



Article Rehabilitation of Disturbed Lands with Industrial Wastewater Sludge

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Abstract: Wastelands of the mining industry are among the largest of disturbed areas that demand revitalization. To reduce environmental impact and to better manage these geo-resources, the formation of sustainable plant and soil complexes and the restoration of self-recovery soil function are critical points. The successful return of vegetative cover at post-mining sites requires eliminating the deficiency of organic matter. For this, we assessed the usability of non-traditional ameliorants to provide a better understanding of benefits from mutual dependencies of environmental resources. To prevent losses and to close resource cycles, we studied the applicability of wastewater sludge from the pulp and paper (SPP) industry as an amendment to counteract soil degradation and rehabilitate human-disturbed lands. Waste rock limestone, beresite, and phosphogypsum substrates of postmining sites were used in vitro for the application of sludge and peat mixture and consequent grass seeding. The formed vegetative cover was analyzed to compare the germination and biomass growth on reconstructed soils. We assessed the efficiency of ameliorant combinations by two approaches: (1) the traditional technique of cutting-off plant material to measure the obtained plant biomass, and (2) digital image analysis for RGB-processed photographs of the vegetative cover ($r^2 = 0.75-0.95$). The effect of SPP on plant cover biomass and grass height showed similar results: land rehabilitation with the formation of a 20 cm soil layer on mine waste dumps was environmentally suitable with an SPP:soil ratio of 1:3. However, excessive application (ratio 1:1 of SPP to the soil) negatively affected seed germination and plant vegetation.

Keywords: land revitalization; post-mining development; sustainable land-use management; resource nexus; waste recycling; soil restoration; biomass production

1. Introduction

1.1. Rehabilitation of Post-Mining Areas

Post-mining sites are classified as technogenically disturbed lands due to the impossibility of using them in accordance with their economic and administrative purposes, a high degree of land degradation, and the adverse environmental effects of wind deflation and water erosion [1,2].

Major impacts arise from disturbed areas of mining waste dumps [3]. These sites are characterized by complex landscape damage [4,5], geochemical transformation, and physical disruption of soils. The local environmental situation can be improved by sustainable land-use management through actions of rehabilitation and (or) conservation [6,7]. A mix of engineering and biological work allows the formation of a sustainable soil–plant complex [8,9] and further phytostabilization [10].

According to the FAO World Reference Base for Soil Resources, the studied soils are classified as technosols, as such, their technical origin prevails over their properties



Citation: Petrova, T.A.; Rudzisha, E.; Alekseenko, A.V.; Bech, J.; Pashkevich, M.A. Rehabilitation of Disturbed Lands with Industrial Wastewater Sludge. *Minerals* **2022**, *12*, 376. https://doi.org/10.3390/ min12030376

Academic Editor: Maja Radziemska

Received: 1 March 2022 Accepted: 15 March 2022 Published: 18 March 2022

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Copyright: © 2022 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). and pedogenesis. The successful development of vegetative cover on these soils requires elimination of the organic matter deficiency [8,11,12]. A restored balance of the mineral and organic components improves the structure of technosols [13–16], optimizes soil conditions [17,18], and provides local biocenosis with nutrients [19–21].

1.2. Wastewater Sludge as a Potential Ameliorant

The proper selection of ameliorants is one of the decisive points in the rehabilitation of post-mining lands: inexpensive organic amendments with a prolonged effect are the priority. In this aspect, sewage sludge (SS) is being studied as a non-traditional ameliorant for the reclamation of human-disturbed lands [22].

Sewage sludge is derived as a residue product from the biological stage of wastewater treatment, and in this way, SS may be characterized by a varied range of products of microorganism vital activity. Sewage sludge contains high concentrations of organic matter and numerous nutrients, including nitrogen and phosphorus. This makes SS a potentially inexpensive organic ameliorant for land rehabilitation [23–25].

The high content of P makes it possible to classify SS as a phosphorus ameliorant. The shares of plant-available P [20,26,27] and N rises [28,29] in soils treated with SS. The high amount of organic matter in the sludge improves aggregate stability, which positively affects the physical characteristics of the soil in terms of its water-holding capacity [20], density, and erosion resistance [14,28,29]. On the other hand, higher levels of several other nutrients, K, Ca, Mg, and Na [20,29], and of metals, Cu, Zn, Pb, Mn, Cr, and Cd [30–33], are also noted in treated soil.

Due to the raised contents of metals and the ecological risk of their leaching, migration, and accumulation, it is necessary to consider the pH of treated soil [34,35]. The pH of SS is mainly determined to be in the range of 6.5–7.5 [20,36–38]. However, the introduction of sludge does not equally affect the acidity and electrical conductivity of the soil [29,39]. Research results indicate both an increase [30,40] and a decrease [24,41] in soil acidity after adding SS.

The application of optimal sludge doses (no more than 15–45%), improves vegetative cover [37,42,43], stimulates biomass production [20,43,44], and positively affects the rate of plant growth [20,30,37,43,45]. However, opposite results may also be achieved. In [27,46,47], excessive SS application has led to plant growth inhibition, which could be due to the phytotoxicity threshold of the sludge being reached.

Differences in the impact of SS on soil–plant complexes can be explained by the heterogeneity of the sludge compositions. The chemical composition and physicochemical properties of the sludge can vary depending on the wastewater itself, the treatment system, and the sludge processing [31,48].

Municipal sewage sludge (MSS) is actively used as a soil additive in agriculture and forestry, land restoration, and reclamation of infertile soil. Even though MSS contains waste products of microorganism activity, mainly attributed to low hazardous substances (e.g., classification of the Russian Federal Law No. 89-FZ, 'On Production and Consumption Waste'), the sludge can contain a significant amount of toxic inorganic and organic compounds, dangerous pathogens, and high concentrations of metals [20,31,42,49]. To prevent soil contamination, SS is processed by stabilization and disinfection, and assessed for compliance with the regulations. Currently, regulation is mainly carried out in terms of general characteristics: chemical composition, the content of metals, and quantity of pathogens (according to GOST R 54534-2011, GOST R 17.4.3.07-2001, and GOST R 54651-2011).

Due to differences in the chemical composition and physicochemical properties of wastewater sludge of industrial origin, their distinct assessment of applicability is required [50]. This study, as a solution, proposes to use wastewater sludge from pulp and paper mills (SPP) for the replenishment of organic matter and nutrients to reclaim dumps. Seven of the ten largest PPMs of the Russian Federation (Figure 1) are in the Northwestern Federal District of the Russian Federation. The production rate accounts for more than three thousand tons of dry SPP per year (without dehydration, the moisture content of

SPP is over 80–90%). In the same district, 123.3 thousand hectares of disturbed area needs remediation (according to the state report 'On the State and Protection of the Environment of the Russian Federation in 2017').





The composition and physicochemical characteristics of SPP indicate their amelioration potential. There is a high content of organic matter, phosphorus, nitrogen, and nutritious macro and microcomponents (such as Ca, Fe, and Mn), as in MSS [51]. However, there are also results with low levels of nutrients [52], that confirm the need for each sludge to be assessed.

Wastewater sludge from pulp and paper mills (SPP) and MSS differ in the presence of impurities of lignin and cellulose fiber [53], and increased C: N ratio, which can be an obstacle for available nitrogen [54]. Concerning the cultivated soil, SPP improves the water-holding capacity [54], and the presence of fiber improves the structure of the soil and reduces the effect of water erosion [55]. That makes SPP a potentially inexpensive organic ameliorant.

Therefore, the main aim of this work is to evaluate the efficiency of SPP as a soil amendment for disturbed post-mining areas, with the following goals: (1) determination of SPP and optimal soil composition for wasteland reclamation, and, (2) evaluation of the growth efficiency and plant cover formation on reclaimed layers of soils and composition of mine waste.

1.3. Assessment of Suitability of Non-Traditional Ameliorant

The assessment of the applicability of non-traditional ameliorants considers two issues: (1) amelioration potential, and, (2) environmental safety of the substrate.

The evaluation of ameliorants is based on two method paths: direct and indirect assessment of the substrate. Direct assessment consists of analyzing the chemical composition and physicochemical characteristics of the ameliorant and their compliance with the regulated norms. Indirect methods imply an evaluation of an ameliorant through an assessment of the impact on: (1) plants—analysis of plant growth and vegetation [56–58], and, (2) soil organisms—the qualitative and quantitative composition of soil microorganisms.

The advantage of such indirect methods is in the assessment of the impact of the ameliorant on the two most influential factors in the ecologically effective restoration of technosols: (1) on plants, to reduce the negative environmental impact by the formation of a turf layer [59], and, (2) on soil organisms, to play an essential role in the main processes of soil formation. This method path reduces the time, labor, and material costs for determining the entire spectrum of possible components and physicochemical characteristics

of the analyzed ameliorant (toxic organic compounds, pesticides, metals, various salts, etc.) [46,58].

2. Materials and Methods

We focused on assessing the rate of formation of the soil–vegetation complex during the amelioration and rehabilitation of disturbed lands. The soil–plant complex formation was evaluated by analyzing the growth and development of grass plants on the formed models of mine waste layers. We used two methods of measurement to analyze the growth and development of vegetative cover: a traditional approach for collecting and recalculating plant material, and alternative digital methods of data processing [60–63].

2.1. Materials

2.1.1. Wastewater Sludge

Wastewater sludge from the pulp and paper mill (SPP) was taken from a biological wastewater treatment facility of sulfite pulp production. The sludge was a grey mass waste, consisting mainly of excess activated sludge with various possible inclusions: lignin substances, alumina, and cellulose fiber [43,53,55]. The sludge was dried and left for an incubation period of up to 90 days to reduce the phytotoxicity of the sediment and stabilize the compounds, according to Hechmi et al. [64]. The main properties of SPP (pH, total C, N, and K, and metals—Mn, Zn, Cu, and Pb) were determined by standard methods: the content of carbon, hydrogen and nitrogen were found in the air-dry state of SPP samples using a LECO CHN628 analyzer (USA); the phosphorus content was determined using a Hach Lange DR 5000 spectrophotometer (Germany); qualitative chemical analysis of metals was carried out using a Shimadzu ICPE 9000 atomic emission spectrometer (Japan). The composition of the sludge is shown in Table 1.

Characteristic	SPP	MSS	SPP					
Characteristic	This Work	Ref.	Ref.					
рН	6.00 ± 0.50	6.6 [33], 6.86 [38] 6.96 [37], 6.98 [65] 7.05 [36] 7.03–7.12 [42]	$\begin{array}{c} 6.11 \ [66] \\ 6.56 \pm 0.09 \ [67] \\ 6.71 \ [68] \\ 7.38 \pm 0.09 \ [69] \end{array}$					
Electrical conductivity, µS/cm	0.56	2.61 [36], 2.83 [26], 2.85 [33]	1.15 ±1.44 [67] 1.70 [68]					
Organic matter, %	96.00 ± 0.1	26.6 [37], 27.57 [38] 52.7 [36], 65.0 [65] 83.2 [26], 83.5 [70]	10.82 [66] 63.7 [68]					
С, %	47.21 ± 0.15	Organic 47.7 ± 13.7 [71] Total 41.6 ± 3.5 [72]	6.28 [66] 26.0 [52] 41.2 [73]					
N, %	0.36 ± 0.05	4.83 [37], 5.22 [26] 19.4 [42] 78 [33]	1.68 [52] 3.90 [68] 4.18 [73]					
P, %	0.16 ± 0.05	2.43 [37], 3.9 [70] 20.2 [42]	0.29 [52] 0.867 [73] 2 [55], 3.83 [68]					
Mn, mg/kg	Below detection limit	210 [20] 560.70 [65]	109.7 ± 3.1 [69]					

Table 1. Results of physical and biochemical properties of used SPP compared with average compositions of municipal sewage sludge (MSS) and sludge from pulp and paper industry (SPP).

	SPP	MSS	SPP					
Characteristic	This Work	Ref.	Ref.					
Zn, mg/kg	430 ± 50	534 [36], 592.8 [70], 667.62 [65] 952.1 [37], 1062 [20]	165 [52] 258.0 ± 7.2 [69]					
Cu, mg/kg	210 ± 10	90.0 [36], 96.00 [38] 162.56 [65] 843.8 [70], 975 [42]	69 [52] 133 ± 15 [69]					
Pb, mg/kg	Below detection limit	13.53 [38], 15.9 [70] 48.2 [20] 186 [42]	33.5 ± 1.1 [69]					

Table 1. Cont.

Depending on the reviewed literature source, the data accuracy may be not presented by the authors. For the parameters with significantly varying numbers (such as organic matter), the numbers were shown in a single row if the values were close enough.

2.1.2. Mining Rocks for the Rehabilitation Layer

The experiment was based on three types of mining waste: (1) waste rock from gold mining, (2) phosphogypsum from storage facilities of the phosphate fertilizer production, and (3) crushed limestone from mine stockpiles. In (1), beresite is a low-temperature metasomatic rock characterized by quartz, sericite, and carbonate, resulting from the replacement of both igneous and sedimentary protoliths. Mining operations in Russia provide large masses of these waste rocks; the composition of the studied samples is shown in Table 2.

Table 2. Average compositions of waste rock, minerals are listed in descending order.

Carbo	naceous Beresite	es, C (Total) = 0.5	5–9.0%	Argillisites–Beresites, C (Total) \leq 0.03–1.20%							
Unaltered	Low Alteration	Pervasive Alteration	High Alteration	Low Alteration	Pervasive Alteration	High A (>5	lteration 0%)				
	(5–15%)	(15–50%)	(>50%)	(5–15%)	(15–50%)	Sericitic	Quartzitic				
quartz	quartz	quartz	quartz	hydrosericite	carbonate rock	sericite	quartz				
plagioclase	biotite	hydrosericite	sulfide (pyrite, arsenical pyrite)	quartz	sericite	carbonate rock	sericite				
K-feldspar	muscovite	carbonate rock (ankerite)	carbonate rock	sericite	quartz	quartz	carbonate rock				
biotite	hydrosericite	sericite	+/- sericite	kaolinite	kaolinite	kaolinite	kaolinite				
muscovite	sericite	carbonaceous matter	hydrosericite	+/- carbonate rock	+/- hydrosericite	pyrite	pyrite				
carbonaceous matter	carbonaceous matter	pyrite		clinkstone	clinkstone	+/- clinkstone	+/- clinkstone				
	+/- carbonate rock	+/- muscovite		muscovite	pyrite						
	tourmaline	tourmaline		pyrite							

In (2), phosphogypsum is the calcium sulfate hydrate formed as a by-product of phosphate fertilizer production, consisting mainly of $CaSO_4 \cdot 2H_2O$ (>80%). The volume

of waste buried at gypsum disposal and storage facilities can reach tens of millions of tons [50].

In (3), crushed limestone is primarily composed of calcium carbonate mineral (>97%) from mine dumps. According to the analysis of particle size distribution, the diameter of crushed chips varied between 8 and 25 mm. Laboratory analysis of mine waste samples indicated their close geochemical proximity to the global abundances of the elements (Table 3).

Beresite V	Vaste Rock	Phosph	ogypsum	Limestone						
Mg	8300	S	236,600	Ca	387,604					
Na	8770	Ca	280,202	Mg	17,000					
Ca	8050	Si	3597	Mn	23.00					
Ti	6000	Р	3226	V	2.10					
Mn	840	Al	1799	Zn	1.20					
Ba	720	Fe	1747	Ni	1.20					
V	180	F	1600	Cd	0.74					
Sr	130	Na	297	Cu	0.47					
Zn	102	Κ	249	Pb	0.10					
Cr	99	Cl	181	As	0.10					
Ni	75	Mg	200	Sb	0.10					
Cu	49	Mn	77							
As	31									
Co	23									
Pb	14									
Cd	2									
Mo	1									

Table 3. The average element composition of used mine waste, mg/kg.

2.1.3. Soil

The soil of natural origin for the control group was sampled in the Leningrad Oblast at the field-protective territory, as human-altered soil (Figure 2, N60.2811, E30.2342). Soil samples represented the upper 30 cm fertile layer of deformed sandy Podzols (>80% of the 0.05–2.00-mm fraction). The soil density was $1.3 \pm 0.05 \text{ g/cm}^3$, the pH_{water} was of 5.00 ± 0.5 , and the content organic matter $6.85 \pm 0.7\%$. The soil was air-dried and passed through a 2-mm sieve.

2.1.4. Peat Mixture

Peat mixture is an alternative experimental ameliorant for comparing and assessing the SSP applicability degree as a soil amendment. Peat mixture was studied because of its highly widespread use as a soil additive in soil rehabilitation works. The peat mixture that was used was a commercial product, being a sifted and deoxidized peat of medium decomposition with the addition of lime (100–180 mg/L nitrogen (NO₃ + NH₄), 135–255 mg/L phosphorus (P₂O₅), 115–215 mg/L potassium (K₂O), and pH~5–6).

2.1.5. Plant Material

The effect of soil additives was assessed on the mix of two plant species of the cereals family: ryegrass *Lolium perenne* and meadow fescue *Festuca pratensis*. Ryegrass and fescue are locally widespread species of flora that adapt well to anthropogenic conditions and are recommended for land reclamation. The seeding rate of the grass mixture was set as 200 t/km² (20 centner /ha), according to GOST R 57446–2017 'Best available techniques. Disturbed lands reclamation. Restoration of biological diversity'.



(a)

Figure 2. Soil sampling area: (**a**) provenance of typical soils of coniferous woodlands in Leningrad Oblast (based on the free blank map, commons.wikimedia.org); (**b**) agricultural land and forest shelter belts with disturbed Podzols westwards and southwards of a cleared field of the rural settlement of Agalatovo (based on the Yandex satellite image).

(b)

2.2. Trial Set-Up

An experimental setup consisted of models of the mine waste layers (dump surface) and soils with plant cover. The models were formed at the working surface of 15×15 cm according to the following scheme: a 15–20 cm thick layer of the dumped waste and 20 cm of cultivated soil with soil additives, as a minimal required layer thickness for land rehabilitation.

The comparative evaluation was conducted in two types of ameliorants (soil additives): a wastewater sludge of pulp and paper industry, and a peat mixture as an alternative soil additive. The application of soil additives was carried out at three established ratios based on recommendations for introducing a peat mixture, recommendations for the optimal soil density for grass plants, a literature review of scientific works in this field, and preliminary analyses of substrates. To assess the effective ratio of soil additive to soil, all other models were formed with the addition of the ameliorants at ratios (by volume) of 1:1, 1:2, 1:3 (SPP/peat:soil).

After stabilization of the complexes (1 week), seeds of the grass plants were evenly sown in all models. The general scheme of the model complexes and their principle of formation is shown in Figure 3.

The complex of models was set-up in favorable microclimatic conditions (T > 20 $^{\circ}$ C; RH (atm) < 50%, W (soil) < 80%) with LED phyto-lighting providing the required lighting conditions (full spectrum of luminescence, 35 W/lamp).

The experiment was carried out for 70 days to complete all grass vegetative periods (60–70 days); the results of the study represent 40 days, to evaluate the exponential growth stage of the grasses (ryegrass *Lolium perenne* and meadow fescue *Festuca pratensis*). The following parameters were measured in the plants: seed germination, biomass growth, and growth rate.

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Ч	-	1:1	1:2	1:3	-	1:1	1:2	1:3	-	1:1	1:2	1:3	-	1:1	1:2	1:3			
		withou	t layer			on was	te rock		on	phosp	hogyps	um	on crushed limestone						
		1:1 Peat No. 6	1:2 Peat No. 8	1:3 Peat No. 10		1:1 Peat No. 12	1:2 Peat No. 14	1:3 Peat No. 16		1:1 Peat No. 18	1:2 Peat No. 20	1:3 Peat No. 22		1:1 Peat No. 24	1:2 Peat No. 26	1:3 Peat No. 28			
	Model with soil	1:1 SPP No. 5	1:2 SPP No. 7	1:3 SPP No. 9	Model with soil	1:1 SPP No. 11	1:2 SPP No. 13	1:3 SPP No. 15	Model with soil	1:1 SPP No. 17	1:2 SPP No. 19	1:3 SPP No. 21	Model with soil	1:1 SPP No. 23	1:2 SPP No. 25	1:3 SPP No. 27			

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Figure 3. Scheme of the formation of 28 model types with side and top views. Models consist of: A group—control models with soil on different layers, B group—six types of models with different substrates (SPP, peat with soil) and their ratios (1:1, 1:2, 1:3) without a layer for rehabilitation, C group—six types of models on a rehabilitation layer of waste rock, D—six types of models on a rehabilitation layer of phosphogypsum, E—six types of models on a rehabilitation layer of crushed limestone.

2.3. Measurements Method by the DIA for Plant Cover Assessment Trial

The assessment of the applicability of non-traditional ameliorants for plant cover includes two measurement approaches: (1) the traditional method measuring physical quantities (seed germination, grass height) and the cutting of plant material, and, (2) a method using digital image analysis (DIA).

The method of DIA consisted of a systematic approach to photographing vegetative cover following the plant material analysis [60,62,74]. The advantage of this method (in comparison with the traditional approach) lies in the obtainment of data without high expenditure of materials and time (the method is carried out without destroying the analyzed plant material) [60,75,76].

The measuring method for DIA consisted of data collection (shooting plant material) and processing digital RGB (Red, Green, Blue) images. Digital processing of images was carried out using the Java-based open-source software ImageJ as follows: (1) removal of the background (soil, stones, various inclusions, etc.) [62,77,78], counting [78], classification [79], information processing based on color correction [62,75], and measurements of determined physical quantities (the number of units of pixels, roundness, lines—for recalculating biomass, seeds, and shoots) [62,80].

2.4. Analysis of Plant Growth and Vegetation

Vegetative cover and the accumulation of plant biomass are essential for restoring the technogenic ecosystem [8]. Plant vegetative cover prevents land degradation and air pollution by wind deflation and water erosion. Sustainable plant and soil cover have a beneficial impact on environmental security and quality.

Vegetative cover assessment was carried out on all growth stages of grass plants: germination and exponential growth stage by biomass and plant height. For plant biomass and growth rate, a *Gompertz sigmoid function* analysis was used [81].

2.4.1. Germination

The germination assay included a comparative assessment of seed germination percentages in the studied models, to the control groups of models. The germination assay was carried out on two species of grass plants—ryegrass *Lolium perenne* and meadow fescue *Festuca pratensis*. The calculation of germination was estimated based on the number of germinated seeds (%) for 5–7 days from the day of planting. The recalculation was carried out using digital image processing.

2.4.2. Plant Cover Biomass

The biomass of the vegetative cover was analyzed by two methods of measurement: the traditional method, with the destruction of plant material, and the digital data processing method. Both methods of measurement were conducted in the period of exponential grass cover growth:

1. Direct visual measurement (traditional method): three-time cuts of aboveground plant material (3 cm from the ground) were made bi-weekly to measure an increase in the biomass.

2. Digital image analysis (alternative method): This was based on a digital RGBimage analysis of vegetative cover. The method was carried out on a 2–3 day RGB image shooting basis to estimate the biomass growth through the LAI (leaf area index). The index characterizes vegetative cover as the area of vegetative cover per unit of surface (land) area. Dimensionless quantity reflects the plant unit's projected area (LAI = leaf area/land area, m^2/m^2) [62,75]. An example of the RGB image processing results is represented in Figure 4.



Figure 4. Example of images for digital RGB image processing (in steps) for grass cover in different vegetation stages (1,2,3 weeks): (a) RGB image before digital image processing; (b) color correction for background removal; (c) removal of the background (soil, stones, various inclusions, etc.).

2.4.3. Plant Cover Height Rate

The height rate of the vegetative cover was analyzed by DIA which was based on a digital RGB image analysis of vegetative cover [82]. The method consisted of 2–3 days of RGB image shooting of grass cover, in height, against a black background, with a measuring scale beside the plant cover. The scale was selected automatically on the image and the pixel/mm ratio was calculated by the scale. The background was separated from the plant semi-automatically. Digitization of grass cover for the estimation of the plant cover height was based on comparison of the obtained measured growth lines with the size marks.

2.4.4. Data Analysis

Origin 8.5 Pro software (OriginLab Corporation, Northampton, MA, USA) and Javabased open-source software ImageJ were used to analyze the experimental data. The normality of distribution and homogeneity of variance were tested; the differences among treatments were analyzed by ANOVA tests.

3. Results and Discussion

3.1. Germination

The germination assay was carried out to estimate the amelioration efficiency on the substrates in comparison with the control soil, and to assess the phytotoxicity of the treated soil. Germination assay identifies unsuitable conditions, i.e., soil salinity, presence of toxic compounds, and plant nutrition deficiency [54,58,83].

The germination and biomass growth assays showed that the germination of seeds in the studied soil–plant complexes of the grass mix (ryegrass *Lolium perenne* and meadow fescue *Festuca pratensis*) depended on: (1) the ratio of the components and, (2) waste rock layers. The germination results are shown in Figure 5.



Figure 5. Diagrams of seed germination (%) by four groups of rehabilitation layers with seven types of models—control model (soil), wastewater sludge SPP and peat mixture: A—without layer, B—on waste rock, C—on phosphogypsum, D—on crushed limestone. The data represent the mean of 4 replicates; the vertical bars indicate standard deviations.

The results showed high germination levels on the control soils (~70–80%) relative to the treated soils, which is explained by the high fertility of the control soil. The optimum conditions for seed germination were obtained at: (1) a ratio of 1:2 (peat:soil)—50–60%, and, (2) a ratio of 1:3 (SPP/peat:soil)—65–75% and 60–70%. Results indicated a non-phytotoxic effect of SPP in optimal ratios for ryegrass and meadow fescue. However, soils treated with peat mixture showed healthier results of seed germination at most types of layers than soils treated with SPP.

Drastic inhibition of seed germination was observed at soils treated with an extensive amount of SPP, 1:1. Similar results were observed in earlier studies [16,69]. Hence, high dosages of SPP should be avoided to prevent a negative effect on plant cover formation.

Evaluation of seed germination of ryegrass and meadow fescue on reclaimed layers showed a difference in germination, which can be explained by: (i) neutralization of the soil layer due to waste forming dumps (layers), (ii) optimal regimes (air, water, and nutrient), and (iii) the degree of soil moisture. Optimal air regimes are determined by the density of the substrate and the ratio of the components; the water regime depends on the moisture capacity and water loss of the substrates; the nutrient ratio depends on the content of the applied components in the initial additive. Factors (ii) and (iii) can be formed by differences in substrate densities and the ratio of components, and the influence of factor (i) is further confirmed by the analysis of the biomass of a vegetative cover and the percentage of germination on waste rock models. The lowest results were observed in model groups formed on the waste rocks, and the highest was found on the crushed limestone layer.

Speaking of the result dissimilarity, we assume that several factors could affect the difference in the seed germination. Low seed germination on the waste rock can be explained by the particle size distribution: the rest of the model mixtures have a forming mineral layer of low water permeability, while the 1–4 cm beresite crushed specimens provide better water drainage and thus can contribute to moisture shortage under equal laboratory precipitation. Furthermore, SPP is characterized by a higher water-holding capacity as compared with peat mixture, so the seed germination on waste rock varies significantly. The control samples confirm this hypothesis: considering the variability of germination over the blank layer, crushed limestone, and phosphogypsum, we see levels close to 80%, while over the waste rock, the percentage is normally below 70%, close to 50%. Moreover, the capillary uptake of metals from the mineral substances may inhibit the grasses as reported in early studies by Aoyama and Kuroyanagi [83] and recent laboratory research by Wiewióra and Żurek, [84] and several other authors.

Overall, the results of the seed germination assay determined that with the application of a rational amount of SPP, there is no phytotoxic effect on seed germination from the treated soil. However, the results of the experiments could be influenced by many other factors: salinity [54,58,84] (and EC [85]), excessive ammonium nitrogen levels [16,54,58], high amount of metals [54], and poor physical structure [86].

3.2. Plant Cover Biomass

An analysis of biomass sections was carried out in three-time measurements by weighing plant material cuts. The results of the plant cover biomass assay (weight of plant material cuts) are shown in Figure 6.



Figure 6. Diagram of the biomass by the weight of plant material cuts (g/m²): A group of models: A without layer, B—on waste rock, C—on phosphogypsum, D—on crushed limestone; with six types of wastewater sludge and peat mixture (SPP, peat) and ratios (1:1, 1:2, 1:3); with 3-time measurements. The data represent the mean of 4 replicates; the vertical bars indicate standard deviations.

The method of plant biomass analysis using DIA and LAI made it possible to analyze the rate of plant biomass growth over the entire growing season (exponential growth stage). The result of 36 days of measurement reflected the main trends and identified the main factors of the impact on the reclaimed layer and formed plant–soils complexes (Figure 7).



Figure 7. Rate of plant biomass growth by DIA and LAI (%): A—on the model without layer, B—on the waste rock of a gold ore deposit, C—on phosphogypsum, D—on crushed limestone, (A1,B1,C1,D1)—on mixture of soil and wastewater sludge SPP, and (A2,B2,C2,D2)—on mixture of soil and peat.

Comparison of the results of the two presented measurement methods on the plant material collecting days showed a correlation dependence of: $r_1^2 = 0.95$, $r_2^2 = 0.75$, and $r_3^2 = 0.75$.

The results of the biomass growth rate showed the dependence of the seed germination on the applied substrate. The effectiveness of the vegetative cover formation in the disturbed areas depended on the deposited layer, ameliorant, and its quantity.

The analysis of the weekly increase in the biomass of the vegetative cover showed that the peat mixture achieved better results of biomass increase (LAI > 50% on phosphogypsum, on crushed limestone, and models without layer) than soil with the addition of SPP (LAI < 40%).

Peat mixture applied into the soil in the ratios of 1:2 and 1:3 showed the highest results of vegetative cover formation and biomass growth rate, where LAI > 50% on control models, LAI > 30% on waste rock layer, LAI > 40% on phosphogypsum layer, and LAI > 60% on crushed limestone. Plant growth was accelerated and overall biomass growth was improved. The closest results of the biomass growth rate were in models where SPP was introduced into the soil in a ratio of 1:3, where LAI > 30% on all types of layers.

Speaking of the amount of application, the ratio 1:1 is not applicable to peat mixture nor SPP (LAI~10%), as an ameliorant. Inhibition of plant growth and lower overall productivity of the biomass of the vegetative cover were noted. These results can be explained by: (1) a high degree of soil lightness, i.e., a low density of the soil substrate unacceptable for the effective formation of grass vegetative cover, or an increase in the phytotoxicity of the soil layer, which in general results in an inhibitory effect on plant growth. Similar research results have already been noted with excessive addition of sewage sludges [8,39].

The analysis of the influence of the recultivated layer determined that the formation of a minimal 20 cm soil layer was the most ecologically effective vegetative cover form on models with a neutral medium (pH 6.5–7.0), due to the characteristics of the dump rocks. Rehabilitation of waste rock dumps (from gold mining) reflected the most negligible results in germination and biomass growth, which, in turn, was also explained by the acidity of the formed conditions.

The decrease in the acidity of the soil layer worsened the efficiency of biomass for reasons of: deterioration of the optimal acidity conditions, and an increase in the migration ability of metals, which led to an increase in the phytotoxic effect of soil layers [15,17].

No phytotoxic effect of the added ameliorants nor reclaimed layers was found in any of the studied models: there were no signs of chlorosis, necrosis, or other plant damage.

High results of biomass growth, normal growth, and vegetation of plants were observed when a peat mixture was added at ratios of 1:3 and 1:2 and SPP 1:3 to the soil, which confirmed the earlier obtained seed germination results.

Based on the obtained growth functions of the grass plant cover (Gompertz sigmoid function) and their correlation coefficients, it can be concluded that the development of the vegetative cover on the formed treated soil models proceeded without deviation and within the standard growth rate ($r^2 = 0.84-0.98$). Analysis of variance (ANOVA) was used to examine differences between types of formed soil–plant models. All statistical analyses were performed at the 95% confidence level (p < 0.05).

3.3. Plant Cover Height Rate

The results of plant cover height showed the dependence of the germination on the applied substrate and deposited layer.

The analysis of the weekly increase in plant cover height mainly confirmed results of biomass growth. The height grass cover formation on SPP mixture (obtaining a maximum of plant cover height ~10 cm) reached a higher maximum than grass cover on peat mixture (>10–15 cm). Results of plant cover height grown on a mixture of soil and SPP showed similar results on all types of rehabilitation layers. The obtained results of grass cover height are shown in Figure 8.

Based on the obtained growth functions of the grass plant cover (Gompertz sigmoid function) and their correlation coefficients, it can be concluded that the development of the vegetative cover on the formed treated soil models proceeded without deviation and within the standard growth rate ($r^2 = 0.85-0.99$). Analysis of variance (ANOVA) was used to examine differences between types of formed soil–plant models. All statistical analyses were performed at the 95% confidence level (p < 0.05). No visual or measurable signs of the impact of lower horizons on vegetation height were found. However, higher values of maximum vegetation height were noted in the following groups of models: (1) on the group without horizon 0, (2) on the rehabilitated layer of waste rock, and, (3) on phosphogypsum. Overall, no visual distortion was recorded, so the results of biomass measurements can be considered more indicative.



Figure 8. Rate of plant cover height (cm): A—on the model without layer, B—on the waste rock of a gold ore deposit, C—on phosphogypsum, D—on crushed limestone, (**A1,B1,C1,D1**)—on a mixture of soil and wastewater sludge SPP, and (**A2,B2,C2,D2**)—on a mixture of soil and peat.

3.4. Plant Growth and Vegetation

Measured characteristics were recounted in comparison with results from control models and maximum obtained values. Recounted characteristics were compiled in the total matrix of plant cover growth and vegetation indicators (Figure 9).

The combined matrix of indicators proved the results of the germination assay and the biomass cover assessment (though LAI), replicating a particularly relevant amount of SPP soil application for disturbed land reclamation. In comparison with peat mixture additives, the recommended ratio of SPP to soil was 1:3, which was based on achieved results of grass cover growth and vegetation development.

Measured characteristics by		WITHOUT LAYER						ON WASTE ROCK					ON PHOSHOGYPSUM						ON CRUSHED LIMESTONE						
			SPP			Peat		SPP		Peat		SPP		Peat			SPP			Peat					
compa	1501, 70	1:1	1:2	1:3	1:1	1:2	1:3	1:1	1:2	1:3	1:1	1:2	1:3	1:1	1:2	1:3	1:1	1:2	1:3	1:1	1:2	1:3	1:1	1:2	1:3
1	Germination	0	18	24	15	34	46	58	124	116	75	44	89	15	4	53	9	16	71	105	9	24	116	79	104
ontro Is	Plant tissue cuts 1	0	14	77	29	54	33	36	133	211	116	144	203	22	36	75	19	64	98	21	23	25	30	194	211
rith c nodel	Plant tissue cuts 2	0	32	109	38	123	118	81	127	219	207	193	432	43	36	119	21	75	121	65	110	115	83	325	224
	Plant tissue cuts 3	0	37	31	22	29	22	47	116	162	67	56	106	22	27	25	13	13	29	35	20	8	29	79	80
н	LAI (biomass), average	3	21	37	39	79	91	18	36	55	35	26	31	33	50	35	47	46	89	17	37	71	35	75	100
iximu	LAI (biomass), max	3	25	41	51	93	98	21	44	53	47	34	39	37	51	45	59	53	91	19	39	74	44	84	100
thme	Plant height, average	0	54	54	70	65	89	53	59	69	56	49	61	54	60	57	84	77	100	54	61	55	53	94	90
wit	Plant height, max	0	56	64	74	69	90	54	54	61	48	47	58	49	58	49	82	77	100	44	55	56	52	91	79

Figure 9. Measured characteristics in comparison with control models/maximum.

The average rate of measured plant indicators showed the optimal SPP ratios for application to soils (soil treating) in disturbed land of the mining industry. Treated soils formed without the mining waste layer showed average values for plant growth and vegetation. The optimal ratios in the absence of a mine waste layer were 1:3 for SPP, and 1:2 and 1:3 for peat mixture, as the obtained values of the average rate of measured plant indicators were > 50%:

1. For the waste rock layer, the optimal ratios were obtained with soil at a 1:3 addition of SPP and peat mixture to the soil (average rate > 50%);

2. For the phosphogypsum layer, the optimal ratios for waste rock dump rehabilitation for SPP were 1:2 and 1:3, and for peat mixture, all types of ratios (1:1, 1:2, and 1:3) resulted in the average rate of measured plant indicators > 50%. The highest value of the average rate of measured plant indicators was obtained with a ratio of 1:3 peat mixture to soil (average rate >100%);

3. For the crushed limestone layer, the obtained results showed a more suitable addition of peat mixture than the addition of peat for crushed limestone; the average rate of measured plant indicators: (1) for SPP 1:2 (>60%) and 1:3 (>90%) to soil; (2) for peat mixture 1:2 (>100%) and 1:3 (>100%).

4. Conclusions

The assessment of the applicability of SPP as a soil additive for the rehabilitation of disturbed lands in the mining industry was carried out based on an evaluation of the plant cover growth, considering the climatic, environmental, and anthropogenic factors of the technogenic substance. The growth efficiency of a plant cover was evaluated by the following parameters: (1) seed germination, (2) plant cover biomass, and (3) plant cover height rate.

The SPP influence on seed germination was measured by the digital image analysis method. In general, a rational application of SPP to the soil does not hurt seed germination (seed germination > 50%). However, excessive application (in ratio 1:1 of SPP to the soil) negatively affected the germination parameter, showing phytotoxic effect and growth inhibition.

The influence on plant cover biomass was analyzed by the digital image analysis method and leaf area index (LAI). The SPP (in ratio 1:3) influence on biomass growth rate reflected similarly on all soil–plant complexes (LAI > 30% on all types of layers). However, it resulted in a lower biomass quantity in comparison with soils with peat mixture application (LAI > 50% on control models, LAI > 30% on waste rock layer, LAI > 40% on phosphogypsum layer, and LAI > 60% on crushed limestone).

The effect of SPP on the plant cover height rate replicated previous results of germination and plant cover biomass. However, it showed no significant difference in SPP ratios or mine waste layer type. Overall, results of the evaluation of plant cover formation showed that peat mixture application resulted in healthier and higher levels of plant cover growth than SPP amendment. Nonetheless, SPP results were close to the result of peat mixture of plant growth influence in a ratio 1:3 to soil: (1) germination: 1:2 peat—50–60%, 1:3 peat—60–70%, and 1:3—SPP 65–75%; (2) biomass: 1:3 SPP LAI = 30–40% (in limestone >60%) compared with peat mixture LAI = 30–80%; (3) height: in SPP ratio 1:3 to soil—10 cm, and peat mixture—10–20 cm.

Land rehabilitation with the formation of a 20 cm soil layer on mine waste dumps is environmentally suitable with an SPP application ratio of 1:3 to the soil. The amount of SPP in ratio 1:1 was found not applicable as an ameliorant.

Author Contributions: Conceptualization, T.A.P., E.R. and J.B.; methodology, M.A.P. and J.B.; software, E.R.; validation and formal analysis, M.A.P. and T.A.P.; investigation, E.R.; resources and data curation, A.V.A.; writing—original draft preparation, E.R.; writing—review and editing, T.A.P. and A.V.A.; visualization, E.R. and A.V.A.; supervision, M.A.P.; project administration, T.A.P.; funding acquisition, T.A.P. All authors have read and agreed to the published version of the manuscript.

Funding: This research received no external funding.

Data Availability Statement: The data presented in this study are available in this article.

Acknowledgments: The studies were performed using the equipment of the Common Use Centre of the Saint Petersburg Mining University. We thank the Academic Editor and three anonymous reviewers for critically reading the manuscript and suggesting substantial improvements.

Conflicts of Interest: The authors declare no conflict of interest. The funders had no role in the design of the study; in the collection, analyses, or interpretation of data; in the writing of the manuscript, or in the decision to publish the results.

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