Mg) [12–15]. It contains oxides, hydroxides, carbonates, and silicates but is poor in nitrogen, which volatilizes during combustion [16]. In addition, its fine-grained (powdered) structure (some fractions are even smaller than <1 µm) ensures a very large reactive surface capable of interacting with heavy metals (cadmium, zinc, copper) [14].

Furthermore, ash exhibits various properties, depending on the type and origin of combusted biomass [17–19]. In general, wood-based ashes (the so-called type C ashes) have higher contents of calcium, magnesium, and manganese and higher pH values than those from annual plants (the so-called type K ashes) enriched in potassium, phosphorus, sulfur, and chlorine [20]. Ashes from wood biomass may differ significantly in terms of elemental composition (heavy metals) [18] and content of organic compounds (e.g., PAHs) [21,22]. Apart from essential nutrients, like phosphorus, potassium, calcium, and magnesium, which play an important physiological role in the synthesis of chlorophyll; nucleotides; phosphatides; alkaloids; and multiple enzymes, hormones, and vitamins [23], ash contains certain toxic substances, including organic compounds (chlorobenzenes, PAHs, and chlorophenols), as well as radioactive (^{137}Cs) and toxic (arsenic, cobalt, copper, nickel, lead, zinc) elements, which adversely affect the natural environment [24] and may be toxic to plants [25,26]. Due to its high toxicity and mobility, cadmium has been claimed the most hazardous metal in wood ash [27,28]. Therefore, soil amendment with high doses of ash may disturb nutrient cycling, which regulates organic matter content, and by this means may adversely affect plant growth and development [29]. Given these vast differences in ashes, great caution should be exercised when applying them as soil quality promoters, especially in agrosystems, because—under specified conditions—they may pose a threat to the natural environment (e.g., elution of heavy metals) [18,20] or may exert adverse health effects (e.g., increased uptake of heavy metals from soil by plants) [21,22].

The effects of applying wood-based ashes resemble those of calcium-based fertilizers, namely, they both regulate soil pH, but the ash offers an additional advantage, as it provides nutrients. However, both the wood-based ash and calcium fertilizers are strongly alkaline and may cause damage to crops when applied in excess doses [30]. The use of ash in crop cultivation may elicit both positive and negative outcomes. A study conducted by Liu et al. [1] demonstrated its positive effect when applied to soil in doses of 20 and 40 g kg^{-1} d.m. soil on the growth of *Zea mays* L. and the enzymatic activity of lead-polluted soil. Romdhane et al. [31] also demonstrated its positive impact (26 g kg^{-1} d.m. soil) on shoot growth and leaf number in two analyzed hybrids of *Zea mays* L. In turn, Pukalchik et al. [32] showed that an ash dose of 44.1 g kg^{-1} d.m. soil negatively affected the enzymatic activity of soil. The positive effects of ash may be ascribed to the greater availability of its P and K, whereas its adverse effects may be due to the reduced N content [13]. Given the high pH and chemical composition of wood-based ash, its application in agriculture has been studied for years [33,34]. Its deposition and irrational use in agriculture may result in environmental contamination [35,36].

In view of the above, an innovation of the present study was the application of ash from *Salix viminalis* aimed to establish the doses eliciting positive effects on plants as well as enzymatic activity and physicochemical properties of soil, and also to determine the doses causing soil contamination. In addition, compost and HumiAgra were applied to determine their efficacy in neutralizing the potential adverse effect of ash from *Salix viminalis*.

Taking into account the above data, the following research hypotheses were formulated: (a) up to a certain level (dose), ash from *Salix viminalis* exerts a positive effect on the biomass of aboveground parts and roots, heat of combustion, and heating value of *Zea mays* L. as well as on the activities of soil enzymes and physicochemical properties of soil; (b) once a certain level (dose) of osier ash is exceeded, it becomes a contaminant and elicits adverse effects on plants as well as enzymatic activity and physicochemical properties of soil; and (c) the adverse effects of high doses of ash on *Zea mays* L. biomass and activity of soil enzymes may be neutralized by soil amendment with compost and HumiAgra.

The major goal of this study was to evaluate the effect of ash from *Salix viminalis* on the growth and development of *Zea mays* L. and its heating value. Additional study aims were to determine the impact of increasing ash doses on the biochemical and physicochemical properties of soil and to establish the usability of soil fertilization with compost and HumiAgra preparation on the neutralization of the adverse effects of ash.

2. Materials and Methods

2.1. Characteristics of Soil and Composition of Ash from Salix viminalis, Compost, and Humic Acids

The soil used to establish the experiment was collected form the arable-humus horizon (0–20 cm) of soil in Tomaszkowo village, Olsztyn commune, Warmia and Mazury Province, Poland (53.7161° N, 20.4167° E). The soil was air-dried and sieved through a screen with 0.5 cm mesh diameter. Then, it was assessed for its fraction size composition and basic chemical and physicochemical properties. Soil properties and the design of the vegetation pot experiment are presented in Table 1.

Table 1. Design of the experiment.

Type of Analysis	Pot Vegetation Experiment
Soil characteristics	Sandy clay: sand 69.41%, silt 27.71%, clay 2.88%; Contents: organic carbon—6.18 g kg^{-1} d.m. soil, total nitrogen—1.27 g kg^{-1} d.m. soil; pH_{KCl}—6.09; Hydrolytic acidity (HAC)—8.81 mmol(+) kg^{-1} d.m. soil; Sum of exchangeable base cations (EBC)—24.00 mmol(+) kg^{-1} d.m. soil; Cation exchange capacity (CEC)—32.81 mmol(+) kg^{-1} d.m. soil; Extent of saturation with base cations (BS)—73.14%.
The mass of soil in the pot	3.5 kg
Experimental plant	*Zea mays* L. variety LG 32.58 (4 plants in one pot)
Fertilization in mg kg^{-1} d.m. of soil	N—150 in the form of $CO(NH_2)_2$ P—70 in the form of KH_2PO_4 K—120 in the form of KCl + KH_2PO_4 Mg—15 in the form of $MgSO_4 \times 7H_2O$
Dose of ash from *Salix viminalis* in g kg^{-1} d.m. of soil	0, 5, 10, 20
The content of elements in the ash (Figure 1a) in % d.m.	C—51.04, H—5.87, S—0.02, N—0.38; Cl—0.02; P—0.09; K—0.17; Mg—0.03; Ca—0.41; Na—0.009
pH in KCl	12.50
Compost	Manufacturer: Ekokonsorcjum-Effekt company (Cracow, Poland; license no. 21/02) from green waste (grass, bushes) gathered in parks, squares, and home gardens; fruit and vegetables picked at market squares; and organic waste from food processing plants. Chemical composition in % of d.m.: C_{org}—23.20; N_{Total}—1.30; P—0.26; K—1.24; Mg—0.30; and Ca—1.43.
HumiAgra	Manufacturer: AgraPlant, Kielce, Poland. Chemical composition in % of d.m.: 90% humic acids (50% humins and 50% fulvic acids), 6% K, and 3% S.
Dose of compost and HumiAgra in g kg^{-1} d.m. of soil	0 and 2.5
Experiment duration in days	60
Number of replications	Four repetitions per combination
Conditions in the vegetation hall	The average temperature was 17.5 °C, air humidity reached 78.5%, and day length was from 13 h 4 min to 15 h 30 min. The soil moisture content—50% of the water capillary capacity.

Investigations conducted by Romdhane et al. [31], Błońska et al. [36], Mundała et al. [37], and Núñez-Delgado et al. [38] have demonstrated that the content of cobalt in wood ash ranged from 3.37 to 5.00 mg kg^{-1}, that of chromium from 12.70 to 28.0 mg kg^{-1}, that of copper from 82.50 to 129 mg kg^{-1}, that of nickel from 5.28 to 27.30 mg kg^{-1}, that of lead from 23.30 to 527.00 mg kg^{-1}, that of zinc from 281.60 to 732 mg kg^{-1}, and that of cadmium from 0.22 to 0.55 mg kg^{-1}. According to Someshwar [39], the content of organic substances, including biphenyl, naphthalene, and phenanthrene, in wood ash is negligible and does not pose threat to the natural environment.

2.2. Study Design

Zea mays L. (Figure 1b) was harvested on day 60 of the experiment (at the BBCH 39 stage) and assessed for the biomass yield of aboveground parts and roots. The aboveground parts of *Zea mays* L. were also analyzed for the heat of combustion and heating value. The leaf greenness index of *Zea mays* L. was evaluated three times throughout the experimental period. In turn, selected soil samples were analyzed for activities of soil enzymes: dehydrogenases, catalase, urease, acid phosphatase, alkaline phosphatase, β-glucosidase, and arylsulfatase. In addition, after crop harvest, the soil samples were analyzed for the following chemical properties: contents of organic carbon (C_{org}) and total nitrogen, and for the following physicochemical properties: pH, hydrolytic acidity (HAC), sum of exchangeable base cations (EBC), total cation exchange capacity (CEC), and extent of saturation with base cations (BS).

(**a**) (**b**)

Figure 1. (**a**) Ash from *Salix viminalis.* (**b**) View of the experiment with *Zea mays* L. in the vegetation hall.

2.3. Biochemical, Chemical, and Physicochemical Analyses of Soil

The experiment included determinations of the activity of soil enzymes as well as basic chemical and physicochemical properties of soil. Analyses of the biochemical soil properties included determinations of activities of: dehydrogenases (Deh) —with Lenhard's method modified by Öhlinger [40]; catalase (Cat)—with Alef and Nannipieri's method [41]; urease (Ure)—with Alef and Nannipieri's method [41]; acid phosphatase (Pac) and alkaline phosphatase (Pal)—with Alef and Nannipieri's method [41]; arylsulfatase (Aryl)—with Alef and Nannipieri's method [41], and β-glucosidase (Glu)—with Alef and Nannipieri's method [41]. Analyses of the chemical properties of soil included determinations of the contents of organic carbon (C_{org}) and total nitrogen (N_{Total}) by means of the Vario MaxCube CN elemental microanalyzer (Hanau, Germany). In turn, the physicochemical soil properties analyzed included: pH—with the potentiometric method in a 1 mol·dm^{-3} aqueous KCl solution, hydrolytic acidity (HAC), and sum of exchangeable base cations (EBC) with the Kappen's method [42]. The HAC and EBC values obtained were used to determine the total cation exchange capacity of soil (CEC) and the extent of its saturation with base cations (BS).

The heating value of *Zea mays* L. was estimated with the combustion method in a C-2000 calorimeter (IKA WERKE, Northchase Pkwy Se, Wilmington, USA). The heat of combustion (Q) was determined acc. to the Polish standard PN-EN ISO 18125:2017 IKA C2000 [43], and the heating value acc. to Kopetz et al. [44]. Contents of carbon, hydrogen, and sulfur were determined by means of an ELTRA CHS 500 automatic analyzer (Neuss, Germany), following PN-G-04584 and PN-G-04517 standard methods [45]. Nitrogen content was determined with the Kjeldahl method (ISO 11261) [46]. Contents of P, K, Mg, and Ca levels in *Salix viminalis* wood ash were determined using inductively coupled plasma (ICP) spectroscopy (ICP—OES ThermoiCAP 6500 DUO, ThermoFisher Scientific, Cambridge, UK), after sample mineralization in a mixture of concentrated nitrogen and perchloric acid. The leaf greenness index was determined using a Spectrum Technologies, Inc., Chlorophyll Meter (KONICA MINOLTA, Inc., Chiyoda, Japan).

Biochemical, chemical, and physicochemical analyses were conducted in three replications. A detailed procedure for enzymatic activity determination was provided in a work by Zaborowska et al. [47], whereas the procedures of determinations of chemical and physicochemical parameters were provided in our previous works [48,49]. In turn, the heating value of *Zea mays* L. was described in the study by Wyszkowska et al. [50].

2.4. Computations and Statistical Analysis

One of the computations included calculating the index of the *Salix viminalis* ash effect on activities of soil enzymes. A detailed description of its concept was provided in our previous works [51,52]. Using analysis of variance (ANOVA), the obtained data were statistically analyzed at the significance level of $p \leq 0.05$ using the STATISTICA 13 program [53]. Homogenous groups were computed using the Tukey's test for the following variables: biomass yield of the aboveground parts and roots of *Zea mays* L., heat of combustion, and heating value. Coefficients of the linear Pearson's correlation between the variables were computed, and the coefficient of percentage variability of all analyzed variables (η^2) was calculated by means of the analysis of variance (ANOVA). In turn, the principal component analysis (PCA) was applied to analyze activities of soil enzymes.

3. Results

3.1. Biomass and Heating Value of Zea mays L. Cultivated in Soil with the Addition of Ash from Salix viminalis

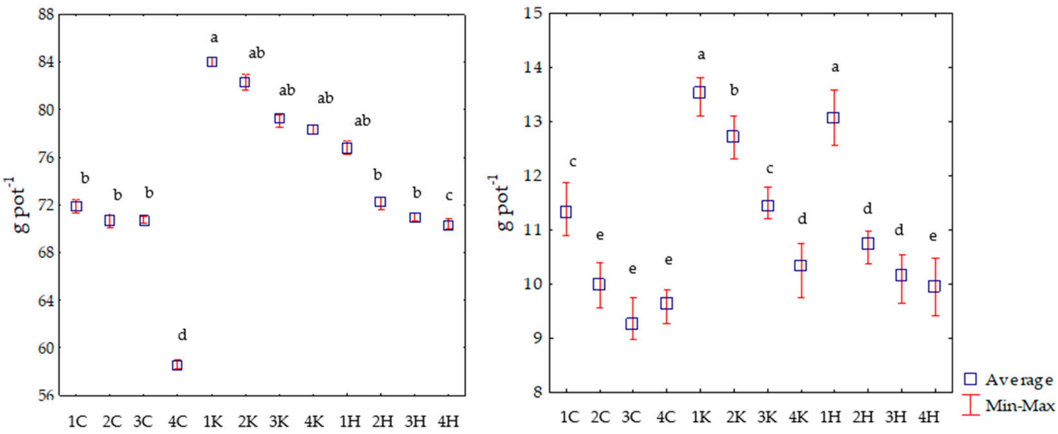
The percentage distribution of variable factors revealed the strongest effect (50.51%) of *Salix viminalis* ash dose on the yield of *Zea mays* L. roots. In turn, the aboveground parts of the experimental plant were most strongly affected (as much as 54.83%) by soil amendment with compost and HumiAgra (Table 2).

Table 2. Coefficient of observed variation η^2 (%).

Variable Factors	Plant Yield *		Enzymes **						
	AP	R	Deh	Cat	Ure	Pac	Pal	Glu	Aryl
Ash dose	26.23	50.51	93.47	80.84	87.01	95.64	68.42	92.92	57.13
Additives	54.83	31.47	2.64	15.85	5.00	3.15	21.99	4.69	36.22
Ash * Additives	7.95	5.50	1.07	2.72	7.89	0.93	6.04	2.19	6.55
Error	10.97	12.51	2.83	0.59	0.10	0.28	3.55	0.20	0.10

* AP—aboveground parts; R—roots; ** Deh—dehydrogenases; Cat—catalase; Ure—urease; Pac—acid phosphatase; Pal—alkaline phosphatase; Glu—β-glucosidase; Aryl—arylsulfatase.

A significant decrease in the biomass of aboveground parts of *Zea mays* L. was noted after soil treatment with a *Salix viminalis* ash dose of 20 g kg^{-1} d.m. soil (Figure 2a,b). Smaller ash doses tested (5–10 g kg^{-1} d.m. soil) did not impair *Zea mays* L. yield. In turn, its root biomass was significantly decreased by all analyzed doses of ash, i.e., 5–20 g kg^{-1} d.m. soil. The fertilization of control soil (without ash addition) with compost stimulated the growth and development of both aboveground parts and roots of *Zea mays* L., whereas soil amendment with HumiAgra showed a promoting effect on roots only. Both organic substances partly neutralized the adverse effect of the 20 g per kg soil ash dose on *Zea mays* L.



(**a**) (**b**)

Figure 2. Dry weight of aboveground parts (**a**) and roots (**b**) of *Zea mays* from soil contaminated with *Salix viminalis* ash in g pot^{-1}. Explanations: 1–4 ash dose g kg^{-1} d.m. soil: 1—0; 2—5; 3—10; 4—20; C—soil without compost and HumiAgra, K—soil with compost, H—soil with HumiAgra. Homogeneous groups (a–e) were created separately for aboveground parts and roots.

The application of ash, compost, both, and HumiAgra to the soil had no significant effect on the leaf greenness index (SPAD) of *Zea mays* L. (Table 3). Its values decreased significantly along with *Zea mays* L. age, i.e., the highest value was recorded on day 14 of crop vegetation and the lowest on day 42. These correlations were determined in the non-fertilized soil and the soil fertilized with compost and HumiAgra.

Table 3. The effect of ash from *Salix viminalis* on the leaf greenness index (SPAD) of *Zea mays* L.

Ash Dose, g kg^{-1} d.m. Soil	14 Days	28 Days	42 Days
Without Additives			
0	43.02 ^{a} ± 1.73	36.71 ^{bcd} ± 2.47	26.03 ^{f} ± 2.23
5	42.37 ^{a} ± 1.47	35.12 ^{cde} ± 1.02	25.37 ^{f} ± 2.17
10	41.23 ^{ab} ± 0.60	35.07 ^{cde} ± 0.69	25.21 ^{f} ± 1.58
20	40.55 ^{ab} ± 0.40	31.43 ^{e} ± 2.56	24.78 ^{f} ± 2.08
$\overline{X}$	41.80 ^{B}	34.58 ^{D}	25.35 ^{F}
r	−0.96 *	−0.97 *	−0.96 *
Compost			
0	43.55 ^{a} ± 2.14	34.49 ^{cde} ± 1.98	24.58 ^{f} ± 3.58
5	43.43 ^{a} ± 1.57	34.07 ^{cde} ± 2.30	24.13 ^{f} ± 1.02
10	42.68 ^{a} ± 1.02	33.58 ^{de} ± 2.43	22.90 ^{f} ± 1.95
20	42.14 ^{a} ± 0.33	33.59 ^{de} ± 1.92	22.69 ^{f} ± 0.75
$\overline{X}$	42.93 ^{A}	33.950 ^{E}	23.58 ^{E}
r	−0.98 *	−0.87 *	−0.97 *
HumiAgra			
0	43.670 ^{a} ± 1.07	38.95 ^{abc} ± 0.63	26.31 ^{f} ± 0.91
5	43.07 ^{a} ± 1.08	35.56 ^{cde} ± 0.97	26.25 ^{f} ± 1.51
10	42.07 ^{a} ± 0.97	34.73 ^{cde} ± 1.56	25.99 ^{f} ± 0.61
20	42.14 ^{a} ± 1.29	34.55 ^{cde} ± 2.11	25.69 ^{f} ± 1.03
$\overline{X}$	42.75 ^{A}	35.95 ^{C}	26.05 ^{F}
r	−0.88 *	−0.80	−1.00 *

* r—correlation coefficient significant at $p = 0.05$; Homogeneous groups ($^{a–f}$) for three terms of the leaf greenness index assessment; homogeneous groups for means were calculated for three terms of the leaf greenness index assessment ($^{A–F}$).

The heat of combustion of *Zea mays* L. ranged from 17.86 MJ kg^{-1} p.dm. in the plants grown on the soil polluted with ash from *Salix viminalis* at a dose of 20 g kg^{-1} d.m. soil in the experimental series with HumiAgra to 18.42 MJ kg^{-1} p.dm. in the plants from the control pot without additives (Table 4), whereas the heating value ranged from 15.93 in the case of plants grown on the soil polluted with ash from *Salix viminalis* to 16.500 MJ kg^{-1} p.dm. in the case grown on the non-polluted soil. The energy obtained from *Zea mays* L. biomass produced from 1 kg of soil amended with compost and HumiAgra was higher than the variants without these additives. These correlations were observed in both the control soil and soil polluted with *Salix viminalis* ash.

Table 4. The effect of ash from *Salix viminalis* on the heat of combustion and heating value of *Zea mays* L.

Ash Dose, g kg^{-1} d.m. Soil	Heat of Combustion	Heating Value	Energy Production MJ kg^{-1}
	MJ kg^{-1} Air-Dried Plant Matter		
Without Additives			
0	18.42 ^{a} ± 0.04	16.50 ^{a} ± 0.03	0.340 ^{b} ± 0.02
20	17.91 ^{c} ± 0.03	15.93 ^{c} ± 0.02	0.27 ^{c} ± 0.02
Compost			
0	18.29 ^{b} ± 0.02	16.44 ^{a} ± 0.03	0.40 ^{a} ± 0.03
20	17.94 ^{c} ± 0.02	16.13 ^{b} ± 0.03	0.36 ^{ab} ± 0.02
HumiAgra			
0	17.90 ^{c} ± 0.05	16.17 ^{b} ± 0.02	0.36 ^{ab} ± 0.04
20	17.86 ^{c} ± 0.02	16.10 ^{b} ± 0.03	0.32 ^{b} ± 0.03

Homogeneous groups ($^{a-c}$) were created separately for columns.

3.2. Biochemical and Physicochemical Properties of Soil

Activities of all soil enzymes were affected to a greater extent (from 57.131% for arylsulfatase to 95.643% for acid phosphatase) by soil treatment with *Salix viminalis* ash than by its fertilization with compost and HumiAgra (from 2.635% for dehydrogenases to 36.220% for arylsulfatase) (Table 2). The results of PCA enabled illustration of the effect of ash from *Salix viminalis* on the soil enzymes (Figure 3). The figure presents the distribution of the analyzed samples in the system of two principal components. The activity of dehydrogenases, catalase, urease, acid phosphatase, alkaline phosphatase, β-glucosidase, and arylsulfatase was negatively correlated with the first principal component explaining 89.24% of the analyzed variability. The *Salix viminalis* ash doses of 5, 10, and 20 g kg^{-1} d.m. soil inhibited activities of the soil enzymes, as indicated by the distribution of cases on the plane. The reciprocal arrangement of vectors describing urease, β-glucosidase, acid phosphatase, dehydrogenases, and catalase indicates their similar response to the soil amendment with *Salix viminalis* ash. In turn, arylsulfatase and alkaline phosphatase formed one group; however, they were more sensitive to the negative effect of the ash. In addition, the distribution of cases in relation to vectors enables concluding that compost was more effective in mitigating the adverse effects of ash from *Salix viminalis* on the biochemical activity of soil than HumiAgra.

The inhibiting effect of ash on the soil enzymes was reflected in the values of the index of the *Salix viminalis* ash effect (IF_{Ash}) on the biochemical activity of the soil. Considering the IF_{Ash} values, the analyzed enzymes were ordered as follows (from the most to the least sensitive to *Salix viminalis* ash): Ure > Pac > Glu > Deh > Pal > Cat > Aryl (Table 5).

The adverse effects of *Salix viminalis* ash were noticed even in the soil samples polluted with its lowest dose (5 g kg^{-1} d.m.) and aggravated along with increasing ash doses, regardless of soil amendment with compost and HumiAgra.

The effect of soil treatment with the organic substances is well reflected in the values of the indices of compost influence (IF_K) and HumiAgra influence (IF_H) on the activities of soil enzymes (Figure 4a,b).

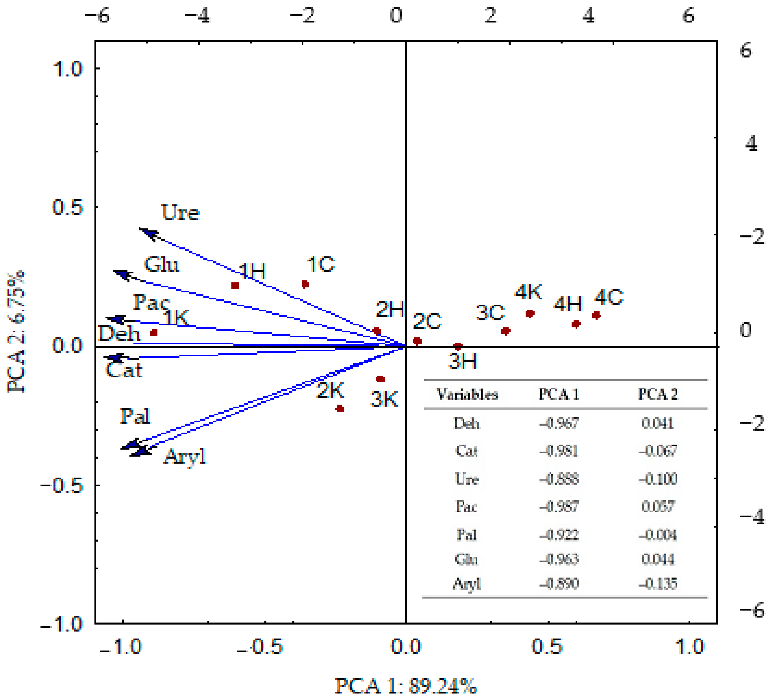
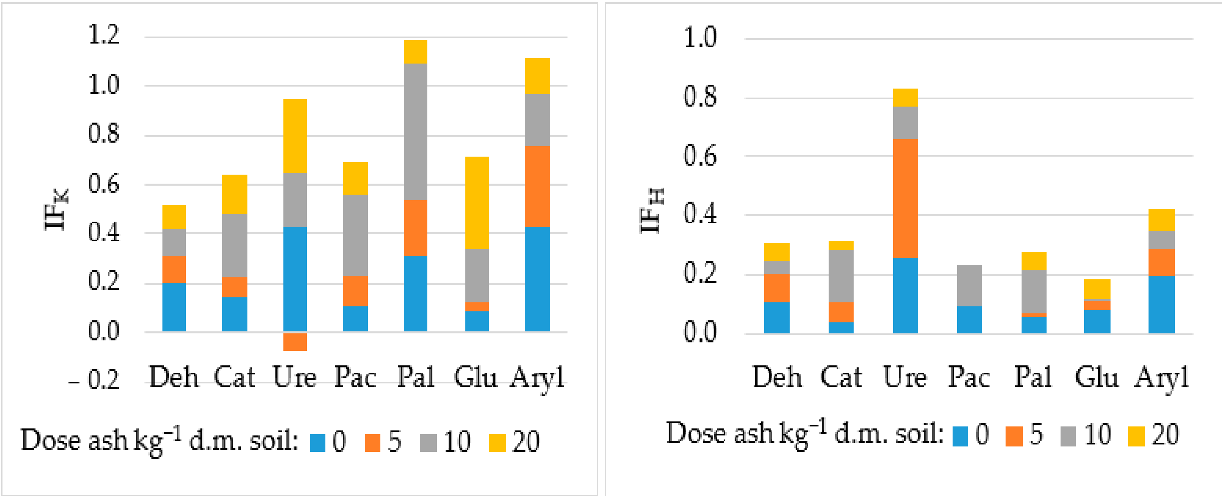


Figure 3. Enzyme activity in soil contaminated with *Salix viminalis* ash presented based on PCA. Explanations: 1–4 ash dose g kg^{-1} d.m. soil: 1—0; 2—5; 3—10; 4—20; C—soil without compost and HumiAgra, K—soil with compost, H—soil with HumiAgra; enzyme abbreviations are provided under Table 2.

Table 5. Index of the effect of *Salix viminalis* ash on the activity of soil enzymes.

Ash Dose, g kg^{-1} d.m. Soil	Deh	Cat	Ure	Pac	Pal	Glu	Aryl
				Without Additives			
5	−0.23 ^{a} ± 0.03	−0.111 ^{a} ± 0.03	−0.42 ^{b} ± 0.07	−0.25 ^{a} ± 0.03	−0.04 ^{a} ± 0.01	−0.26 ^{a} ± 0.03	−0.030 ^{a} ± 0.03
10	−0.26 ^{a} ± 0.04	−0.254 ^{d} ± 0.02	−0.51 ^{c} ± 0.05	−0.47 ^{c} ± 0.07	−0.30 ^{e} ± 0.02	−0.40 ^{d} ± 0.02	−0.06 ^{a} ± 0.01
20	−0.61 ^{c} ± 0.05	−0.31 ^{ef} ± 0.02	−0.56 ^{d} ± 0.02	−0.60 ^{d} ± 0.05	−0.39 ^{f} ± 0.04	−0.53 ^{f} ± 0.04	−0.23 ^{e} ± 0.02
$\overline{X}$	−0.37 ^{A}	−0.23 ^{B}	−0.50 ^{B}	−0.44 ^{B}	−0.24 ^{B}	−0.40 ^{B}	−0.11 ^{A}
				Compost			
5	−0.29 ^{b} ± 0.08	−0.15 ^{b} ± 0.01	−0.62 ^{f} ± 0.08	−0.23 ^{a} ± 0.02	−0.10 ^{b} ± 0.02	−0.29 ^{ab} ± 0.02	−0.10 ^{b} ± 0.01
10	−0.31 ^{b} ± 0.07	−0.18 ^{c} ± 0.03	−0.57 ^{d} ± 0.04	−0.36 ^{b} ± 0.03	−0.17 ^{c} ± 0.02	−0.33 ^{c} ± 0.02	−0.20 ^{d} ± 0.01
20	−0.65 ^{c} ± 0.05	−0.30 ^{de} ± 0.06	−0.60 ^{e} ± 0.03	−0.59 ^{d} ± 0.06	−0.49 ^{g} ± 0.04	−0.41 ^{d} ± 0.05	−0.38 ^{g} ± 0.04
$\overline{X}$	−0.42 ^{B}	−0.21 ^{B}	−0.60 ^{C}	−0.40 ^{A}	−0.25 ^{B}	−0.34 ^{A}	−0.23 ^{C}
				HumiAgra			
5	−0.24 ^{a} ± 0.06	−0.09 ^{a} ± 0.05	−0.35 ^{a} ± 0.03	−0.31 ^{b} ± 0.01	−0.08 ^{b} ± 0.05	−0.30 ^{ab} ± 0.04	−0.12 ^{bc} ± 0.01
10	−0.30 ^{b} ± 0.04	−0.16 ^{b} ± 0.02	−0.56 ^{d} ± 0.02	−0.45 ^{c} ± 0.03	−0.24 ^{d} ± 0.02	−0.44 ^{e} ± 0.02	−0.17 ^{c} ± 0.02
20	−0.63 ^{c} ± 0.09	−0.31 ^{f} ± 0.03	−0.63 ^{f} ± 0.06	−0.64 ^{e} ± 0.06	−0.39 ^{f} ± 0.03	−0.54 ^{f} ± 0.01	−0.31 ^{f} ± 0.03
$\overline{X}$	−0.39 ^{A}	−0.19 ^{A}	−0.39 ^{A}	−0.35 ^{A}	−0.18 ^{A}	−0.32 ^{A}	−0.20 ^{B}

Explanations in the Table 2. Homogeneous groups ($^{a–g}$) were created separately for each enzyme; homogeneous groups for means were calculated for each enzyme ($^{A–C}$).



(**a**) (**b**)

Figure 4. Index of the effect of compost [IF_K] (**a**) and HumiAgra [IF_H] (**b**) on the activity of soil enzymes. Explanations as in Table 2.

The IF_K and IF_H values computed for the ash-polluted soil samples pointed to the positive effects of both substances on the activities of dehydrogenases, urease, and arylsulfatase—and, in the case of HumiAgra, also on the activity of β-glucosidase. In turn, soil treatment with ash from *Salix viminalis* contributed to decreased IF_K and IF_H values, indicating a minor pollution-mitigating effect of these organic substances. The lowest value of the IF_K index was noted in the case of urease, which turned out to be the most sensitive enzyme to *Salix viminalis* ash application even at the smallest tested dose, i.e., 5 g kg^{-1} d.m. soil. In the case of the IF_H index, its lowest value was determined for acid phosphatase in the soil polluted with the highest ash dose (20 g kg^{-1} d.m. soil). A comparison of IF_K and IF_H values demonstrated that HumiAgra proved less effective in mitigating the adverse changes caused by the *Salix viminalis* ash than compost.

Soil pollution with an ash dose of 20 g kg^{-1} d.m. soil caused a significant increase in C_{org} content, which was not observed upon soil amendment with the lower ash doses. The highest content of organic carbon was determined in the soil samples fertilized with compost, whereas the values found in the control soil sample and in the samples treated with HumiAgra were similar. The highest tested dose of ash from *Salix viminalis* (20 g kg^{-1} d.m. soil) also increased the total nitrogen content of the soils without and with the addition of compost and HumiAgra. In addition, the N_{Total} content of the soil was significantly affected by the ash dose of 10 g kg^{-1} d.m. soil in the variants with compost addition. Regardless of compost and HumiAgra addition to the soil, its pH increased and its hydrolytic acidity decreased under the influence of *Salix viminalis* ash (Table 6).

Values of these two parameters were correlated with the sum of exchangeable base cations, which was observed to increase upon soil pollution with the ash. Similar observations were made for CEC and BS, whose values increased along with increasing ash doses. The addition of compost and HumiAgra to the soil positively affected its physicochemical properties.

Table 6. The effect of ash from *Salix viminalis* on the chemical and physicochemical properties of soil.

Ash Dose, g kg^{-1} d.m. Soil	Total Organic Carbon (C_{org})	Total Nitrogen (N_{Total})	pH_{KCl}	Hydrolytic Acidity (HAC)	Total Exchangeable Base Cations (EBC)	Total Cation Exchange Capacity of Soil (CEC)	Base Cations Saturation Ratio in Soil (BS)
	g kg^{-1}			mmol$^{(+)}$ kg^{-1} Soil			%
Without Additives							
0	7.87 ^{g} ± 0.18	1.68 ^{d} ± 0.01	4.35 ^{f} ± 0.05	15.71 ^{c} ± 0.04	44.10 ^{k} ± 0.05	59.81 ^{j} ± 0.26	73.73 ^{i} ± 0.31
5	8.10 ^{fg} ± 0.09	1.71 ^{cd} ± 0.02	6.85 ^{d} ± 0.04	5.81 ^{d} ± 0.04	122.00 ^{h} ± 1.00	127.81 ^{g} ± 0.04	95.45 ^{f} ± 0.01
10	8.27 ^{fg} ± 0.15	1.71 ^{cd} ± 0.01	7.00 ^{c} ± 0.03	3.71 ^{f} ± 0.04	123.00 ^{h} ± 0.10	126.71 ^{g} ± 1.04	97.07 ^{d} ± 0.01
20	10.34 ^{b} ± 0.11	1.73 ^{bc} ± 0.02	7.45 ^{b} ± 0.04	2.74 ^{h} ± 0.04	201.10 ^{e} ± 0.50	203.84 ^{e} ± 0.54	98.66 ^{a} ± 0.03
$\overline{X}$	8.64 ^{B}	1.708 ^{A}	6.41 ^{A}	6.99 ^{B}	122.55 ^{C}	129.54 ^{C}	91.23 ^{B}
r	0.94 *	0.923 *	0.80 *	−0.81 *	0.96 *	0.96 *	0.76
Compost							
0	8.57 ^{ef} ± 0.10	1.72 ^{bc} ± 0.02	4.50 ^{e} ± 0.04	17.14 ^{a} ± 0.11	107.00 ^{i} ± 0.05	124.14 ^{h} ± 1.11	86.20 ^{g} ± 0.18
5	9.19 ^{cd} ± 0.13	1.74 ^{bc} ± 0.01	7.05 ^{c} ± 0.05	6.04 ^{d} ± 0.04	197.50 ^{f} ± 0.50	203.54 ^{e} ± 0.14	97.03 ^{d} ± 0.03
10	9.48 ^{c} ± 0.19	1.81 ^{a} ± 0.02	7.50 ^{b} ± 0.03	4.35 ^{e} ± 0.08	229.00 ^{a} ± 1.00	233.35 ^{a} ± 0.92	98.14 ^{c} ± 0.01
20	10.99 ^{a} ± 0.11	1.82 ^{a} ± 0.01	7.80 ^{a} ± 0.03	3.79 ^{f} ± 0.04	220.00 ^{b} ± 0.05	223.79 ^{b} ± 0.04	98.31 ^{bc} ± 0.01
$\overline{X}$	9.56 ^{A}	1.77 ^{A}	6.71 ^{A}	7.83 ^{A}	188.38 ^{A}	196.20 ^{A}	94.92 ^{A}
r	0.99 *	0.91 *	0.81 *	−0.77	0.76	0.76	0.74
HumiAgra							
0	8.06 ^{g} ± 0.08	1.71 ^{cd} ± 0.01	4.45 ^{ef} ± 0.05	16.43 ^{b} ± 0.07	50.30 ^{j} ± 0.05	66.73 ^{i} ± 0.23	75.38 ^{h} ± 0.03
5	8.16 ^{fg} ± 0.13	1.73 ^{bc} ± 0.03	6.85 ^{d} ± 0.05	6.00 ^{d} ± 0.07	136.50 ^{g} ± 0.30	142.50 ^{f} ± 0.38	95.79 ^{e} ± 0.02
10	8.82 ^{de} ± 0.08	1.76 ^{b} ± 0.01	7.05 ^{c} ± 0.05	3.98 ^{f} ± 0.07	204.00 ^{d} ± 0.30	207.985 ^{d} ± 0.08	98.09 ^{c} ± 0.04
20	10.34 ^{b} ± 0.09	1.81 ^{a} ± 0.02	7.55 ^{b} ± 0.05	3.26 ^{g} ± 0.08	215.00 ^{c} ± 0.30	218.26 ^{c} ± 1.26	98.51 ^{ab} ± 0.11
$\overline{X}$	8.85 ^{B}	1.75 ^{A}	6.48 ^{A}	7.42 ^{A}	151.45 ^{B}	158.87 ^{B}	91.94 ^{B}
r	0.97 *	1.00 *	0.82 *	−0.80	0.89 *	0.81 *	0.75

* r—correlation coefficient significant at p = 0.05; homogeneous groups ($^{a–k}$) were created separately for each parameter; homogeneous groups for means were calculated for each parameter ($^{A–C}$).

3.3. Correlations between the Analyzed Parameters

The biomass yield of aboveground parts and roots of *Zea mays* L. (Table 7) was positively correlated with the activities of the analyzed soil enzymes and hydrolytic acidity (HAC) of the soil but negatively correlated with soil pH, sum of EBC, CEC, soil saturation with base cations (BS), and content of organic carbon (C_{org}). Activities of all analyzed enzymes were positively correlated with each other and with HAC, as well as negatively correlated (likewise *Zea mays* L. biomass) with soil pH, EBC, CEC, BS, and C_{org} content. Enzymatic activity was not significantly correlated with N_{Total} content of the soil, but a positive correlation was found between contents of C_{org} and N_{Total}, as well between their contents and soil pH, EBC, CEC, and BS.

Table 7. Correlation coefficients between variables.

Variable Factors	R	Deh	Cat	Ure	Pac	Pal	Glu	Aryl	C_{org}	N_{Total}	pH	HAC	EBC	CEC	BS
AP	0.66 *	0.92 *	0.91 *	0.67 *	0.80 *	0.90 *	0.85 *	0.81 *	−0.84 *	−0.14	−0.73 *	0.70 *	−0.56 *	−0.55 *	−0.62 *
R	1.00	0.65 *	0.71 *	0.65 *	0.64 *	0.60 *	0.65 *	0.68 *	−0.52 *	−0.03	−0.57 *	0.59 *	−0.40 *	−0.39 *	−0.51 *
Deh		1.00	0.91 *	0.66 *	0.74 *	0.95 *	0.74 *	0.78 *	−0.89 *	−0.15	−0.65 *	0.62 *	−0.53 *	−0.52 *	−0.52 *
Cat			1.00	0.78 *	0.89 *	0.91 *	0.89 *	0.86 *	−0.82 *	−0.10	−0.79 *	0.78 *	−0.56 *	−0.55 *	−0.70 *
Ure				1.00	0.91 *	0.67 *	0.87 *	0.67 *	−0.54 *	−0.15	−0.77 *	0.81 *	−0.55 *	−0.53 *	−0.69 *
Pac					1.00	0.74 *	0.97 *	0.76 *	−0.68 *	−0.21	−0.93 *	0.94 *	−0.72 *	−0.70 *	−0.87 *
Pal						1.00	0.76 *	0.83 *	−0.79 *	0.01	−0.60 *	0.59 *	−0.41 *	−0.40 *	−0.48 *
Glu							1.00	0.79 *	−0.66 *	−0.15	−0.90 *	0.91 *	−0.67 *	−0.64 *	−0.83 *
Aryl								1.00	−0.58 *	0.09	−0.59 *	0.59 *	−0.42 *	−0.41 *	−0.51 *
C_{org}									1.00	0.49 *	0.72 *	−0.64 *	0.65 *	0.65 *	0.60 *
N_{Total}										1.00	0.41 *	−0.33	0.46 *	0.46 *	0.37 *
pH											1.00	−0.99 *	0.84 *	0.82 *	0.98 *
HAC												1.00	−0.81 *	−0.79 *	−0.98 *
EBC													1.00	0.99 *	0.83 *
CEC														1.00	0.81 *

Explanations in the Tables 2 and 5. * r—coefficient of correlation significant at: $p = 0.05$, $n = 36$.

4. Discussion

4.1. Biomass and Heating Value of Zea mays L. Grown in Soil with the Addition of Ash from Salix viminalis

Soil amendment with *Salix viminalis* ash doses of 5 and 10 g kg^{-1} d.m. did not inhibit either the growth or the development of *Zea mays*, whereas soil pollution with the ash dose of 20 g kg^{-1} d.m. significantly impaired them both. The adverse effect of ash applied at the highest tested dose may be due to its high alkalinity (pH_{KCl} = 12.5) and low nitrogen content (N = 0.38%). Varshney et al. [54] and Cruz et al. [33] have also emphasized that high ash doses applied may cause excessive soil salinity, thereby ultimately contributing to worse conditions for plant growth and root development. However, previous investigations conducted by Liu et al. [1] and Romdhane et al. [31] proved that wood-based ash applied in doses from 20 to 40 g kg^{-1} soil might positively affect *Zea mays* L. biomass. Differences observed for ash effects in the present study and experiments of the aforementioned authors are mainly due to the differences in the chemical composition of the analyzed ashes. In turn, Ondrasek et al. [22] showed that the application of wood-based ash intensified chemical sorption in the rhizosphere, contributing to the enhanced immobilization of elements delivered with fertilizers. In addition, soil treatment with ash may strongly affect its texture, aeration, and water-retention capacity, and by this means determine the dynamics of root growth, leading to multiple potential effects on plant growth [25,26].

The analyzed ash from *Salix viminalis* had no negative impact on the heat of combustion or the heating value of *Zea mays* L., whose values did not change under ash doses applied. This finding indicates the possibility of using biomass from the aboveground parts of this plant for energetic purposes when there is a need for ash management. Also, our previous investigations [55,56] addressing the reclamation of soil contaminated with Cd^{2+}, Co^{2+} and Ni^{2+} demonstrated the usability of *Zea mays* L., *Elymus*, and *Festuca rubra* biomass for energetic purposes. Table 8 below presents the most important results regarding the calorific value compared with the results of other authors and presented in the table.

Table 8. Heating values of *Zea mays* obtained in various studies.

Factor Tested in the Soil	Heating Value MJ kg−1 Air-Dried Plant Matter	Reference
Salix viminalis ash content	15.93–16.50	Our research
Maize variety	7.62–10.79	[57]
Corn grain drying process	13.70–14.94	[6]
Pellets from biomass	15.68	[58]
Cr (VI) content	14.60–15.40	[50]
Maize cultivation	17.51	[59]
Ni^{2+}, Co^{2+}, Cd^{2+} content	14.79–14.97	[55]

An added value of the present study is the finding that the soil amendment with an organic substance (compost and HumiAgra) alleviates the adverse effects of *Salix viminalis* ash dose of 20 g kg^{-1} d.m. soil on *Zea mays* L. biomass. Both published data [60] and our previous research [56] indicate that soil fertilization with compost mitigates the negative impact of inorganic compounds on plant yield. Soil amendment with compost and HumiAgra preparation increased the pool of readily available organic compounds. In addition, compost affects soil properties by enriching it with organic matter susceptible to microbiological degradation. Organic matter mineralization activates its nutrients that are essential to plants. It may also regulate sorption properties of the soil by, e.g., reducing retrogradation of phosphates. In turn, the weaker effect of HumiAgra compared to that of compost may be due to a more diversified chemical composition of the latter [61,62].

4.2. Biochemical and Physicochemical Properties of Soil

Enzymatic activity of soil is one of the key factors driving its fertility [1,63–65]. Soil enzymes mediate soil organic matter degradation and catalyze the main metabolic processes of carbon, nitrogen, and phosphorus [66–68]. Microbes secrete intracellular and extracellular enzymes essential for soil nutrient cycling and contribute to soil fertility and health [69–71]. However, works by Pukalchik et al. [32], Smenderovac et al. [72], and Błońska et al. [36] provide conflicting data related to the impact of ash on the biological properties of soil. Smenderovac et al. [72] and Perucci et al. [73] draw attention to the temporary negative impact of ashes on the activity of soil enzymes. In their studies, the activity of alkaline phosphatase and arylsulfatase was inhibited for the first 4 months after the application of wood ash to the soil. This may be due to the greater release of ions into the solution with a higher ash dose. Therefore, in our own research, the negative effect of *Salix viminalis* ash probably increased with the increase in the ash dose. The present study results demonstrated that ash negatively affected the activity of the analyzed soil enzymes. Previous investigations by Pukalchik et al. [32] show also that the application of wood-based ash suppressed the activities of dehydrogenases, acid phosphatase, and β-glucosidase, as well as the activity of fluorescein diacetate (FDA), which pointed to the inhibited microbial activity in the soil. In turn, Perucci et al. [73] and *Smenderovac* et al. [72] demonstrated that the wood-based ash may strongly affect soil texture, aeration, and water retention capacity [74–77], thereby influencing root growth dynamics and consequently leading to plant growth impairment [78,79].

Organic carbon and nitrogen are two of the key elements of soil that inhibit plant growth [1]. In the present study, ash from *Salix viminalis* applied at a dose of 20 g kg^{-1} soil significantly increased the contents of both C_{org} and N_{total} in the soil. Apart from valuable macroelements, the wood-based ash contains heavy metals [12]. Hence, its small doses may meet nutritional demands of plants, but excess doses may exert toxic effects. This was the likely cause of *Zea mays* biomass reduction observed upon soil amendment with the ash dose of 20 g kg^{-1} in the present study. Wood ash has a high density, is porous and fine-grained, and swells in contact with water. These features of ash contribute to the blocking of soil pores, resulting in a modified soil texture and aeration [11,15].

Plant growth and productivity depend primarily on the availability of nutrients and the physicochemical properties of soil, which in turn are determined by the content of exchangeable base cations (EBC) and soil pH [31]. A study conducted by Lucchini et al. [80] demonstrated that wood-based ash improved the physicochemical properties and nutrient availability of soil. The pH value of osier ash used in the present study (pH = 12.5) was similar to that described in previous research [31,35]. The alkaline character of ash analyzed in the present study caused soil pH to increase significantly. The above results are consistent with previous findings reported by Lucchini et al. [80] and Adekayode and Olojugba [81]. Although ash is known for its neutralizing effect on acidic soils, some works did not demonstrate any significant changes in soil pH upon its use [31,82]. The efficacy of acidic soil neutralization is determined by the method of alkalizing fertilizer application, its dose, and its qualities, including its neutralizing value, fraction size, and dissolution rate, as well as by the composition and type of soil [31,35,80].

4.3. Correlations between the Analyzed Parameters

In the case of soils not exposed to the pressure of pollutants, crop yield is usually positively correlated with EBC, soil saturation with bases (BS), CEC, and contents of carbon and nitrogen [83]. In the present study, the soil was exposed to the effect of ash from *Salix viminalis*, and despite increased EBS, BS, and CEC values and also C_{org} content as a result of its application, no positive correlation was observed between the produced *Zea mays* biomass and these parameters. This lack of correlation was probably due to the introduction of not only Ca^{2+}, Mg^{2+}, Na^{+}, and K^{+} ions essential to plants but also excess amounts of heavy metals with ash [24–26,84]. Soil pollution with heavy metals usually upsets its biological properties, which is manifested as adverse effects on plants [85,86] and soil microbiota [87], which ultimately lead to severe disorder in the enzymatic activity of soil [88–91] because heavy metals may cause enzyme denaturation [92,93]. The present study demonstrated a negative correlation between the activity of seven soil enzymes and basic physicochemical properties of soil changing upon the influence of osier ash.

5. Conclusions

The heating value of *Zea mays* L. remained stable and unmodified by the excess content of ash from *Salix viminalis* in the soil, which makes it a viable energy crop to be grown on soils fertilized with large doses of ash. All the more, it is relatively resistant to adverse ash effects because its biomass was significantly reduced in the present study upon soil pollution with the highest ash dose tested (20 g kg^{-1} d.m. soil). Nevertheless, ash may cause unbeneficial changes in the soil environment, manifested as suppressed enzymatic activity. Although the ash increases soil pH and sorption capacity, these positive effects do not compensate for losses evoked by disorders in the biochemical properties of soil. These adverse effects may, in part, be mitigated by soil fertilization with compost and HumiAgra preparation. The choice of these fertilizers should, however, be driven by their efficacy, with compost shown to surpass HumiAgra in this respect.

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Abbreviations

C	soil without compost and HumiAgra;
K	soil with compost;
H	soil with HumiAgra;
AP	yield of aboveground parts;
R	yield of roots;
Deh	dehydrogenases;
Cat	catalase;
Ure	urease;
Pac	acid phosphatase;
Pal	alkaline phosphatase;
Glu	β-glucosidase;
Aryl	arylsulfatase;
C_{org}	total organic carbon;
N_{total}	total nitrogen;
HAC	hydrolytic acidity;
EBC	total exchangeable base cations;
CEC	total cation exchange capacity of soil;
BS	basic cation saturation ratio in soil.

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