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Effects of Sewage Sludge and Biochar Amendments on Heavy Metal Immobilization and Plant Growth at Degraded Zn-Pb Industrial Sites

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Abstract

The management of sewage sludge and the remediation of industrial landfills contaminated with heavy metals remain significant environmental challenges. The aim of this study was to evaluate the potential for using sewage sludge and the biochar derived from it as additives to support the phytostabilization of Zn-Pb industrial landfills. Two types of ore-washing waste, differing in their physicochemical properties and metal content (WW1 and WW2), were modified by adding sewage sludge (5–15%), biochar (5–15%), and sand (25%) in various combinations. Tall fescue (*Festuca arundinacea*) was used as the test plant and was grown for 30 days under controlled conditions. Germination, biomass growth, and the content of Zn, Pb, and Cd in the plant's aboveground parts were evaluated. Both sewage sludge and biochar improved plant growth; however, their effectiveness depended on the properties of the waste. Sewage sludge was more effective with waste WW1, while biochar had a particularly beneficial effect on waste WW2. The highest biomass increase for WW2, amounting to 2.66 g and corresponding to a 106% increase compared to the control treatment (waste without additives), was obtained for the WW60/B15/S25 variant. All additives used reduced the accumulation of Zn, Pb, and Cd in the biomass compared to the control treatment. The results indicate that biochar derived from sewage sludge, particularly when combined with a structure-forming additive (sand), can effectively support the phytostabilization of highly contaminated Zn-Pb industrial waste, while also enabling the management of sewage sludge in accordance with the principles of a circular economy.

Keywords: sewage sludge; biochar from sewage sludge; phytostabilization; Zn-Pb industrial waste; heavy metals; *Festuca arundinacea*; metal immobilization

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Academic Editor: Jon Alvarez

Received: 12 October 2026
Revised: 12 November 2026
Accepted: 18 November 2026
Published: 20 November 2026

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1. Introduction

Areas subjected to long-term zinc and lead mining and smelting activities are often heavily contaminated with heavy metals, particularly zinc, lead, and cadmium. These metals are highly persistent in the environment, toxic to living organisms, and capable of bioaccumulation, posing a threat to ecosystem functioning and human health. Their elevated concentrations also impair the physicochemical properties of the substrate, inhibit plant growth, and hinder the reclamation of post-industrial sites.

Waste generated during the mining and processing of zinc–lead ores and deposited over extensive areas covering several hundred hectares represents a major source of contamination. This waste is characterized by high phytotoxicity, a fine-grained texture, and a deficiency of organic matter and nutrients essential for proper plant growth, all of which

substantially hinder its biological reclamation. The presence of Zn, Pb, and Cd may further cause physiological disturbances and inhibit seed germination, plant growth, and root system development. Although zinc is an essential micronutrient, excessive concentrations are phytotoxic and may inhibit plant growth [1]. Lead has no biological function in plants, and its uptake may disrupt metabolic and photosynthetic processes and inhibit root system growth [2]. Excessive accumulation of this element may reduce root growth by as much as 42% [3]. Cadmium, in turn, is among the most toxic heavy metals and may disrupt physiological processes, nutrient uptake, and plant development [4–6]. The adverse effects of cadmium include inhibition of carbon assimilation, reductions in chlorophyll content and photosynthetic efficiency, suppression of antioxidant system activity, chlorosis, inhibition of lateral root formation, reduced root respiration, decreased transpiration, and lower relative water content in leaves.

The lack of effective biological stabilization of the surfaces of Zn-Pb industrial waste disposal sites intensifies erosion processes, resulting in dust emissions and the dispersal of fine particles containing heavy metals beyond the areas where the waste was deposited. A key challenge in reclaiming such sites is therefore to reduce metal mobility and bioavailability while improving substrate properties and establishing conditions that support sustained vegetation growth.

The use of sewage sludge in the reclamation of metal-contaminated sites may offer benefits by improving substrate physicochemical properties, increasing organic matter and nutrient contents, and promoting plant growth; however, it may also carry the risk of introducing additional metals and other contaminants into the substrate [7,8].

In recent years, there has been growing interest in methods based on the use of appropriate soil amendments that enable the immobilization of metals in forms that are biologically inaccessible. This approach is considered economically viable, minimally invasive, and effective, especially in the case of heavily degraded post-industrial soils. One of the materials used for this purpose is sewage sludge, which can stabilize the surface of waste materials prone to dust emissions and erosion, while also exhibiting beneficial fertilizing and soil-forming properties [7,8]. This is primarily due to its content of organic matter and mineral components, including N, P, K, Ca, Mg, Fe, and S [9–11]. However, the direct use of sewage sludge may be limited due to the presence of heavy metals, pathogenic microorganisms, and other organic contaminants [12,13]. The presence of heavy metals in sewage sludge may lead to an increase in their total content in the growing medium; however, this does not necessarily result in a proportional increase in their bioavailability and accumulation in plants, as these processes depend, among other factors, on the properties of the growing medium, the sludge application rate, the plant species [14,15], and the chemical forms of the metals, as they may occur, for example, in poorly soluble forms such as sulfides, oxides, hydroxides, silicates, and phosphates [16,17].

An alternative to the direct application of sewage sludge is its thermochemical conversion into biochar. Using sewage sludge as a feedstock for biochar production enables the utilization of a waste stream and the production of a stable carbonaceous material with potential environmental applications, thereby supporting circular economy principles [18].

Biochar is characterized by a well-developed porous structure, the presence of surface functional groups, and a generally alkaline pH. These properties enable metal immobilization through surface adsorption, complexation, ion exchange, and the precipitation of sparingly soluble compounds, including metal hydroxides and phosphates. Biochar application may also increase substrate pH and cation exchange capacity, reducing metal solubility and uptake by plants. For sewage-sludge-derived biochar, pyrolysis has been shown to substantially reduce the bioavailability of metals, including Zn, Pb, and Cd, despite an increase in their total concentrations in the material resulting from the concentration of mineral con-

stituents during thermal treatment [19]. The properties of sewage-sludge-derived biochar are determined by feedstock characteristics and thermochemical processing conditions, particularly pyrolysis temperature, residence time, and heating rate [20–22]. These parameters affect the chemical composition, stability, surface structure, and sorption properties of the resulting material, thereby determining its suitability for heavy metal immobilization and the reclamation of degraded substrates. Previous studies have demonstrated the potential of sewage-sludge-derived biochar for the remediation of metal-contaminated soils. Wang et al. [18] showed that its application to soils co-contaminated with Cu and Cd improved substrate properties and reduced metal mobility and bioavailability by promoting their redistribution into more stable fractions. Radziemska et al. [23] also demonstrated the beneficial effects of biochar-containing materials on the phytostabilization of contaminated soils, observing increases in plant biomass and soil pH, among other effects.

The study by Namdari et al. [19] is particularly relevant for comparing the potential use of sewage sludge and its derived biochar in the remediation of contaminated sites. The authors demonstrated that the thermochemical conversion of sludge into biochar reduced the bioavailability of potentially toxic elements and their accumulation in the tissues of *Zea mays* L., while also promoting plant biomass production.

Previous attempts to stabilize and biologically reclaim zinc–lead industrial waste, particularly ore-washing waste, have not yielded satisfactory results, as a persistent and complete vegetation cover has not been established (Figure 1).



Figure 1. Area for the disposal of ore-washing waste (southern Poland, the Zn-Pb industry region).

Preliminary studies conducted by the authors in 2016–2017 [24] assessed the concentrations of bioavailable forms of selected heavy metals in Zn-Pb ore-processing waste that may have contributed to the difficulties encountered in reclaiming these waste disposal sites. The findings provided a basis for further research in this area.

The aim of the present study was to evaluate the effects of sewage sludge and its derived biochar on plant growth and heavy metal mobility in a substrate composed of zinc–lead industrial waste, with a view to assessing its potential for biological reclamation. The study was conducted using Zn-Pb ore-washing waste and tall fescue (*Festuca arundinacea*), selected for its high tolerance to extreme environmental conditions, including temperature and moisture stress.

2. Materials and Methods

2.1. Research Procedure

To prepare the substrate mixtures used in the experiment, waste from the zinc–lead industry (designated as WW) was used, along with the following additives: sanitized and dried (granulated at a temperature above 100 °C) sewage sludge (designated as SS), biochar produced from this sludge (designated as B), and sand (designated as S).

The waste was collected from a landfill located in southern Poland. This is an area affected by the zinc and lead industry. This landfill was selected because of the difficulties in achieving biological remediation, despite many years of attempts to do so. Based on the selected materials, mixtures were prepared according to the scheme described in Table 1. Following this procedure, separate mixtures were prepared for samples collected from two locations at the landfill—the sample labeled WW1 (Figure 2) came from areas covered with vegetation, while the sample labeled WW2 (Figure 2) was collected from areas without such vegetation.

Table 1. Composition of mixtures and their identification.

Mixtures	Ore-Washing Waste (WW) *w*/*w* %	Sewage Sludge (SS) *w*/*w* %	Biochar (B) *w*/*w* %	Sand (S) *w*/*w* %
WW1/95/SS5	95	5	-	-
WW2/95/SS5	95	5	-	-
WW1/90/SS10	90	10	-	-
WW2/90/SS10	90	10	-	-
WW1/85/SS15	85	15	-	-
WW2/85/SS15	85	15	-	-
WW1/70/SS5/S25	70	5	-	25
WW2/70/SS5/S25	70	5	-	25
WW1/65/SS10/S25	65	10	-	25
WW2/65/SS10/S25	65	10	-	25
WW1/60/SS15/S25	60	15	-	25
WW2/60/SS15/S25	60	15	-	25
WW1/95/B5	95	-	5	-
WW2/95/B5	95	-	5	-
WW1/90/B10	90	-	10	-
WW2/90/B10	90	-	10	-
WW1/85/B15	85	-	15	-
WW2/85/B15	85	-	15	-
WW1/70/B5/S25	70	-	5	25
WW2/70/B5/S25	70	-	5	25
WW1/65/B10/S25	65	-	10	25
WW2/65/B10/S25	65	-	10	25
WW1/60/B15/S25	60	-	15	25
WW2/60/B15/S25	60	-	15	25
WW1/75/S25	75	-	-	25
WW2/75/S25	75	-	-	25
WW1/100	100	-	-	-
WW2/100	100	-	-	-

w/*w* %—denotes the percentage by mass of each component relative to the total mass of the mixture prepared for the experiment.

Zn-Pb industrial waste is characterized by high Zn, Pb, and Cd contents and a carbonate-rich mineral composition, reflecting the properties of the mined ores. A detailed characterization of this waste was provided by Sobik-Szołtysek et al. [24]. Municipal sewage sludge (SS) was collected from a large wastewater treatment plant located in the Silesian Voivodeship, Poland, treating approximately 16 million m^3 of municipal wastewater annually. Before the mixtures were prepared, the individual substrate components were air-dried.

Sewage sludge was pyrolyzed in a PRW-S100x780/11 furnace (Czylok, Jastrzębie-Zdrój, Poland). After the process was completed, the material was left to cool in the reactor. The produced biochar samples were placed in airtight containers. The process parameters are presented in Table 2.



Figure 2. A view of the area from which the WW1 and WW2 waste was collected (southern Poland, the Zn-Pb industry region).

Table 2. Pyrolysis process parameters.

Heating Time	Reaction Time	Process Temperature	Inert Gas (N_2) Flow Rate
120 min	90 min	550 °C	5 L min^{-1}

Both the sewage sludge and the biochar derived from it were in granular form and were ground to a particle size below 500 µm to increase the surface area available for contact with the substrate and plants.

The materials used to prepare the mixtures were analyzed for selected physicochemical properties relevant to the study, and their selected heavy metal contents were determined. The experiment was conducted separately for samples WW1 and WW2. The prepared mixtures were used as substrates for growing tall fescue (*Festuca arundinacea*) under controlled conditions in a plant growth test. At the end of the test, the aboveground plant biomass was harvested, dried at 80 °C, and weighed. Following digestion, the biomass was analyzed for heavy metal content.

2.2. Plant Testing Procedure

A plant growth test using tall fescue (*Festuca arundinacea*) was conducted to assess the potential of the amendments to enhance the biological reclamation of ore-washing waste. This species was selected for its high tolerance to temperature and moisture stress and its ability to grow in nutrient-poor and degraded soils. Furthermore, owing to its ability to develop an extensive root system and form dense turf, *Festuca arundinacea* is widely used for surface stabilization and erosion control [25,26].

The plant growth test was conducted in accordance with the standard cited in [27]. Plants were grown in non-porous pots measuring 10 × 10 cm and 20 cm in height, filled with the prepared substrate to 1 cm below the upper rim. The substrates were prepared according to the combinations presented in Table 1, and separately for waste samples WW1 and WW2.

Tall fescue seeds purchased directly from the producer were sown at a rate of 0.4 g per pot, with three replicates per treatment. Plants were grown under controlled conditions

in a phytotron growth chamber. The growth conditions in the phytotron chamber were as follows:

- Phytotron chamber: Biogenet FS360 (BIOGENET, Józefów, Poland);
- Cultivation period: 30 days;
- Photoperiod: 16 h light/8 h dark;
- Temperature: 21 °C during the light period and 18 °C during the dark period;
- Light source: photosynthetic LED lighting;
- Light intensity: 4000 lx;
- Irrigation method: manual spraying;
- Irrigation water: deionized water;
- Irrigation dose: 100 mL every two days;
- Initial irrigation: irrigation was first performed using a sufficient volume of deionized water to completely fill the pore space of the substrate.

2.3. Analytical Procedures

Solid samples intended for analysis were dried at room temperature and ground in a mortar prior to analysis. Metal concentrations in solid and liquid samples were determined by inductively coupled plasma optical emission spectrometry (ICP-OES) using a Thermo Scientific™ IRIS Intrepid II XSP DUO instrument (Thermo Fisher Scientific, Waltham, MA, USA). Analyses were performed using an internal standard, and the accuracy of the results was verified using reference material BL 12-1-12. Prior to analysis, solid samples were digested in *aqua regia* according to the procedure specified in the standard cited in [28].

Physicochemical analyses were performed using the following methods:

- PH was measured in aqueous extracts using the multiparameter HI 9828 multiparameter meter (Hanna Instruments, Woonsocket, RI, USA) (in accordance with the standard cited in [29];
- Dry matter (d.m.) content was determined by drying at 105 °C to constant mass;
- Organic matter (o.m.) content was determined by loss on ignition in a muffle furnace at 500 °C for 5 h;
- Determination of total Kjeldahl nitrogen (TN) content was carried out in accordance with [30] using the K-439 BÜCHI mineralizer and the K-355 BÜCHI steam distillation apparatus (BÜCHI Labortechnik AG, Flawil, Switzerland);
- Available phosphorus (P_{avail}) extraction was performed by the Egner–Riehm method; samples were extracted with a calcium lactate solution buffered to a pH value of approx. 3.6 and phosphorus was determined in the obtained extract by the spectrophotometric method with ammonium molybdate in accordance with [31] using the Hach-Lange DR 5000 spectrophotometer (Berlin, Germany);
- Specific surface area was determined by the BET method using an ASAP 2020 Plus analyzer (manufactured by Micromeritics Atlanta, GA, USA).

Images of biochar morphology were obtained using a Nova NanoSEM 200 scanning electron microscope (FEI Europe, Eindhoven, the Netherlands) at a magnification of 3000×. The particle size distribution of the ore-washing waste was determined using an LAU-10 laser particle size analyzer (Opole, Poland).

2.4. Statistical Analysis

Statistical analysis was performed using Prism 9.0 software package (GraphPad Software, Inc., Boston, MA, USA). Data expressed on a quantitative scale were presented as the mean with standard deviation (SD). Differences between the groups were analyzed with a one-way ANOVA followed by a Dunnett's post-hoc test. Results were considered statistically significant when $p < 0.05$.

3. Results and Discussion

3.1. Characteristics of the Substrates Used in the Study

The characteristics of the materials used as substrate components for growing the test plant are presented in Tables 3 and 4.

Table 3. Selected physicochemical parameters and the TN and P_{avail} content of the substrates used to prepare the mixtures.

Substrate	pH	d.m.	o.m.	TN	P_{avail}
		%	%	%	%
SS	7.13 ± 0.07	94.64 ± 0.12	60.39 ± 0.03	4.087 ± 0.048	0.1120 ± 0.0008
B	8.19 ± 0.13	98.77 ± 0.22	33.48 ± 0.28	3.130 ± 0.057	0.1401 ± 0.0009
WW1	7.53 ± 0.21	98.06 ± 0.21	2.3 ± 0.14	0.38 ± 0.04	0.0023 ± 0.00003
WW2	7.48 ± 0.30	97.96 ± 0.23	0.190 ± 0.01	0.06 ± 0.02	0.0013 ± 0.00002
S	7.06 ± 0.005	99.72 ± 0.03	0.040 ± 0.001	0.028 ± 0.013	0.0052 ± 0.0010

SS—sewage sludge; B—biochar; WW1—zinc–lead industrial waste collected from a site with a biological cover; WW2—zinc–lead industrial waste collected from a site without a biological cover; S—sand; d.m.—dry matter; o.m.—organic matter; TN—total nitrogen; P_{avail}—available phosphorus.

Table 4. The content of selected metals in substrates used to prepare mixtures.

Parameter	Substrate				
	SS	B	S	WW1	WW2
	$mg \cdot kg^{-1}$ d.m.				
Fe	38,126.2 ± 217.8	43,822.0 ± 137.0	1956 ± 30.35	189,800.0 ± 2847.2	220,300.2 ± 2643.6
Zn	573.7 ± 13.1	3896.2 ± 17.1	66.4 ± 1.28	59,530.2 ± 1250.13	105,600.7 ± 1932.5
Pb	189.2 ± 2.3	288.2 ± 4.3	12.45 ± 0.23	10,150.4 ± 152.25	17,910.0 ± 268.65
Cd	8.7 ± 0.2	10.5 ± 0.3	0.598 ± 0.01	409.4 ± 7.78	268.6 ± 5.1
Ca	44,396.6 ± 116.6	74,125.8 ± 556.8	353.3 ± 67.8	203,940.0 ± 14,275.8	215,471.0 ± 15,729.4
Mg	4494.4 ± 22.2	7739.6 ± 24.8	1477.5 ± 24.6	90,220.6 ± 1290.15	87,118.3 ± 1245.79
Na	1293.6 ± 9.9	1976.6 ± 29.0	158.35 ± 0.98	2300.9 ± 30.37	2285.8 ± 31.17

SS—sewage sludge; B—biochar; WW1—zinc–lead industrial waste collected from a site with a bi-ological cover; S—sand; WW2—zinc–lead industrial waste collected from a site without a biological cover.

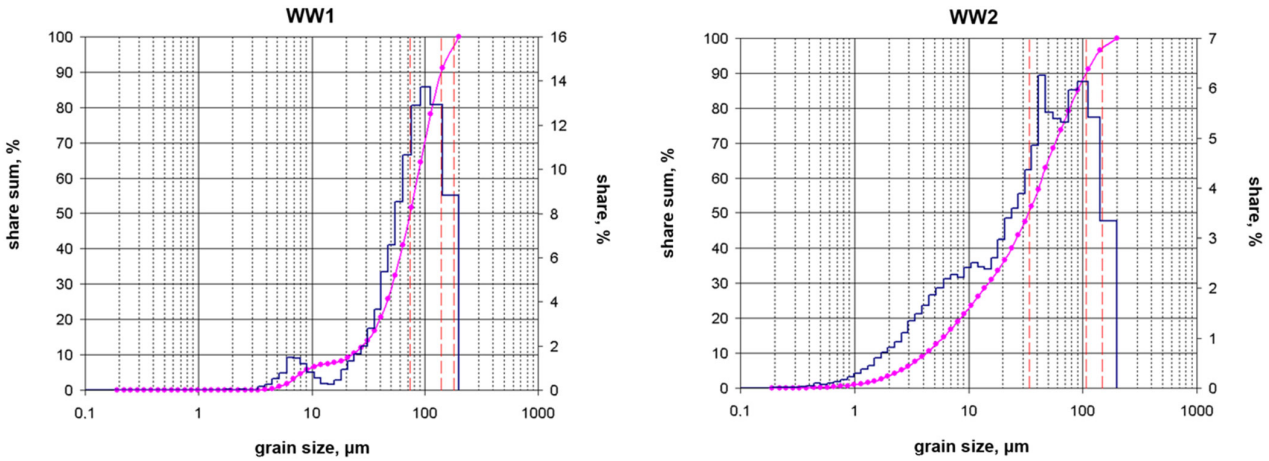
The materials analyzed had similar pH values ranging from 7.06 to 7.53, with the exception of biochar, which had a pH of 8.19. The dry matter content of all substrate components exceeded 94%. Municipal sewage sludge had the highest organic matter content, reflecting its origin. The organic matter content of the biochar produced from this sludge was approximately 33%, reflecting the production process. The remaining materials had very low organic matter contents, below 1%, except for WW1, in which the content exceeded 2%. Regarding the nutrients N and P, which are essential for plant nutrition, nitrogen contents of 3–4% were recorded only in sewage sludge and biochar, whereas those in the remaining materials did not exceed 0.5%. Phosphorus contents in sewage sludge and biochar ranged from 0.11 to 0.14%. In the remaining components, phosphorus contents ranged from 0.0013 to 0.0052%.

Given the origin of the waste (WW) used to prepare the mixtures, its selected metal contents were determined. The other mixture components were subjected to the same analysis (Table 4).

Analysis of heavy metal concentrations in the substrate components (Table 4) showed that WW1 and WW2 had the highest Zn, Pb, Cd, and Fe contents. This reflects the mineralogical characteristics of the Zn-Pb ores from which the waste originated. Zn, Pb, and Fe concentrations were higher in WW2 compared to WW1, whereas the Cd concentration was lower. A distinctive feature of WW2 was the absence of vegetation cover at its disposal loca-

tion, which may suggest that elevated Zn and Pb concentrations adversely affect biological reclamation. None of the other mixture components exhibited similarly high concentrations of Zn, Pb, Cd, and Fe. Therefore, these amendments did not pose a risk of substantially increasing the concentrations of these metals in the mixtures. For alkaline metals (Ca, Mg, and Na), the highest contents were again recorded in WW1 and WW2, which is also associated with the carbonate-rich nature of Zn-Pb ores. No substantial differences in the contents of these metals were observed between WW1 and WW2, reflecting their origin from the same ore-bearing formations.

Difficulties in effectively reclaiming ore-washing waste disposal sites may be attributable not only to the presence of heavy metals and a lack of nutrients but also to the physical structure of the deposited waste. Optimal conditions for plant growth and development occur in soils with clayey silt, light loam, and medium silty loam textures. Particle size analysis was performed on WW1 and WW2 to determine their particle size distributions. The results are presented in Figure 3.



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

Figure 3. Grain size distribution of the wash water waste for samples (**a**) WW1 and (**b**) WW2.

The sand fraction (particles ranging from 0.05 to 0.2 mm) accounted for the largest proportion of WW1, whereas the silt fraction (particles ranging from 0.002 to 0.05 mm) predominated in WW2. According to the USDA soil textural classification [32], WW1 is classified as sandy loam and WW2 as silt loam. This particle size distribution makes the waste susceptible to water erosion and, when highly compacted, as occurs at the disposal site, hinders root system development. Such a substrate cannot provide optimal conditions for vigorous plant growth because of poor aeration, severe surface crusting, strong water retention, and limited water availability to plants. This suggests that the particle size distribution needs to be modified through the addition of suitable mineral amendments. Sand was therefore selected as one of the components of the prepared soil mixtures.

Numerous studies [19,33] have shown that heavy metal bioavailability can be reduced by applying biochar as a soil amendment. Given these properties of biochar, its specific surface area and pore morphology were characterized. The BET specific surface area was 42.022 $m^2 g^{-1}$. Figure 4 shows a scanning electron micrograph of the biochar before grinding.

Pores ranging in diameter from approximately 5 to over 20 µm are visible in the image. The pores have irregular, angular, and highly intricate three-dimensional shapes. They are open and unevenly distributed. The pore walls consist of clusters of fine particles forming a loosely packed structure.

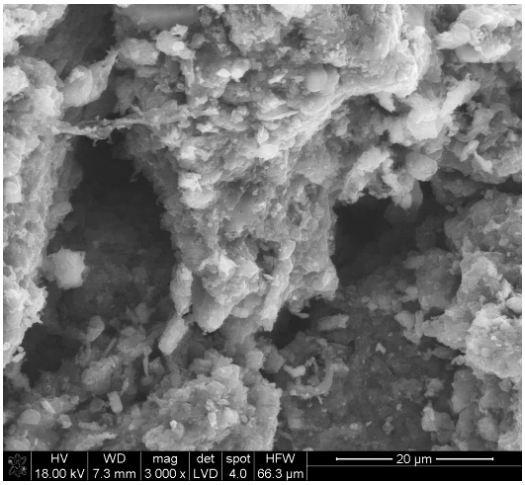


Figure 4. A photo of biochar magnified 3000 times. HV—accelerating voltage of the electron beam; WD—distance between the specimen surface and the objective lens pole piece; Mag—magnification of the SEM image; Det—type of detector; Spot—electron beam spot-size setting; HFW—horizontal width of the imaged area.

3.2. Biomass Growth

Tall fescue biomass was produced on the prepared substrates during the experiment. The biomass yields obtained, expressed in grams, are presented in Figure 5.

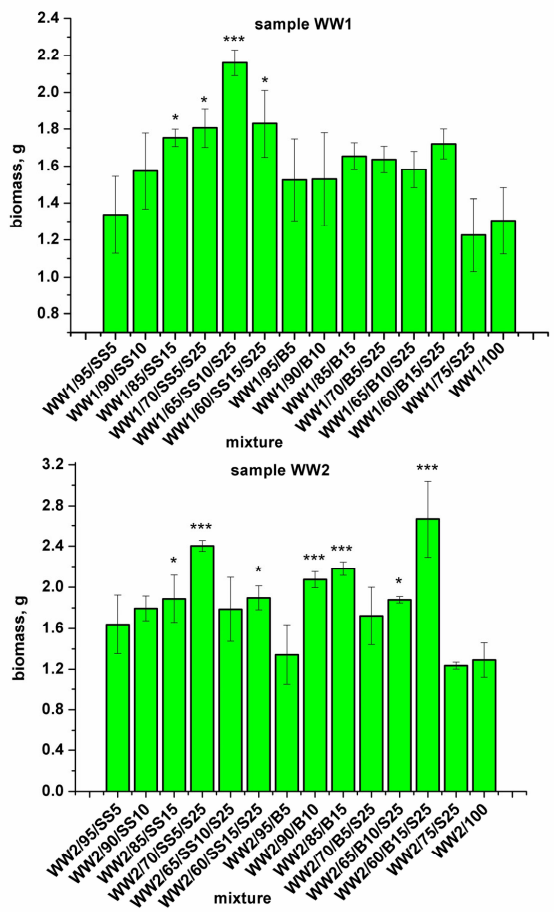


Figure 5. Biomass growth on substrates based on WW1 and WW2 waste; Dunnett's post-hoc test; * $p < 0.05$; *** $p < 0.001$.

For WW1, the highest biomass yield was obtained in the WW1/65/SS10/S25 treatment (2.16 g), representing a 65% increase relative to the unamended waste control (Figure 5). The lowest biomass yield was observed in the WW1/75/S25 treatment, which was 6% lower than that of the control (WW1/100).

Analysis of the effects of sewage sludge addition showed that increasing the sludge application rate resulted in higher biomass yields. Increasing the sludge application rate from 5% to 10% resulted in a marked increase in biomass of approximately 17.7%. A further increase of 5 percentage points produced a smaller effect, with biomass increasing by only 11.4%. This pattern is supported by the findings of Eid et al. [34], who applied sludge as a biofertilizer at different rates (10, 20, 30, and 40 g kg^{-1}). The highest growth indices for the test plant (*Corchorus olitorius*) were recorded at a sludge application rate of 20 g kg^{-1}. Given the fine-grained nature of the ore-washing waste, an additional series of sewage sludge treatments was prepared with 25% sand in each mixture. In all cases, sand addition increased biomass yield compared with the corresponding treatments amended with sewage sludge alone. This positive effect was confirmed by statistical analysis, which showed significantly higher biomass yields for WW1/70/SS5/S25 ($p < 0.05$), WW1/60/SS15/S25 ($p < 0.05$), and WW1/65/SS10/S25 ($p < 0.001$).

The use of biochar as another amendment also increased biomass yield by 17–27% relative to the control (WW1/100). The greatest increase was recorded at a biochar application rate of 15%. This relationship is supported by the findings of Velli et al. [35], who used sewage-sludge-derived biochar as an amendment to soil substrates. The authors reported increased aboveground and belowground biomass of the test plant. Adding sand to the biochar-amended mixtures further enhanced biomass production, with the highest yield obtained in the WW1/60/B15/S25 treatment. This represented a 4% increase in biomass compared with the WW1/85/B15 treatment.

A comparison of biomass yields between the waste-only substrate (WW1/100) and the substrate amended with sand alone (WW1/75/S25) showed better results for the control. This may have resulted from the loosening of the waste structure, which could have reduced its water retention capacity.

For substrates containing WW2, the highest biomass yield was obtained in the WW2/60/B15/S25 treatment (2.66 g), representing a 106% increase relative to the unamended waste control (WW2/100) (Figure 5). The lowest biomass yield was observed in the WW2/75/S25 treatment, which was 4.3% lower than that of the control (WW2/100).

Analysis of the effects of sewage sludge addition showed that increasing the sludge application rate increased biomass yield, although the differences between application rates did not exceed 10%. Statistical analysis confirmed that biomass yield in the WW2/85/SS15 treatment was significantly higher than that of the control ($p < 0.05$). Adding sand to the substrate alongside sewage sludge further increased biomass yield. The best results were obtained for the WW2/70/SS5/S25 treatment, and this effect was confirmed by statistical analysis ($p < 0.001$). Compared with the treatment containing the highest sludge application rate (WW2/85/SS15), the WW2/70/SS5/S25 treatment produced a 27.8% higher biomass yield. Of the remaining treatments (WW2/65/SS10/S25 and WW2/60/SS15/S25), only WW2/60/SS15/S25 showed a statistically significant effect ($p < 0.05$).

As observed for WW1, amending the waste with biochar increased biomass yield. The greatest effects were obtained at biochar application rates of 10% and 15%, with statistical analysis confirming highly significant differences ($p < 0.001$). Adding sand to the biochar-containing mixtures produced the greatest effect at the 15% biochar application rate (WW2/60/B15/S25). This treatment resulted in the highest biomass yield among all treatments based on WW2. The effect was statistically significant ($p < 0.001$).

As observed for WW1, the mixture containing WW2 and sand (WW2/75/S25) produced a lower biomass yield than the control (WW2/100).

A comparison of the experimental results for WW1 and WW2 substrates showed that sewage sludge was the most effective amendment for WW1. For WW2, however, biochar was the most effective amendment. These patterns may reflect the fact that WW1 originated from vegetated locations at the waste disposal site and that sewage sludge addition increased microbial activity. As demonstrated by Hechmi et al. [36], the effects of sewage sludge application depend, among other factors, on the properties and texture of the receiving soil. Ciarkowska et al. [37] reported that sewage sludge application for the phytostabilization of flotation tailings produced better results than mineral fertilization. Sewage sludge addition improved conditions for plant growth, including increased contents of $C_{organic}$, N_{total}, P, and Mg [24]. Sewage sludge helps stabilize soil pH, which directly affects the bioavailability of micronutrients and macronutrients.

In WW2, biochar increased water retention, which was particularly important because the fine-grained texture of this waste hindered the establishment of vegetation cover.

However, these effects depended on the addition of sand to the mixtures. Thus, alongside the organic amendments, sewage sludge and biochar, sand can be considered a third key component contributing to increased biomass yield of the test plant. Sand loosens the fine-grained ore-washing waste, increasing water retention and substrate permeability while improving its structure.

In order to verify the effects observed in the controlled vase experiment, it would be necessary to conduct field studies that take into account the influence of variable climatic factors and long-term interactions between plants and the additives used. The transition from the laboratory to the field scale is therefore the next step in assessing the durability of the observed effects and the practical applicability of the proposed solutions in the remediation of degraded and heavy-metal-contaminated sites. This issue will be the subject of further research.

3.3. Plant Germination Efficiency (Time)

The time to first seedling emergence was assessed for the tested substrates. The observations are summarized in Table 5.

Table 5. Germination time of plants on different growing media.

Substrate	WW1			WW2		
	Day 7	**Day 10**	**Day 14**	**Day 7**	**Day 10**	**Day 14**
WW95/SS5	0	0	+	+	+	+
WW90/SS10	+	+	+	0	0	+
WW85/SS15	0	0	+	0	0	+
WW70/SS5/S25	0	+	+	+	+	+
WW65/SS10/S25	0	+	+	+	+	+
WW60/SS15/S25	0	0	+	0	0	+
WW/95/B5	+	+	+	0	+	+
WW90/B10	0	0	+	+	+	+
WW85/B15	0	0	+	0	+	+
WW70/B5/S25	+	+	+	0	0	+
WW65/B10/S25	0	+	+	0	0	+
WW60/B15/S25	0	0	+	+	+	+
WW75/S25	+	+	+	0	0	+
WW100	0	+	+	0	0	+

+ full germination; 0 no germination.

Germination time varied depending on the mixture used, as confirmed by observations on days 7, 10, and 14 of cultivation. For substrates consisting solely of waste (WW1/100 and WW2/100), germination occurred earlier in WW1. A similar effect was observed in treatments in which sand was the only amendment. Earlier seedling emergence in WW1 may be attributable to several factors, including higher organic matter, nitrogen, and phosphorus contents than in WW2 (Table 3), as well as lower Zn and Pb contents (Table 4). Current research indicates that elevated concentrations of metals such as Zn and Cd may impair seed germination and seedling growth. The degree of phytotoxicity depends on factors including metal dose, plant species and genotype, and soil physicochemical properties, which determine metal mobility and bioavailability [1,38–40]. Zhang et al. demonstrated the adverse effects of Zn on plant growth and development, including inhibited lettuce root growth, expressed as reductions in root length, surface area, volume, and number of root tips, as well as decreased photosynthetic pigment contents (chlorophyll a, chlorophyll b, and carotenoids) [41]. Vasilachi-Mitoseru et al., among others, reported that higher soil Pb concentrations inhibited seed germination and stem growth in *Medicago sativa* L. and *Zea mays* L. [42].

The timing of seedling emergence was similar in mixtures of WW1 and WW2 with sewage sludge, although differences occurred depending on the sludge application rate. In WW2-based mixtures, germination was clearly delayed as the proportion of sewage sludge increased. This relationship was not observed in mixtures of WW1 and sewage sludge. The earliest germination in this group was recorded at a sewage sludge application rate of 10%.

Adding sand to mixtures of waste and sewage sludge accelerated germination in substrates containing 5% and 10% sludge. At a sludge application rate of 15%, however, germination was delayed.

Regardless of the application rate, biochar addition produced the earliest germination in WW2-based mixtures, in which germination was complete after 10 days. In WW1-based mixtures, this stage was reached only after 14 days.

The earliest germination was recorded in the WW1/75/S25 mixture, with maximum germination reached after only 7 days. This effect was not observed in the WW2/75/S25 mixture. Adding sand to WW1, which had higher organic matter and nutrient contents, improved the mixture structure and facilitated germination.

3.4. Metal Content in Biomass

3.4.1. Zinc Content in Biomass

Figure 6 presents Zn concentrations for biomass grown in the prepared mixtures containing WW1 and WW2.

Biomass grown in the substrate consisting solely of WW1 had the highest Zn concentration (1262.50 mg kg^{-1}) among all treatments containing this waste. Adding sand to the waste (WW1/75/S25) reduced the Zn concentration in biomass to 949.23 mg kg^{-1}, representing a decrease of 24.8%. Sewage sludge addition, both alone and in combination with sand, further reduced Zn concentrations in the harvested biomass compared with treatments containing waste alone or waste amended with sand. These findings were supported by statistical analysis, which showed significantly lower Zn concentrations ($p < 0.001$) in all treatments containing sewage sludge and sand compared with the control (WW1/100). A relationship was observed between the sewage sludge application rate and Zn concentrations in the resulting biomass. The difference between the highest Zn concentration (WW1/95/SS5) and the lowest (WW1/90/SS10) was 14%.

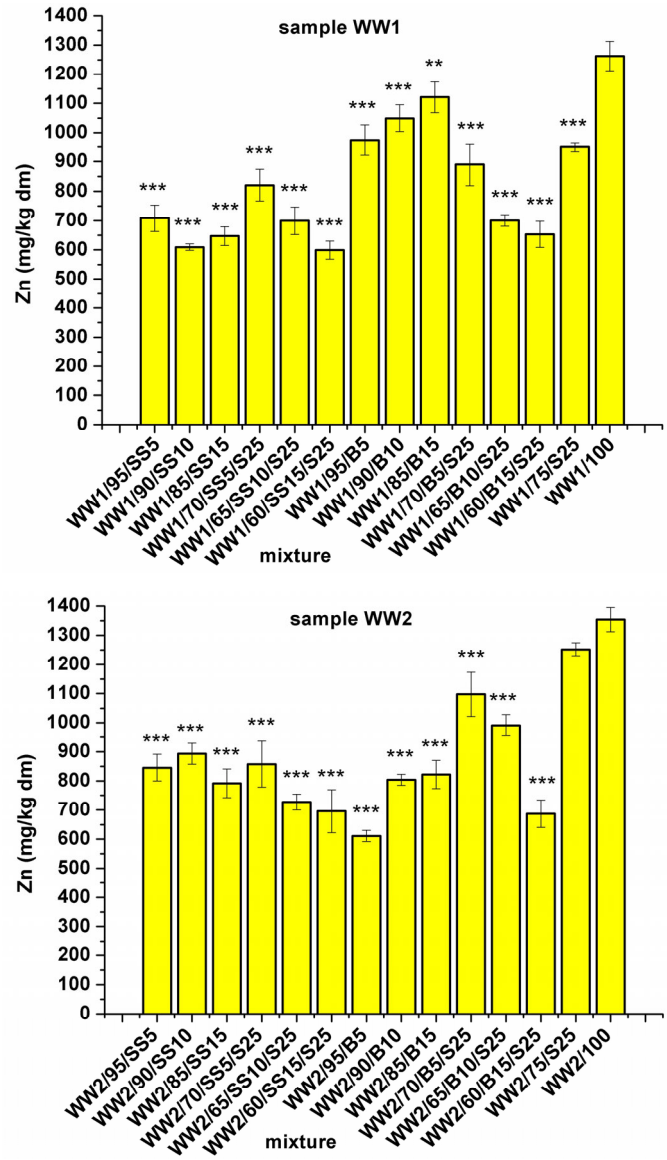


Figure 6. Zn content in biomass grown on substrates containing WW1 and WW2 waste; Dunnett's post-hoc test; ** $p < 0.01$; *** $p < 0.001$.

Similar patterns were observed in biomass grown in WW2-based substrates. The Zn concentration in biomass grown in WW2 was 1353.77 mg kg^{-1}, the highest value recorded in the entire experiment. Sand addition reduced Zn concentrations, while sewage sludge addition, both alone and in combination with sand, produced an effect similar to that observed for WW1. These findings were supported by statistical analysis, which showed significantly lower Zn concentrations ($p < 0.001$) in all treatments containing sewage sludge and sand compared with the control (WW2/100). The variation in biomass Zn concentrations with sewage sludge application rate, expressed as the difference between the highest and lowest concentrations, was smaller than for WW1 and amounted to 11.43%.

Biochar addition reduced Zn concentrations in biomass compared with the control for both WW1 and WW2. However, biomass Zn concentrations increased with increasing biochar application rates for both waste types. The difference in Zn concentrations between biochar application rates of 5% and 15% was 13.1% for WW1 and 25.65% for WW2.

Analysis of the effect of biochar application rate revealed a clear trend of increasing biomass Zn concentrations with increasing rates for both WW1 and WW2. The opposite trend was observed in treatments containing both biochar and sand, where increasing

the biochar application rate at a constant sand proportion of 25% reduced biomass Zn concentrations. This trend may result from dilution of the soil matrix with quartz, the principal constituent of sand, potentially reducing sorption capacity and accelerating Zn^{2+} leaching. Furthermore, sand addition increased oxygen diffusion into the soil, potentially promoting the partitioning of Zn into less soluble forms, including associations with iron and manganese oxides [43].

The maximum Zn concentrations measured in biomass from biochar treatments (1120.87 ± 52.69 mg kg^{-1} for WW1/85/B15 based on WW1 and 1097.44 ± 76.12 mg kg^{-1} for WW2/70/B5/S25 based on WW2) were markedly higher than the maximum values obtained for sewage sludge treatments (820.49 ± 53.50 mg kg^{-1} for WW1/70/SS5/S25 based on WW1 and 893.57 ± 36.10 mg kg^{-1} for WW2/90/SS10 based on WW2). These patterns are associated with the higher Zn content of biochar compared with sewage sludge (Table 4).

Regardless of the amendment used (sewage sludge, biochar, or their combinations with sand), biomass Zn concentrations were always lower than in the control (WW1/100 and WW2/100). Statistical analyses confirmed that all these differences were significant at $p < 0.001$.

3.4.2. Lead Content in Biomass

Figure 7 presents Pb concentrations in biomass grown in the prepared mixtures containing WW1 and WW2.

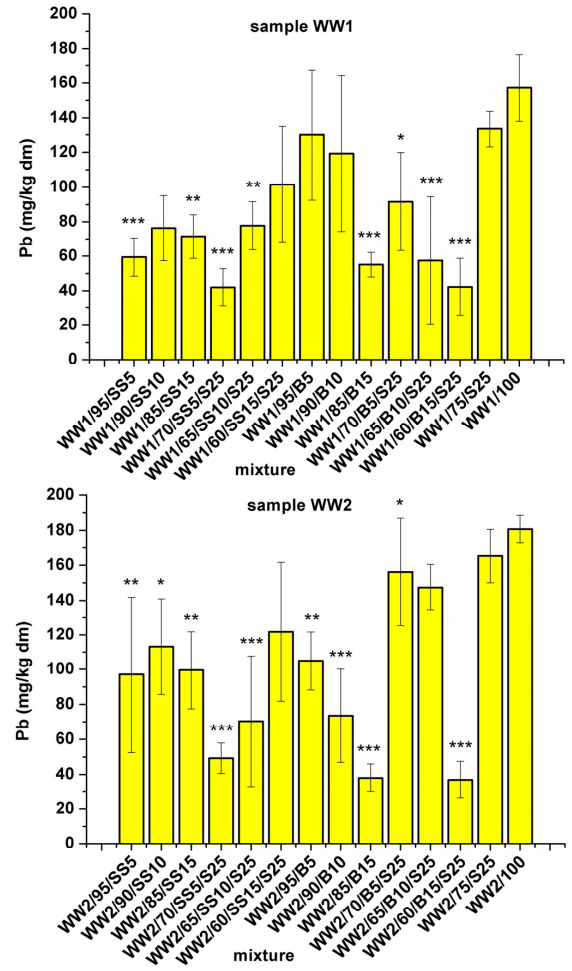


Figure 7. Pb content in biomass grown on substrates containing WW1 and WW2; waste Dunnett's post-hoc test; * $p < 0.05$; ** $p < 0.01$; *** $p < 0.001$.

Analysis of Pb concentrations in biomass grown in WW1- and WW2-based substrates showed that sewage sludge, biochar, and their combinations with sand reduced Pb uptake by the test plant.

The Pb concentration in biomass grown in unamended WW1 (WW1/100) was 157.30 mg kg^{-1}, whereas that for WW2 (WW2/100) was 180.830 mg kg^{-1}. Sand addition reduced this concentration by 34.3% for WW1 and 8.6% for WW2. When interpreting Pb concentrations in aboveground plant biomass, its relatively low mobility within the plant should be considered. Most absorbed Pb is retained in the root system, while its transport to aboveground tissues is generally limited and depends on factors including plant species and soil properties [44,45].

The addition of sewage sludge, alone or in combination with sand, resulted in varying levels of Pb translocation to aboveground tissues. For both waste types (WW1 and WW2), the optimal sludge application rate was 5%, resulting in the lowest biomass Pb concentrations, 59.47 mg kg^{-1} for WW1 and 97.1 mg kg^{-1} for WW2. Increasing the sewage sludge application rate increased biomass Pb concentrations, possibly because greater biomass production was accompanied by more extensive root system development and increased root contact with the contaminated substrate. A moderate sewage sludge application rate promotes Pb immobilization, whereas at higher rates, other mechanisms associated with the organic matter present in sewage sludge become active [46]. As shown in the study by Eid, E.M. et al. [45], the relationship between sludge dose and lead content in plants need not be perfectly linear, a finding confirmed by our study, in which the maximum lead content in biomass was observed at the intermediate dose (10% SS).

Treatments containing both sewage sludge and sand showed a clear trend of increasing biomass Pb concentrations with increasing sludge application rates at a constant sand proportion. These patterns were supported by statistical comparisons with the unamended waste controls (WW1/100 and WW2/100). For both WW1 and WW2, significantly lower biomass Pb concentrations were obtained in the WW1/95/SS5, WW2/95/SS5, WW1/85/SS15, WW2/95/SS5, WW1/70/SS5/S25, WW2/70/SS5/S25, and WW1/65/SS10/S25, WW2/70/SS5/S25 treatments, with significance levels ranging from $p < 0.05$ to $p < 0.001$. In the remaining treatments, the differences were not statistically significant ($p > 0.05$).

Most studies indicate that biochar immobilizes Pb and reduces its concentrations in plants [46,47]. This relationship was also observed in the present study. Biochar application, both alone and in combination with sand, reduced biomass Pb concentrations as the biochar application rate increased. For WW1, statistical analysis showed significant differences in the WW1/85/B15, WW1/65/B10/S25, and WW1/60/B15/S25 treatments ($p < 0.001$). In the remaining treatments, the differences were not statistically significant ($p > 0.05$). For WW2, statistically significant differences were obtained in the WW2/95/B5, WW2/90/B10, WW2/85/B15, and WW2/60/B15/S25 treatments, with significance levels ranging from $p < 0.05$ to $p < 0.001$.

3.4.3. Cadmium Content in Biomass

Figure 8 presents Cd concentrations for biomass grown in the prepared mixtures containing WW1 and WW2.

Analysis of biomass grown in unamended waste substrates (WW1/100 and WW2/100) showed Cd concentrations of 6.90 mg kg^{-1} for WW1 and 8.57 mg kg^{-1} for WW2. Cadmium has no biological function and can adversely affect organisms even at very low concentrations [48]. The roots of plants grown in Cd-contaminated soils readily absorb this element, which subsequently accumulates in edible plant parts, such as fruits and seeds [49].

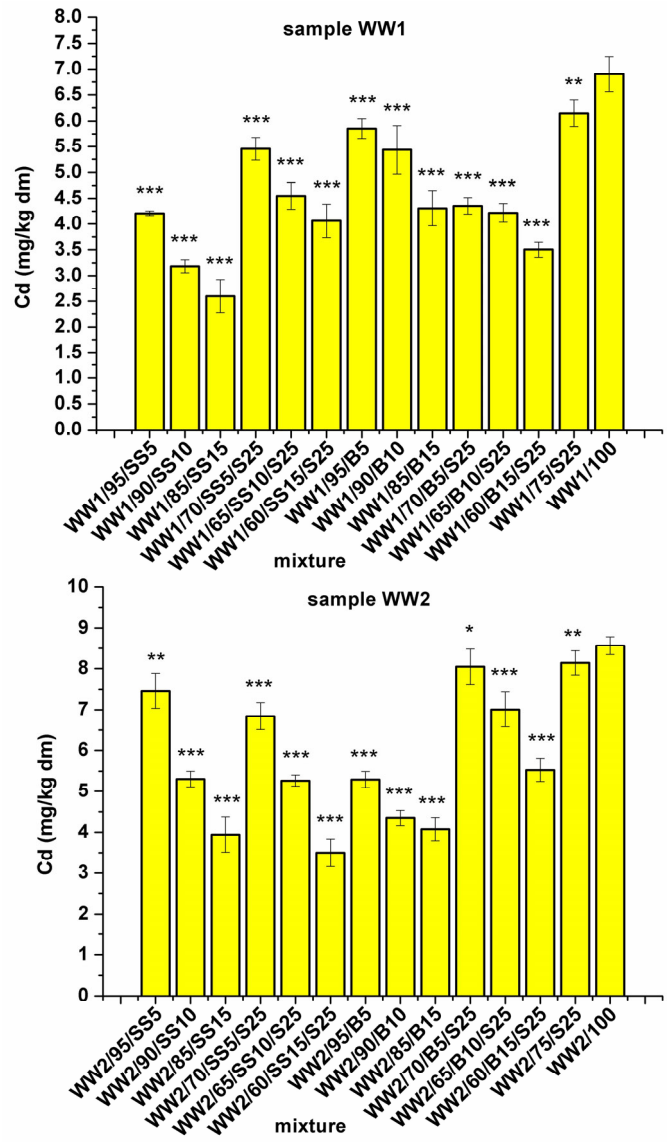


Figure 8. Cd content in biomass grown on substrates containing WW1 and WW2 waste; Dunnett's post-hoc test; * $p < 0.05$; ** $p < 0.01$; *** $p < 0.001$.

All treatments involving sewage sludge, biochar, and sand reduced Cd translocation to the aboveground tissues of the test plant compared with plants grown in unamended waste. Among the amendment treatments, mixtures containing sewage sludge alone resulted in the lowest Cd translocation. As the sewage sludge application rate increased, the cadmium concentration in the harvested biomass decreased for both WW1 and WW2 waste. This finding is supported by the literature. Grobelak et al. [50] demonstrated that cadmium concentrations in plant biomass were lower following sewage sludge application. Reduced cadmium uptake from the substrate may be associated with the presence of organic matter in sewage sludge, which provides numerous functional groups capable of complexing cadmium [51]. For WW1 waste, the addition of sand increased the cadmium concentration in biomass compared with mixtures amended with sewage sludge alone. This may result from a reduction in the immobilisation capacity of sewage sludge following the addition of sand, which has a low sorption capacity and dilutes the organic matter fraction, thereby reducing the number of active sorption sites. This trend was less pronounced for WW2 waste. This waste has a finer particle size distribution than WW1 waste (Figure 3), and the addition of 25% sand alters the physical properties of the substrate by increasing the

proportion of macropores, permeability, and aeration. Under these conditions, cadmium ions present in the soil solution may migrate beyond the plant root zone.

The addition of biochar to WW1 and WW2 waste decreased cadmium concentrations in biomass as the biochar application rate increased (Figure 8). Greater cadmium mobility, reflected in higher cadmium concentrations in biomass, was observed in mixtures containing WW2 waste than in those containing WW1 waste, although WW1 had a higher total Cd concentration than WW2 (Table 4). This may be associated with competition between zinc and cadmium cations, whereby a higher zinc concentration in the substrate reduces the number of sorption sites available to cadmium. The addition of sand to biochar-amended mixtures produced different effects for WW1 and WW2 waste. In the mixture containing WW1 waste, sand decreased the cadmium concentration in biomass, whereas in the mixture containing WW2 waste, it increased the concentration of this metal.

4. Summary and Conclusions

Landfills where waste from the mining and processing of Zn-Pb ores has been accumulated for many years present a particularly challenging case for reclamation, and previous attempts to restore vegetation cover have not yielded lasting results. This waste is characterized by a very low content of organic matter and biogenic elements, a high content of Zn, Pb, and Cd, and unfavorable structural properties resulting from the fine-grained nature of components (Table 4, Figure 3). Therefore, establishing stable vegetation cover is of particular importance for reducing wind erosion and secondary environmental contamination.

The significant heterogeneity of waste deposited within the same landfill (WW1 and WW2 waste), manifested by variations in its properties, is a key factor determining plant growth conditions and, consequently, the effectiveness of bioremediation. The results obtained indicate that the selection of material to support reclamation should take into account the chemical, granulometric, and physical properties of the waste simultaneously, rather than relying solely on the type or dose of the additive used. This confirms the practical value of the research methodology we employed, which aims at selecting conditioning additives that will ensure the establishment of a sustainable plant cover.

To summarize the research results, the following conclusions were drawn:

- The application of sewage sludge and sludge-derived biochar improved the growth conditions for *Festuca arundinacea*, with amendment effectiveness depending on waste properties, such as sewage sludge produced more favourable effects for WW1, whereas biochar was more effective for WW2;
- The maximum biomass increase for waste WW1 was achieved with the WW1/65/SS10/S25 combination, while for waste WW2, it was achieved with the WW2/60/B15/S25 combination;
- Sand applied as the sole amendment did not improve biomass production, and its beneficial effects were mainly evident when it was combined with sewage sludge or biochar, reflecting its role in improving substrate structure;
- The amendments reduced the accumulation of Zn, Pb and Cd in the aboveground parts of the test plant compared with the control substrate (WW1/100 and WW2/100), indicating their potential to reduce the translocation of these metals from the waste to plant shoots.

The study focused on evaluating the effectiveness of the additives used in limiting the accumulation of heavy metals in the above-ground parts of plants, thereby reducing the risk of their further movement in the environment. In the next phase of the research, a speciation analysis of the metals is planned, which will allow for a more in-depth assessment of their mobility and bioavailability, as well as a better understanding of the mechanisms by which the additives interact.

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

Funding: This research was funded by the statute subvention of Czestochowa University of Technology (Faculty of Infrastructure and Environment).

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: The original contributions presented in this study are included in the article. Further inquiries can be directed to the corresponding author.

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

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