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Biochar-Supported Phytoremediation of Dredged Sediments Contaminated by HCH Isomers and Trace Elements Using *Paulownia tomentosa*

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Abstract: The remediation of dredged sediments (DS) as a major waste generation field has become an urgent environmental issue. In response to the limited strategies to restore DS, the current study aimed to investigate the suitability of *Paulownia tomentosa* (Thunb.) Steud as a tool for decontamination of DS, both independently and in combination with a sewage sludge-based biochar. The experimental design included unamended and biochar-supplemented DS with the application rates of 2.5, 5.0, and 10.0%, in which vegetation of *P. tomentosa* was monitored. The results confirmed that the incorporation of biochar enriched DS with the essential plant nutrients (P, Ca, and S), stimulated biomass yield and improved the plant's photosynthetic performance by up to 3.36 and 80.0 times, respectively; the observed effects were correlated with the application rates. In addition, biochar enhanced the phytostabilisation of organic contaminants and shifted the primary accumulation of potentially toxic elements from the aboveground biomass to the roots. In spite of the inspiring results, further research has to concentrate on the investigation of the mechanisms of improvement the plant's development depending on biochar's properties and application rate and studying the biochar's mitigation effects in the explored DS research system.

Keywords: complex contamination; waste valorisation; *Paulownia tomentosa*; biochar dose; circular economy

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1. Introduction

Sediment dredging, an essential process required for water navigation, construction, reclamation, mining, and environment replenishment activities [1], simultaneously leads to significant waste generation [2], nowadays reaching up to 44, 50, 56, 80, 152, and 360 M m^3 y^{-1} in the UK, Germany, France, Brazil, the USA, and India, respectively [3–5]. To reduce the volume of dredged sediments (DS) waste, and in line with the R^6 concept of a circular economy, this waste has to be re-used [6], ensuring the major R^5 functions of DS utilisation [7].

Recent studies have proposed different ways for reusing the waste DS in the construction of buildings and roads (concrete materials, bricks, and ceramics) [3,6,8–10] without any constraints for contamination levels [9]. There are proposals to reuse DS in agriculture for the prevention of soil erosion or as a peat substitution [3,4,11,12]; however, the approaches have implementation's barrier because of the strict request for recycled material in terms of contamination [13–18]. Consequently, although the utilisation of waste sediments in agriculture looks very promising, prior to application, DS have to be significantly cleaned from contaminants of both organic and inorganic origin. Indeed, given the substantial volume of sediments designated for dredging and recycling, estimated in the Czech Republic as 200 M m^3 [19], the further valorisation of DS demands the development of sustainable approaches aimed at their remediation.

The biological cleaning of DS remains underexplored due to their heterogeneous composition and limited oxygen availability [20]. Numerous studies have demonstrated the viability of phytoremediation for the revitalisation of sediments both ex situ and in situ [21–30]. The selection of plants should be based on the phytoremediation potential in relation to the target contaminants and ecological adaptability [31]. *Paulownia tomentosa* (Thunb.) Steud (*P. tomentosa*) is among the viable candidates due to the plant's tolerance to xenobiotics, essential yield achieved even under the non-optimised growth conditions, and ability to accumulate trace elements (TEs) and organochlorine pesticides [32,33]. The application of the widely utilised phytoagent *Miscanthus* × *giganteus* [34] in DS that is complexly contaminated by pesticides and TEs, may be unsuccessful due to earlier reported essential stress plant experiences in heavily pesticide-contaminated soil [35]. The substitution of *Miscanthus* × *giganteus* for *Miscanthus sinensis*, proposed for Kazakhstan [35,36], cannot be implemented in some European countries, including the Czech Republic, where *Miscanthus sinensis* is classified as an invasive crop [37,38]. Despite the approved attractiveness of *Paulownia* sp. biomass for valorisation in biorefineries [39], the investigations covering the scientific framework for utilisation are limited: from 1971 to 2021, only 820 scientific documents were published on the related topics [40]. The studies dedicated to the exploration of *P. tomentosa* as a phytoagent are almost absent, and the existing ones focus on the phytoremediation of TE-contaminated soils [41–45]. Our previous research on the behaviour of *P. tomentosa* in soils complexly contaminated with organochlorine pesticides and TEs was pioneering [33] in terms of the utilisation of this plant for the phytoremediation of complexly contaminated soil. The current study aimed to continue investigation on the potential of *P. tomentosa* to develop within the same contamination nature but in a different substrate. In addition, recent phytoremediation studies give some preference to tree plants since, along with greater yield performance and economically viable valorisation options, they can capture carbon dioxide (CO_2) and release oxygen into the atmosphere more intensively compared to grass phyto-agents [46,47].

According to the integrated phytoremediation–bioenergy strategy [48], suggesting to advance the phytoremediation process by utilising soil amendments, incorporation of biochar into the phytoremediation process will provide an additional value due to its climate change-mitigating potential by increasing C storage and decreasing GHG emissions [49]. Biochar is a C-rich substance produced by the pyrolysis of organic wastes; it boosts crop productivity, improves soil biological and physicochemical properties, and increases the activity of soil microbial communities [50]. Biochar is gaining increasing attention as a novel stabilising agent that immobilises organic contaminants and TEs through direct mechanisms such as electrostatic attraction, ion exchange, complexation, and precipitation [51]. Additionally, it alters contaminant availability through indirect mechanisms, i.e., influencing soil properties, i.e., pH, cation exchange capacity, mineral composition, microbial abundance, and organic carbon content [51,52]. However, the positive effects of biochar depend on factors such as the precursor material, pyrolysis conditions, and application rates [53–55], and the benefits are not always guaranteed [49,56]. Kononchuk et al. [57] found that increasing the biochar application rate does not necessarily improve the biomass yield, while Xu et al. [58] highlighted that biochar may introduce environmental risks into phytoremediated systems, such as nitrate leaching [51] or secondary contamination [55]. Recent studies have shown that biochar incorporation can stimulate a removal efficiency of over 77% for Cu, Zn, and Pb [59]. Moreover, a comparative analysis of slaked lime, phosphogypsum, bone meal, and rice husk-derived biochar for their sorption capacity related to As, Cd, and Pb revealed that biochar had a significantly higher sorption capacity [60]. Thus, despite holding a great potential, biochar remains an underexplored "black gold" that requires further in-depth research and validation across various systems and environmental matrices.

The developing strategy on valorising DS through phytoremediation using the fast-growing timber species *P. tomentosa* aligns with selected Sustainable Development Goals (SDGs) outlined in the 2030 Agenda for Sustainable Development [61]. Specifically, the approach supports SDG 6.6, which focuses on protecting and restoring water-related ecosystems, is in line with SDG 14.a, which requests to strengthen scientific knowledge and research capacity in improving the state of the environment, and SDG 15.3, which targets the restoration of degraded land and soil by 2030 [61]. The proposed strategy fits within SDG 6.6, as the contaminated DS investigated in this study are fluvial sediments. The valorised (remediated) DS are intended for use as peat substitutes in sustainable agriculture, aligning with SDG 15.3. As of 2 May 2024, progress toward pointed SDGs is varied: 35–50% of targets are experiencing stagnation or regression, 30–65% show moderate progress, and 0–20% have been fully met [62]. These results underscore the urgent need to develop sustainable measures and new technological approaches, including those for DS.

The current study aimed to evaluate the phytoremediation potential of *P. tomentosa* in a 'sediment–biochar' system. In the case of positive results, the approach may be proposed for the agriculture sector to receive the cleaned DS to be utilised for improving the soil quality and expanding the land bank.

2. Materials and Methods

2.1. Sediments Collection

The dredged sediments utilised in the experiment were collected manually (by shovel) in Hajek, Czech Republic (GPS 50°17′31.5″ N 12°53′35.2″ E). The sediments were complexly contaminated with trace elements (TEs) and hexachlorocyclohexane (HCH) isomers (α-, β-, γ-, δ-, and ε-). The contamination of the site originated from HCH production since the 1960s, primarily consisting of ballast isomers and chlorobenzenes [63]. Concentrations of HCH isomers and TEs in the DS are presented in Table 1.

Table 1. Initial concentrations of HCH isomers (μg kg^{-1}) and TEs (mg kg^{-1}) in DS and biochar.

Contaminant	MPC	Sediments	MAT	Biochar
α-		18.1 ± 1.53	–	–
β-		102 ± 9.02	–	–
γ-	10.3 [a]	59.6 ± 5.88	–	–
δ-		283 ± 21.5	–	–
ε-		560 ± 41.0	–	–
∑		1023 ± 56.8	–	–
Mg	–	40,321 ± 1546	–	22,147 ± 1783
Al	–	90,957 ± 816	–	42,842 ± 1057
Si	–	197,636 ± 966	–	83,524 ± 979
P	–	11,935 ± 345	–	176,149 ± 6165
S	–	382 ± 30.0	–	10,257 ± 12.5
K	–	12,154 ± 830	–	13,425 ± 613
Ca	–	45,115 ± 944	–	132,669 ± 4541
Ti	–	25,713 ± 510	–	5253 ± 250
Cr	200	459 ± 20.5	–	242 ± 9.00
Mn	–	2888 ± 107	–	4039 ± 220
Fe	–	124,914 ± 468	–	46,049 ± 2481
Cu	100	152 ± 9.50	143–6000	763 ± 37.5
Zn	300	131 ± 7.00	416–7400	3616 ± 287
Rb	–	181 ± 6.00	–	43.6 ± 1.12
Sr	–	400 ± 8.00	400	830 ± 60.0
Zr	–	378 ± 6.00	–	143 ± 2.52
Nb	–	137 ± 4.51	–	–
Pb	100	202 ± 15.0	121–300	103 ± 5.65

Note: MPC—Maximum Permissible Concentrations [13]; MAT—maximum allowable thresholds [64]; [a]—MPC for Benthic community (freshwater sediment) [65].

2.2. Experiment Layout

The planting material was the one-year-old seedlings of *Paulownia tomentosa* (Thunb.) Steud (www.fascinujicipaulownia.cz, accessed on 25 September 2024). DS were amended with 2.5, 5.0, and 10% biochar. The material was produced by Agmeco s.r.o. via pyrolysis at 600–650 °C of sewage sludge from the municipal wastewater treatment plant in Brno, Czech Republic. The results of biochar proximate and ultimate analyses are presented in detail in Pidlisnyuk et al. [66,67]. The research biochar has an increased content of ash (56.5 ± 0.21 wt.%), an alkaline pH (9.34 ± 0.26), and a first carbon (C) storage class with the potential to sequester 190 g C kg^{-1} in soil for 100 years [68]. TEs content in biochar is presented in Table 1. To obtain 2.5, 5, and 10% (w.w) biochar-amended sediments, 1.5, 3.0, and 6.0 kg of biochar, respectively, were added to 58.5, 57.0, and 54.0 kg of sediments (20 kg per pot × 3) and thoroughly mixed 1 h in a concrete mixer. The seedlings were planted in pots containing 20 kg of biochar-amended sediments and kept during the experiment outside (yard of Crop Research Institute, Chomutov, the Czech Republic; GPS 50°27′50.54″ N, 13°22′49.093″ E). The experimental design was as follows:

Unamended sediments + *P. tomentosa*;

A total of 2.5% biochar-amended sediments + *P. tomentosa*;

A total of 5.0% biochar-amended sediments + *P. tomentosa*;

A total of 10% biochar-amended sediments + *P. tomentosa.*

Each variant was conducted in triplicate. The experiment was established on 1 June 2021, and finished on October 18, 2021, when plant biomass was harvested. During the experiment, soil moisture was maintained when necessary. Physiological parameters, i.e., plant height, stem diameter, and leaf length and width, were measured monthly. The chlorophyll *a* fluorescence was analysed using a portable fluorimeter HandyPEA+ (Hansatech Instruments Ltd., Norfolk, UK). The detailed description of fluorescence transient and biophysical parameters examined was provided earlier [69].

2.3. Sample Collection at Harvest

The aboveground biomass (AGB) and roots of *P. tomentosa* were harvested following the ISO 11464:2007 standard [70] and dried in the open air until a constant weight was reached, i.e., when the difference between two consecutively measured weights was within 0.0001 g. Before drying, root samples were thoroughly washed under a tap water. The dry weight (DW) was calculated for leaves, stems, and roots; each sample was separately collected into the labelled plastic zip-lock bag and stored at room temperature until chemical analysis was provided.

2.4. Chemical Analysis of Substrate and Plant Samples

The preparation of DS and plant samples for the chemical analysis was performed following ISO 11465-2001 [71] and 11464:2007 [70]. The procedure was explained in detail earlier [72]. Briefly, the samples of DS were dried at 105 °C to a constant mass; thereafter, they were placed onto a clean sheet of paper and small stones, plant particles, and other inclusions were removed. Bigger clods were ground in a porcelain mortar and mixed with the main part of the DS sample. Then, the thoroughly mixed substrate was put on a piece of clean paper in a square and divided into four equal parts using a spatula. Two opposite parts were removed, and two others were combined, remixed, and used for the analysis. This average sample was additionally sieved (0.25 mm pore size). The preparation of plant samples is described in Section 2.3.

2.4.1. Chemical Analysis of Trace Elements Content

The TE content in the DS and plant samples was analysed using X-ray fluorescence analysis following the United States Environmental Protection Agency [73] standard using an Elvax Light SDD Analyzer (Elvatech, Kyiv, Ukraine). The layout of the analysis was described in detail earlier in Pidlisnyuk et al. [74]. Briefly, plant samples were combusted at 400 °C for 4 h, cooled for 1 h in desiccators, weighed, and processed for the analysis.

The device used can detect chemical elements in a range from ^{11}Na to ^{92}U with a high accuracy (0.01%). The time of data collection was 2 × 180 s for all the samples. The limits of the absolute measuring error were ±0.05–0.20% (with the time of one measurement being 180 s). Three parallel measurements were taken for each sample. The level of TEs in the soil was determined in mg kg^{-1}. The level of TEs in the biomass was determined in mass units in the ash and then recalculated to mg kg^{-1} based on the ash content of the initial plant material. For the overall calculation, the concentration was expressed in mg kg^{-1} DW. In the case of the DS analysis, the samples (~2 g) were placed on ultra-thin (4 µm) polypropylene film (supplied with the device), which is transparent to X-rays, and further accurately transferred to the device, where a measurement was performed. In the case of biomass tissues, the combusted samples (ash) of roots, stems, and leaves (~0.5 g) were placed inside a plastic ring (d = 1.25 cm), which was located on a similar thin polypropylene film, and compacted using a glass rod. The resulting sample was transferred into a device for measurement.

2.4.2. Chemical Analysis of HCH Isomers Content

The concentrations of HCH isomers in the DS and plant samples were determined following the procedure described earlier [63] using two GC-MS assemblies. An RSH/Trace 1310/TSQ 8000 GC-MS array (Thermo Fisher Scientific, Waltham, MA, USA) with a Scion-5MS column (SCION Instruments, Goes, The Netherlands) was used. The limit of quantification (LOQ) was <0.01 µg L^{-1}. Samples were extracted using the headspace SPME technique, either by using a PDMS/DVB fibre with a coating thickness of 100 µm (Supelco, Bellefonte, PA, USA) or by directly injecting the sample in static headspace mode. Prior to extraction, samples were derivatised so that acetylated chlorophenols were formed. An isotopically labelled compound (γ-HCH D_6) was used as an internal GC-MS/MS analysis standard.

2.5. Phytoremediation Coefficients

The phytoremediation potential of *P. tomentosa* in relation to the complexly contaminated DS was evaluated using bioconcentration (BCF) and translocation (TLF) factors and a comprehensive bio-concentration index (CBCI) [75].

The BCF was calculated according to Zayed et al. [76]:

$$BCF = \frac{Contaminant\ concentration\ in\ plant\ tissues\ at\ harvest}{Initial\ contaminant\ concentration\ in\ DSs} \tag{1}$$

The TLF was calculated according to Yanqun et al. [77]:

$$TLF = \frac{Contaminant\ concentration\ in\ aboveground\ biomass}{Contaminant\ concentration\ in\ roots} \tag{2}$$

The CBCI was calculated to evaluate the plant's ability to accumulate multiple TEs according to Zhao et al. [78]:

$$CBCI = \frac{1}{n}\sum_{i=1}^{n} \frac{BCF_i - BCF_{i.min}}{BCF_{i.max} - BCF_{i.min}} \tag{3}$$

where n is the total number of TEs and i is a particular TE.

2.6. Techno–Economic Analysis

To conduct the techno–economic analysis, two contrasting scenarios for plant biomass valorisation were applied. The following parameters were used in both scenarios: (a) the cost of planting materials was CZK 69 per seedling (~EUR 2.72), based on actual costs from a supplier (www.fascinujicipaulownia.cz, accessed on 25 September 2024); (b) labour requirements for farming operations such as ploughing, harrowing, planting, and weed control were 2, 1.29, 23.48, and 1 man h^{-1} ha^{-1}, respectively [79–81]; (c) the labour cost was set at EUR 7.93 h^{-1} [82]; (d) the market price of biochar was EUR 499 t^{-1} [81].

Additionally, the economic model was adapted to the functional unit of 1 m^3 and developed to account for the volume of sediments designated for dredging and recycling, which is estimated at 200 M m^3 in the Czech Republic [19]. Typically, economic or environmental assessments of phytoremediation or plant cultivation are calculated per hectare (ha); therefore, the 200 M m^3 of dredged sediments was converted to hectares, corresponding to approximately 10 ha, assuming a 2 m depth as optimal for a tree root development.

While the cost of sediment dredging typically plays a crucial role in the overall investment, it was excluded from the current model. This decision was made to focus on the value added by biochar as a soil amendment under the assumption that the sediments had already been dredged. In addition, dredging, transport, and disposal are the common steps in contaminated sediment remediation, with associated costs of USD 22.4 m^{-3} (~EUR 20.4 m^{-3}) for non-hopper dredging of contaminated sediments [83], USD 120 h^{-1} (~EUR 109 h^{-1}) for transport [84], and USD 150 m^{-3} (~EUR 137 m^{-3}) for disposal [84]. The total cost of dredging 200 M m^3 of sediments would be EUR 4,080,000 (or EUR 408,000 ha^{-1}). The total cost of disposing of contaminated dredged sediments would be EUR 27,400,000 (or EUR 2,740,000 ha^{-1}). These calculations were used to evaluate the feasibility of the proposed phytoremediation strategy for valorising DS.

2.6.1. Scenario 1: Dual-Use Production for Timber and Woodchips

The first scenario followed the dual-use production approach proposed by Testa et al. [85], focusing on *P. tomentosa* cultivation for high-quality wood biomass production. For this purpose, the planting density should be ~600 plants per hectare (pl ha^{-1}) [86]. Specifically, a planting density of 625 pl ha^{-1} was used, with a 20-year lifecycle, harvesting every 4 years to produce stems with a 25 cm diameter and 6 m length. The harvest cost was estimated at EUR 800 [86]. The market price for timber was set at EUR 140 t^{-1}, and, for wood chips, EUR 80 t^{-1} [85].

2.6.2. Scenario 2: Biomass–Biogas–Electricity Value Chain

The second scenario involved valorising plant biomass through a "biomass–biogas–electricity" value chain [27,81]. For energy purposes, the planting density was increased to 2000–3500 pl ha^{-1} [86]. Specifically, 3000 pl ha^{-1} was used, with a 14-year lifecycle and annual harvests. The harvest cost was set at EUR 600 [86]. The substrate-specific methane yield (SMY) was 194 m^3 (to DM)$^{-1}$ [86]. According to Suhartini et al. [87], 1 m^3 of methane produces 0.036 GJ, and 1 GJ of electricity costs EUR 45.12 [88].

The economic efficiency was determined based on the following equation:

$$Revenue = Production\ value - Total\ production\ cost \quad (4)$$

2.7. Statistical Analysis

Statistical analysis was performed using RStudio software (version 2023.06.0 Build 421, RStudio PBC, 2023). An analysis of variance (ANOVA) was performed to detect statistically significant differences between the examined treatments. If a significant difference was detected by ANOVA, Tukey's HSD test was performed for pairwise comparison. The treatments were categorised according to results of the test (by letters in descending order), and respective boxplots and graphs were produced.

3. Results and Discussion

3.1. Influence of Biochar Incorporation on DS Elemental Composition

Chemical analysis of DS on TEs content revealed that chromium (Cr), copper (Cu), and lead (Pb) concentrations exceeded the MPC defined for sediments by 2.30, 2.08, and 2.02 times, respectively (Tables 1 and S1). Incorporation of biochar at three application rates differently influenced the concentrations of essential for plant development (EEs) and potentially toxic (PTEs) elements. In the case of EEs, the DS were benefited gradually, in terms of P, S, and Ca, with increasing biochar application rates, whereas the concentration of Mg, Al, and Ti in research sediments decreased with the incorporation of biochar (Figure 1).

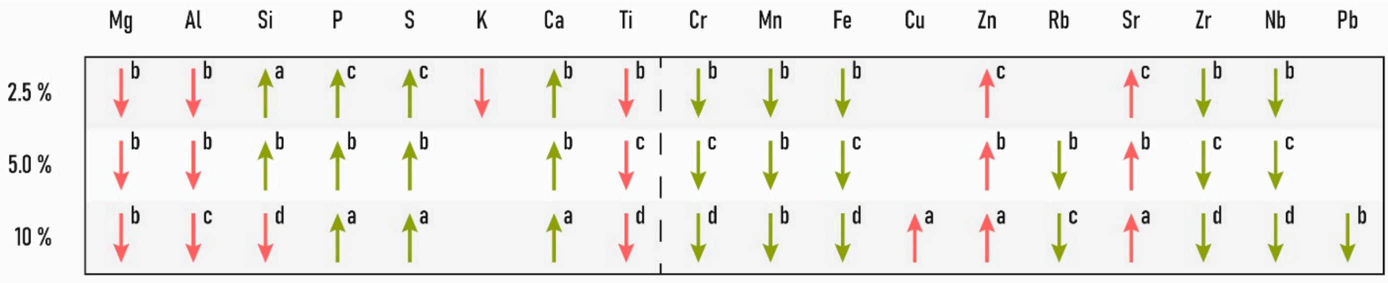

Figure 1. Influence of biochar application rates on sediments elemental composition. Different letters indicate statistically significant difference between treatments within one element. Arrows and letters were placed in relation to control.

Considering PTEs, concentrations of Cr, Mn, Fe, Zr, Rb, Nb, and Pb decreased in biochar-amended sediments, while the content of Cu, Zn, and Sr increased by 29.1, 389, and 27.9% at the highest application rate of 10% (Figure 1). The reduction of PTEs concentrations in DS corresponded to biochar application rates (except for Mn), specifically, up to 47.0, 23.1, 24.4, 24.1, 21.7, 31.9, and 45.5%, respectively. Indeed, biochar affected Cu and Pb contents at the highest application rate only. Furthermore, even at 2.5% biochar, Zn concentration in sediments exceeded the MPC value (Table S1).

3.2. Influence of Biochar Incorporation on Plant Physiological Parameters

Incorporation of biochar at distinct application rates led to a significant increase in all parameters measured (Figure 2). *P. tomentosa* height increased by 45.7–49.8% at 2.5–5.0% biochar application rates, peaking at 10% biochar (by 82.0%). Stem diameter was significantly influenced by 10% biochar only, increasing by 54.4% (Figure 2b). Meanwhile, aboveground biomass (AGB) DW gradually increased with increasing biochar doses by 85.5, 182, and 336%, respectively (Figure 2c). Indeed, stem DW was more sensitive to biochar incorporation with a significant increase even at 2.5% biochar, whereas a significant difference in leaf DW was observed at higher application rates. Root biomass DW increased equally without dependence on biochar application rates by up to 243% (Figure 2d). In the case of leaf length, the biochar effect mirrored the pattern observed in plant height, increasing the parameter by up to 74.1% (Table S2). Considering leaf width, biochar incorporation exerted an impact similar to that observed for stem diameter, with an increase of up to 78.5% (Table S2).

3.3. Influence of Biochar Incorporation on Plant Chlorophyll Fluorescence

To evaluate the structural and functional alterations in photosynthetic apparatus in response to the complex contamination and biochar incorporation, twenty-three OJIP-test parameters were examined (Table S3; Figures 3 and S1). All parameters either gradually increased or decreased with increasing biochar application rates. Biochar incorporation retarded the reaction centre (*RCs*) closure rate (M_0) by inhibiting trapped energy flux (TR_0/RC) and enhancing electron transport (ET_0/RC), which led to an increased probability of moving electrons to the ETC by trapped exciton (ET_0/TR_0). The maximum quantum yield of primary photochemistry (TR_0/ABS or F_V/F_M) and RCs' density per excited cross-section (*CS*) increased with biochar incorporation, whereas the photon flux absorbed by chlorophylls (*ABS*/*RC*) decreased. However, overall absorption-based performance (PI_{ABS}) essentially increased by up to 80 times.

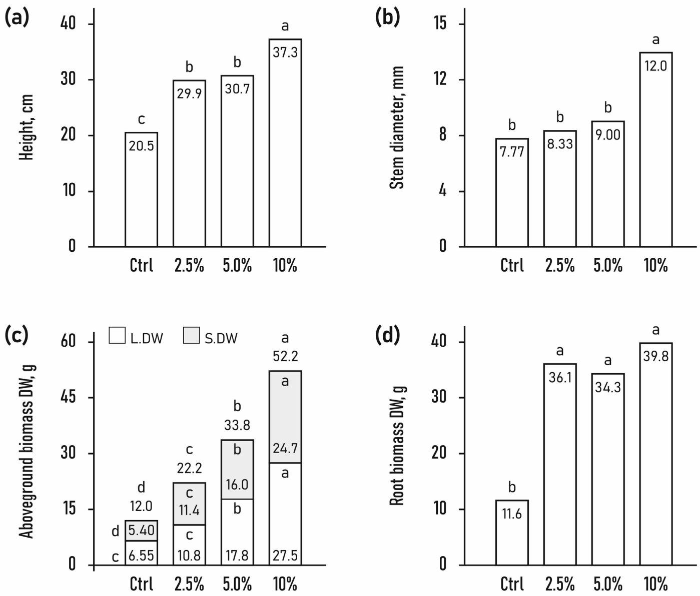

Figure 2. Influence of biochar incorporation on *P. tomentosa* physiological parameters. (**a**) Plant height; (**b**) stem diameter; (**c**) aboveground biomass DW; (**d**) root biomass DW. Different letters within the parameter indicate a statistically significant difference ($p < 0.05$).

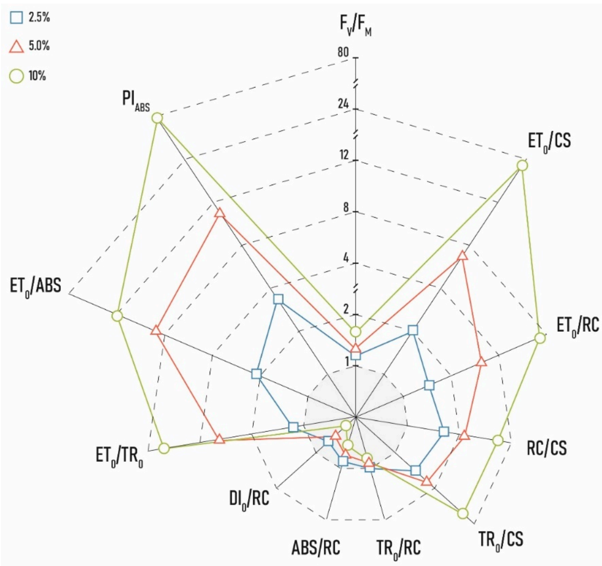

Figure 3. Specific and phenomenological parameters of chlorophyll fluorescence of *P. tomentosa* grown in biochar-amended sediments. Presented values normalised referring to control. Note: *ABS*—absorption; *RC*—reaction centre; *TR*—trapped energy; *ET*—electron transport; *DI*—dissipation; *CS*—cross-section; *PI*—performance index [89,90].

3.4. Influence of Biochar Incorporation on Plant Phytoremediation Potential

Analysing the uptake of HCH isomers by *P. tomentosa*, noticeable accumulation was solely observed in the plant roots (Figure 4). Furthermore, β- and δ-isomers were only found in plant roots among the five isomers detected in the DS. In the 5.0 and 10% biochar-amended sediments, the root BCFs for β- and δ-isomers increased by up to 119%.

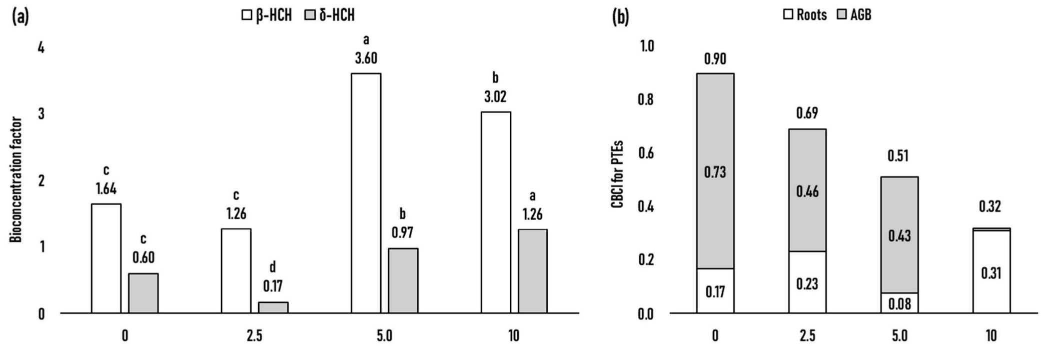

Figure 4. Phytoremediation potential of *P. tomentosa* in relation to HCH isomers (**a**) and PTEs (**b**). Different letters within the parameter indicate a statistically significant difference ($p < 0.05$).

Analysing the phytoremediation potential of *P. tomentosa* concerning elements in DS revealed distinct accumulation trends as biochar application rates rose (Table S4): Mg, Al, Si, Ti, Cr, Mn, Fe, Sr, Zr, and Pb decreased in AGB but increased in roots; P, S, Ca, and Zn decreased in both plant tissues; K increased in both plant tissues; Cu and Rb increased in AGB till 5.0% biochar with a subsequent decrease at 10% biochar.

Hence, upon evaluating the CBCI values separately for EEs and PTEs, it is evident that biochar incorporation into sediments prompted a shift in the primary accumulation site from AGB to roots, resulting in an overall decrease in TE accumulation within *P. tomentosa* (Figures 4 and S2). Specifically, a notable reduction in EE accumulation occurred in both AGB (0.81–0.40) and roots (0.32–0.09) when plants were grown in 0–5.0% biochar-amended sediments. However, in 10% biochar-amended sediments, EE accumulation in roots increased to 0.45 (Figure S2).

Unexpectedly, at 2.5% biochar, the translocation of S and K to AGB increased, while Ca, Zn, and Pb translocation remained unchanged (Figure S3). However, in 5.0% biochar-amended sediments, the translocation of P, S, K, Ca, Cu, Zn, Rb, Zr, and Pb to AGB increased, while Mg, Al, and Mn translocation remained constant. Interestingly, in sediments with 10% biochar, translocation decreased for all elements except K and Pb.

3.5. Principal Component Analysis

In order to investigate the interconnection between varied applied treatments and plant physiology, morphology, and phytoremediation potential, a correlation within physiological, morphological, and phytoremediation parameters was estimated (Tables S5–S7), and biplots of PCA were plotted (Figure 5). The first principal component (PC1) and second principal component (PC2) accounted for 55.6 and 30.8% of the data variance, respectively, covering 86.4% of data variability. The control treatment located on the left side is separated from the biochar-amended treatments by PC1 (Figure 5a). At the same time, the control and 10% biochar-amended treatments are also differentiated from the remaining biochar-amended treatments by PC2.

PC1 was mainly contributed by Zn and S accumulation in the roots, P accumulation in the aboveground biomass, plant root biomass, and stress indicator (Fv/Fm). It can be concluded that the incorporation of sludge-based biochar into contaminated DS positively affects plant biomass productivity, eliminating the negative impact of complex contamination. Furthermore, since Cr accumulation in roots, Cu, Mg, and K accumulation in the aboveground biomass, and plant stem diameter are the main contributors to PC2, lower application rates (2.5 and 5%) can be considered more suitable for application in *P. tomentosa* phytoremediation of DS as it permitted them to avoid Cr accumulation in plant roots.

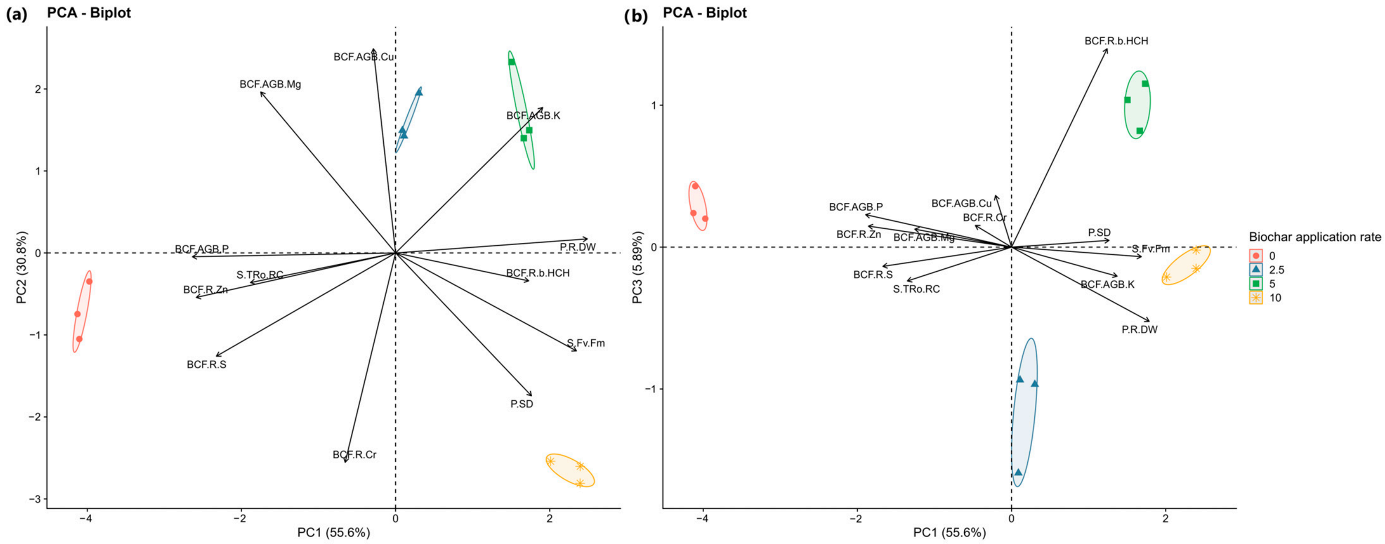

Figure 5. PCA biplots: (**a**) PC1-PC2; (**b**) PC1-PC3.

3.6. Techno–Economic Analysis

In the establishment year, total labour requirements and costs for ploughing, harrowing, planting, and weed control were 29.8 man h^{-1} ha^{-1} and EUR 1036 ha^{-1} for the unamended system, and 53.3 man h^{-1} ha^{-1} and EUR 1222 ha^{-1} for the biochar-amended system (Table 2). Diesel consumption and costs were 50.8 L ha^{-1} and EUR 75.2 ha^{-1} for all experimental treatments. Similar figures were obtained for scenario 2 except labour cost, specifically EUR 836 ha^{-1} for the unamended system and EUR 1022 ha^{-1} for the biochar-amended system (Table 3). For the second and subsequent years, total labour requirements, labour costs, and diesel usage across all experimental treatments were 3 man h^{-1} ha^{-1}, EUR 824 (or 624) ha^{-1}, 22.5 L ha^{-1}, and EUR 33.3 ha^{-1}, respectively.

Table 2. The economic costs of valorising *P. tomentosa* biomass via dual-use production for timber and woodchips (Scenario 1).

Operation		Labour	Diesel	Materials	Total Inputs	
		EUR ha^{-1}	**EUR ha^{-1}**	**EUR ha^{-1}**	**EUR ha^{-1}**	**EUR 200 M m^{-3}**
Tillage	Ploughing	15.9	37.0	–	52.9	529
	Harrowing	10.2	4.93	–	15.2	152
Soil amending	Ctrl	–	–	–	–	–
	Biochar 2.5%	186	–	156	342	3421
	Biochar 5.0%	186	–	312	498	4981
	Biochar 10%	186	–	624	810	8099
Planting		186	–	1700	1886	18,862
Weed control		23.8	33.3	74.5	132	1316
Harvest		–	–	–	800	8000
Total investments						
1st year	Ctrl	236	75.2	1775	2086	20,858
	Biochar 2.5%	422	75.2	1930	2428	24,279
	Biochar 5.0%	422	75.2	2086	2584	25,839
	Biochar 10%	422	75.2	2398	2896	28,958
Following years	No harvest	23.8	33.3	74.5	132	1316
	Harvest	23.8	33.3	74.5	932	9316

Table 3. The economic costs of valorising *P. tomentosa* biomass via the "biomass–biogas–electricity" value chain (Scenario 2).

Operation		Labour	Diesel	Materials	Total Inputs	
		EUR ha^{-1}	EUR ha^{-1}	EUR ha^{-1}	EUR ha^{-1}	EUR 200 M m^{-3}
Tillage	Ploughing	15.9	37.0	–	52.9	529
	Harrowing	10.2	4.93	–	15.2	152
Soil amending	Ctrl	–	–	–	–	–
	Biochar 2.5%	186	–	749	935	9347
	Biochar 5.0%	186	–	1497	1683	16,832
	Biochar 10%	186	–	2994	3180	31,802
Planting		186	–	8160	8346	83,462
Weed control		23.8	33.3	74.5	132	1316
Harvest		–	–	–	600	6000
Total investments						
1st year	Ctrl	236	75.2	8235	8546	85,458
	Biochar 2.5%	422	75.2	8983	9481	94,805
	Biochar 5.0%	422	75.2	9732	10,229	102,290
	Biochar 10%	422	75.2	11,229	11,726	117,260
2nd and following years		23.8	33.3	74.5	732	7316

According to the results presented in Tables 2 and 3, valorisation of the contaminated DS through phytoremediation holds economic preference compared to disposal, which incurs a total cost of EUR 27,400,000 200 M m^{-3} (or EUR 2740,000 ha^{-1}). In both scenarios for the establishment year, the largest share of the investment was attributed to the cost of plant materials. However, in Scenario 1, the share of plant materials ranged from 58.7 to 81.5% (Table 2), while, in Scenario 2, the share was higher, ranging from 69.6 to 95.5%, which can be explained by the higher planting density (3000 pl ha^{-1}) [91]. Moreover, the data obtained for Scenario 2 align with the results of the economic profitability analysis of cultivating *Miscanthus* × *giganteus* in non-agricultural soils [81]. Regarding the cost of biochar amendments at different application rates, in Scenario 1, the share of costs was 6.43, 12.1, and 21.6% for 2.5, 5.0, and 10% application rates, respectively (Table 2). In Scenario 2, the corresponding values were 7.90, 14.6, and 26.6% (Table 3).

The economic revenue for both scenarios was estimated conservatively without factoring in expected biomass yield growth over the plantation's lifecycle. This approach was used to confirm that the proposed valorisation strategy is viable and merits deeper investigation. The preliminary findings indicated that (i) phytoremediation of contaminated DS by *P. tomentosa* without biochar does not achieve a positive return within the 20-year lifecycle of Scenario 1 or the 14-year lifecycle of Scenario 2; (ii) Scenario 1 is less efficient than Scenario 2, as only the 5.0 and 10% biochar treatments showed positive revenue starting from the 8th and 12th years (or the 2nd and 3rd harvests), respectively (Figure 6). However, only 10% biochar application rate reaches the point of breaking-even within the 20-year lifecycle; (iii) valorising post-phytoremediation *P. tomentosa* biomass via Scenario 2 allows to determine the optimal biochar application rate to be 5% since its pay-off period is only 1 year later than the 10% biochar application rate (Figure 7). In addition, valorisation of *P. tomentosa* biomass grown in 2.5, 5.0, and 10% biochar-amended DS through the biomass–biogas–electricity value chain will generate total revenues of EUR 26,556 ha^{-1}, EUR 49,610 ha^{-1}, and EUR 85,869 ha^{-1}, respectively, over the 14-year lifecycle (Figure 7).

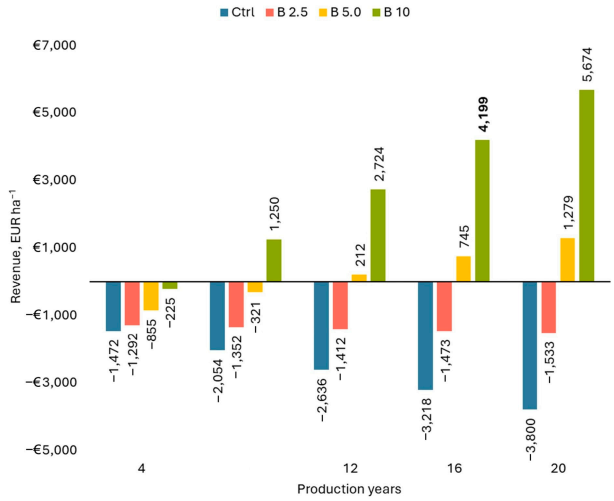

Figure 6. Revenue of valorising *P. tomentosa* biomass produced during phytoremediation of contaminated DS via dual-use production for timber and woodchips (Scenario 1). Figures marked in bold indicate the pay-off moment.

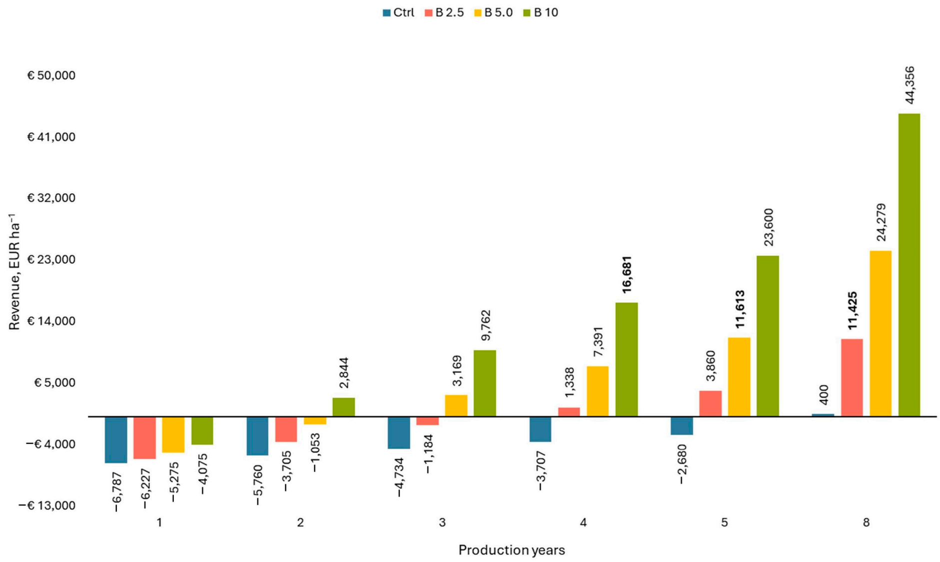

Figure 7. Revenue of valorising *P. tomentosa* biomass produced during phytoremediation of contaminated DS via biomass–biogas–electricity value chain (Scenario 2). Figures marked in bold indicate the pay-off moment.

4. Discussion

4.1. Biochar–Sediment Interplay

The study findings revealed that amending DS with sewage sludge-based biochar supplemented DS with P, S, and Ca. Similar patterns considering P content were observed when the studied biochar was applied to the petroleum hydrocarbon-spiked soils at application rates of 3.5 and 7%; specifically, biochar incorporation influenced the increase in P content in soil by 79.3–116 and 26.2–34.5% irrespective of application rates for soils spiked with 10 and 20 g PHs kg^{-1}, respectively [66]. Whereas, in our study, an increase in P content was linked to biochar application rates, in particular, an increase from 121 to 419%. Despite the assumption that the remediation of DS (ex situ) is closely linked to the remediation of PTE-contaminated soils [92], this may not be entirely accurate, as evidenced by the differing behaviour of the same biochar in DS compared to soil. Indeed, the existing research has focused on changes in the proportions of metal fractions [93–95], making it challenging to compare the specific impact of biochars on the content of EEs in DS.

Cd, Cu, Zn, and Pb are the most commonly detected PTEs in DS [94]. Consequently, the majority of studies on the biochar-supported remediation of DS have concentrated on these PTEs, and only a few studies have explored phytoremediation with another pollutants. For instance, Wang et al. [93] investigated the potential of biochar derived from palm sawdust (at 550 °C), at the same application rates as in our study, for the remediation of Cu- and Pb-spiked sediments, with concentrations of 631–644 and 725–731 mg kg^{-1}, respectively. After 60 days, the bioavailability of Cu and Pb in 10% biochar-amended sediments significantly decreased, while the residual fraction of PTEs increased by up to 87.7%. This phenomenon can be attributed to the highly alkaline nature of the biochar they used (pH of 11.7) in contrast to the biochar investigated in the current study (pH of 9.34) [93], which reduced the availability of hydrogen cations in the DS, thereby promoting the binding of metal ions to ligands and lowering their concentrations [95]. Another study tested biochar produced from *Phyllostachys pubescens* biomass (at 600 °C) at various application rates (0.5–15%) in sediments contaminated by Cr, Cu, Zn, and Pb with concentrations of 287, 135, 766, and 44.3 mg kg^{-1}, respectively, [96]. The incorporation of biochar into DS at application rates higher than 5% resulted in a reduction of the bioavailable fraction of Cu, Zn, and Pb by up to 79.7, 49.8, and 73.2%, respectively, while having no effect on Cr bioavailability. Additionally, the leachability of Zn and Pb decreased, starting at a 3% biochar application rate, and, for Cu, the reduction was observed starting at 5% [96].

Zhang et al. [30] investigated the phytoremediation of DS contaminated with Cd (10.9 mg kg^{-1}), Zn (510 mg kg^{-1}), and Pb (83.6 mg kg^{-1}) using *Lolium perenne* in the presence of corn straw-derived biochar (at 300 °C) at an application rate of 3%. The incorporation of biochar to DS had minimal effect on Zn fractions, whereas the reducible fraction of Pb decreased, and the residual fraction increased by 7.31% and 8.60%, respectively. These findings contrast with the results of the current study, particularly for Zn, which could be explained by the different feedstock used for biochar production. Indeed, biochar produced from the sewage sludge typically contains higher concentrations of PTEs compared to plant-based biochar, leading to greater PTE bioavailability [94]. In our study, the Zn concentration in the biochar was 3616 ± 287 mg kg^{-1} (Table 1). Additionally, pyrolysis temperature plays a significant role in the bioavailability of PTEs in the presence of biochar. The biochar investigated in the current study was produced at 600–650 °C, whereas the corn straw-derived biochar was produced at 300 °C [30]. Increasing the pyrolysis temperature from 350 to 600 °C led to an increase in Cu, Zn, and Pb concentrations in sludge-based biochar by 18.2, 25.0, and 33.3%, respectively [97], likely due to the decomposition of organic matter in the feedstock [98].

Thus, applying biochar in the phytoremediation of DS is considered a viable option to create more favourable conditions for plant growth and mitigate contamination-induced abiotic stress by supplying nutrient elements and sequestering or transforming potentially toxic elements into non-available forms. In this regard, it is essential to continue investigating biochar's role in the remediation of DS, taking into consideration the precursor materials, pyrolysis conditions, and application rates.

4.2. Plant Morpho–Physiology Influenced by Biochar

When *P. tomentosa* was grown in unamended post-military soil complexly contaminated with diesel (1 g kg^{-1}) and PTEs, plant height was higher by 51.2% compared to our results (control), while stem diameter was lower by 42.3% [99]. However, when the same biochar was incorporated into the soil at a 5% rate, plant height decreased by 12.9%, contrasting with the results of the current study [99]. Biochar produced from *Kandelia obovata* biomass at 600 °C was applied to PAH-spiked DS at 1% application rate, resulting in a 46.7% increase in *K. obovate* biomass DW, aligning with our results [25]. A mixture of olive and hazelnut biochars applied to Cu- and Zn-contaminated DS significantly improved the biomass of *Tamarix gallica* and *Carex acutiformis*, with the optimal application rate being 3% [100]. A notable increase in leaf (46%), stem (33%), and root (18%) biomass of ramie grown in DS contaminated by Cd, Cr, Cu, and Pb was observed with biochar application rates of 0.05–0.1, 0.05, and 0.01%, respectively, using biochar derived from tea waste at 300 °C [22]. 3% corn straw-derived biochar applied to Cd-, Zn-, and Pb-contaminated DS alone had no significant effect on *L. perenne* biomass but did improve root length by 13.5% compared to the control [30]. When combined with fulvic acid, the biochar increased both the stem and root biomass by 16.9 and 17.1%, respectively [30].

Due to the widespread use of F_V/F_M as a sensitive indicator of PS performance [101], our data were compared with the published findings. Under PTE contamination—specifically, Cd, Zn, and Pb—the PS performance of *P. tomentosa* initially exhibited lower F_V/F_M (0.74–0.78). However, after four weeks, it increased to 0.81–0.82, indicating plant tolerance to these PTEs [41]. In another study [102], under the favourable conditions (using commercial peat-moss substratum), *Paulownia* spp. exhibited F_V/F_M values of 0.74–0.75. Thus, a trend observed in the current study of increasing F_V/F_M values in biochar-amended sediments (from 0.45 to 0.75) validates the assertion that biochar incorporation significantly improves *P. tomentosa* development while mitigating the phytotoxicity of xenobiotics.

4.3. Influence of Biochar on P. tomentosa Phytoremediation Potential

Published studies on *Paulownia* sp. phytoremediation of soils contaminated with organochlorine pesticides are limited, while investigations employing *Paulownia* sp. for the remediation of DS are almost not presented. Moreover, existing research has primarily focused on spiked soils; in contrast, in the current study, the historically contaminated DS were examined. Our previous study examining the phytoremediation potential of *P. tomentosa* grown in soil complexly contaminated by 24 organochlorine pesticides and 8 PTEs, with γ-HCH and other isomers concentrations of 76.4 and 600 µg kg^{-1}, respectively, reported different behaviour in relation to HCH isomers [33]. In particular, the plant BCF values of γ-HCH for aboveground biomass and roots were 0.4 and 0.2, respectively [33].

As for PTEs, it was decided to compare the phytoremediation potential of *P. tomentosa* with findings obtained in a similar system (DS) amended with biochar. For instance, amending DSs with a mixture of olive and hazelnut biochars had different impacts on Cu and Zn accumulation in leaves of *Tamarix gallica* and *Carex acutiformis*; specifically, it gradually increased Zn accumulation in *Carex acutiformis* leaves with increasing biochar application rates of 3 to 10% and decreased Cu and Zn accumulation in leaves of *Tamarix gallica* starting from 6 and 3% application rates, respectively [100]. The incorporation of 3% corn straw-derived biochar into DS alone did not influence Zn accumulation either in the stem or roots of *L. perenne*, whereas, in combination with fulvic acid, Zn accumulation in both plant parts increased by 30.0 and 70.2%, respectively [30]. Pb accumulation decreased in the stems and roots of *L. perenne* by 39.1–62.1 and 40.2–59.9%, respectively, in the presence of biochar alone or when combined with fluvic acid [30].

4.4. Research Highlights and Future Implications

The study results suggest that *Paulownia* can be an optimal candidate for transformation of the contaminated DS into legislation-compliant peat substitutes. This potential is supported by the plant's ability to accumulate 41 out of 67 elements, including 8 noble metals and 16 rare earth elements, from flotation tailings, which can be classified as "man-made sediments" [103]. Recent studies have highlighted the growing interest in cultivating *Paulownia* trees in European countries, positioning them as a strong alternative to native tree species [104,105]. It is anticipated that the profitability of growing *Paulownia* will become apparent soon, given its multi-purpose biomass utilised as an industrial raw material, i.e., pulp, pencils, crayons, energy production, paper, nanocellulose, biochar, biopolymers, wood plastics, and composites [85,86,104]. In light of Europe's goal to become the first "climate-neutral" continent by 2050 [106], the region is encouraged to cultivate fast-growing lignocellulosic species, such as *Populus*, *Eucalyptus*, *Salix*, *Robinia*, and *Paulownia*, as short rotation coppice species capable of thriving on marginal and abandoned lands [105]. A techno–economic analysis of *Paulownia* dual-use (timber and woodchip) production in

Southern Italy showed an annual gross margin of EUR 358 ha^{-1}, compared to EUR 237 ha^{-1} for wine grapes [85], further confirming the profitability of *Paulownia* cultivation.

In addition to its economic viability, *Paulownia* has been proposed as a sustainable tool for CO_2 mitigation. Ghazzawy et al. [107] reported that planting 1.5 million *Paulownia* trees on 2400 ha could capture 1.04 Mt of atmospheric CO_2 over 10 years while producing 568,301 t of biomass. Considering an investment cost of USD 1128 billion and the marketing value of the wood produced over 10 years (USD 1137 billion), the economic analysis suggests that *Paulownia* trees could serve as a highly profitable, large-scale CO_2 capture tool with the potential to contribute to global climate goals by 2050 [106,107]. Therefore, *Paulownia* should be considered a promising phytoagent for the ex situ remediation of contaminated sediments.

The urgent need to restore DS was further highlighted by Soleimani et al. [27] during the evaluation of the economic and environmental life cycle assessment (LCA) of phytoremediation of chloride-contaminated DS using *Arundo donax*. The biomass was valorised through the Combined Heat and Power value chain. The study found that the costs and environmental impact of the phytoremediation of 1 m^3 of DS, whether conducted alone or integrated with biomass valorisation, were significantly lower than those of sediment landfilling. While the LCA model tested scenarios with no additives, mycorrhiza, humic acid, and combinations of these additives, the incorporation of biochar could further reduce costs and shorten the revenue time, making the approach even more cost-effective than conventional sediment management methods [27,81].

Biochar has proven to be a valuable addition to the phytoremediation system [28,29,98,108,109]. Applying biochar derived from *Miscanthus* × *giganteus* (600 °C) at a 5% application rate improved the dry weight of stems and the leaves-to-stem ratio in *Paulownia* plants grown in flotation tailings during the first year of vegetation [103]. In line with the circular economy concept, i.e., closing the economic loop of DS phytoremediation, post-phytoremediation *Paulownia* biomass can be converted into biochar and reintegrated into the phytoremediation process. *Paulownia*-derived biochar, produced at 700–800 °C, has shown promising results when applied to contaminated soil at 2, 4, and 6% application rates. At the highest dose, it reduced the acid-soluble fraction of Cu by 35.0% and decreased the Fe/Mn oxide fraction of Pb by 9.85 and 8.37% at 2 and 6% application rates, respectively [110]. Additionally, it increased the organic-bound fraction of Pb by 61.3% at the 6% application rate and doubled the organic-bound fraction of Cd at both the 4 and 6% application rates [110].

In conclusion, the suitability of *Paulownia* for the phytoremediation of the contaminated DS is well supported by plant's ability to accumulate a wide range of elements, including rare and noble metals. The incorporation of biochar into the system enhances remediation efficiency and accelerates economic viability, aligning with both circular economy principles and global climate goals. Thus, phytoremediation using *Paulownia* supported by biochar presents a viable and economically profitable approach for the ex situ remediation of contaminated sediments.

Supplementary Materials: The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/su16209080/s1, Figure S1: Experimental parameters of chlorophyll fluorescence of *P. tomentosa* grown in biochar-amended sediments. Presented values normalised referring to control; Figure S2: Comprehensive bio-concentration index representing overall ability of *P. tomentosa* to accumulate EEs in biochar-amended sediments; Figure S3: Translocation factor values of *P. tomentosa* grown in biochar-amended sediments; Table S1: Elemental composition of sediments amended with different doses of biochar (mg kg^{-1}); Table S2: Influence of biochar incorporation on leaf length and width (cm); Table S3: The experimental, specific, and phenomenological parameters of chlorophyll fluorescence of *P. tomentosa* grown in biochar-amended sediments. Numbers in brackets are the normalised values relative to the control; Table S4: BCF values calculated for *P. tomentosa* grown in biochar-amended sediments; Table S5: Correlation matrix for plant physiological parameters; Table S6: Correlation matrix for plant chlorophyll fluorescence indicators; Table S7: Correlation matrix for bioconcentration factor values of aboveground biomass.

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