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Revegetation and Quality Indicators of Technosols in Restored Mine Fields with Saline Mine Spoils

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Abstract: The European Union prioritizes nature restoration, particularly in semiarid Mediterranean regions where integrating degraded coal mining areas into the landscape is essential. This involves maximizing water use and controlling runoff. A rehabilitation project in a former mining quarry was conducted with the objective of constructing suitable Technosols to support vegetation, limit erosion, and reduce rehabilitation costs. To prepare the substrate, mine spoils (saline materials) were mixed with residual materials, including discarded lignite powder, sewage sludge, pig slurry, and straw. Pig slurry was also introduced as a mulch in the experiment. A complete randomized block design with three replicates was set up, with each block containing two plots of the prepared substrate. In one of the plots, pig slurry was applied on the surface as a mulch to enhance infiltration and promote plant establishment. The quality of the newly created Technosols and the benefits of mulch application were evaluated 2 and 4 years after the rehabilitation. After two years, salt-tolerant plant species colonized the rehabilitated areas, providing sufficient vegetation cover to control water, soil, and nutrient losses, keeping soil losses below a 2.2 Mg ha^{-1} yr^{-1} threshold. Four years later, the new Technosols showed a fourfold increase in soluble organic-C content (up to 0.59 g kg^{-1}) and higher soil respiration rates compared to the mine spoils and lignite powder in the surrounding degraded quarry areas. No significant differences were observed in any parameters due to superficial slurry application. Addressing salinity and optimizing vegetation cover are crucial for the successful formation and sustainability of Technosols in these environments.

Keywords: erosion; microbial activity; mining filed; nitrates; runoff; salt-tolerant plants; sewage sludge; slurries; soluble organic-C

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1. Introduction

In northeastern (NE) Spain, particularly within the Ebro river basin, there is a significant area affected by salinity. The marine deposits in this area include clays, sandstones, shales, limestones, and gypsum. Poor drainage and high evaporation in the southern parts of the basin have led to the formation of saline soils [1]. In the southeastern (SE) distal part of the Ebro basin, on the border between the NE Spanish regions of Aragon and Catalonia, the predominant geological formation is the Mequinenza formation, dating back to the Chattian stage of the Oligocene period [2]. This formation features an alternation of calcareous sandstones, sandy limestones, marlstones, and varicolored claystones, with veins of secondary gypsum visible in some marlstone layers. Seven lignite layers, resulting from palustrine deposits of organic matter, are also identifiable [2]. Lignite has been exploited since the 19th century [3], with an efficiency of about 12%, and overburden materials from this mining activity have been deposited on terraced slopes. Along the Ebro and Cinca riverbanks surrounding the mining area, colluvial formations dating from the Pleistocene (Quaternary) are composed of gravel and pebbles embedded in a silty-clayey matrix, along with alluvial formations [2].

Coal mining-affected areas, like the one focused on in this study, are central to climate change adaptation efforts due to their rehabilitation activities, including revegetation. The United Nations Convention to Combat Desertification highlights the importance of revegetating degraded areas [4]. In Europe, Directive 2004/35/CE [5] addresses environmental responsibilities, including repairing environmental damage (e.g., reducing organic carbon stock in cropland mineral soils, or decreasing forest cover), while emphasizing prevention through national plans. Furthermore, the European Council has adopted a regulation on nature restoration [6] aiming to restore at least 20% of the European Union's land and sea areas by 2030, and all ecosystems in need of restoration by 2050. In Spain, specific legislation [7] mandates the rehabilitation of mining affected areas, including measures to prevent erosion and integrate restored areas into the landscape.

A successful rehabilitation project must generate the necessary conditions to initiate and accelerate the reintegration of the affected areas into their surroundings, thereby recreating ecosystems [8]. In a circular economy, the value of products, materials, and resources is maintained for as long as possible, minimizing waste generation [9]. Following these principles, ecosystem reconstruction should prioritize reusing in situ resources, such as overburden mine materials [10].

Mediterranean arid zones (<250 mm rainfall year^{-1}) and semiarid zones (250–450 mm rainfall year^{-1}) [11] present additional challenges for rehabilitation, such as water scarcity. Measures to enhance water infiltration and availability are crucial in the Ebro river basin. Erratic heavy rainfall events also increase the risk of soil erosion. In the study area, maximum 24-h rainfall for a 10-year return period can reach 75 mm [12]. Despite these challenges, existing research has been conducted in temperate and tropical climates [13], leading to a lack of information on arid and semiarid regions like the study area [14].

Sustainable mining reclamation requires measures to promote soil cover by vegetation and improve water infiltration [15]. Preventing soil compaction is essential to controlling runoff and erosion [16]. In the Ebro river basin, saline materials limit water availability for plants by reducing the soil's osmotic potential [17,18].

A successful rehabilitation project must supply nutrients and organic matter for vegetation establishment, as these are critical for physical, chemical, and biological soil fertility [19,20]. Adding organic materials supports the formation of "minesoils" [21]. Organic fertilizers from livestock farming are significant sources of N and other nutrients, as well as organic matter. Notably, Aragon and Catalonia lead in Spain and in Europe in livestock farming, particularly pig farming [22], which supports extensive winter cereal production [23]. However, the environmental problems associated with pig slurry, such as greenhouse emissions, water pollution, and eutrophication, necessitate diversification of its use [24,25]. Sewage sludge, another potential resource (ca. 1.2 million tons of dry matter year^{-1} [26]), is also available. Although legislation exists to prevent heavy metal accumulation in agricultural fields when using sewage sludge as fertilizer [27], no equivalent regulations apply to mine restoration, likely because it typically involves a one-time intervention, and the vegetation is not normally intended for human or animal consumption.

According to different studies [28,29], a minimum of 3 years is required to develop an A-horizon (surface soil layer) in rehabilitated soils. The accumulation of pedogenic organic-C is considered to be an indicator of the success of mine soil development. In restored lignite-rich mine soils, different organic fractions can be identified [30], including lignite from the parent substrate and decomposed plant residues. Although lignite is not an inert component and can decompose microbiologically [31], it does not serve as a nitrogen source for plants. Instead, soluble organic-C, the most dynamic soil C pool [32], comprises readily biodegradable organic compounds such as amino acids, carbohydrates, organic acids, and sugars. Other authors quantify the labile soil organic-C in an aqueous solution of a neutral salt (as potassium sulfate), to obtain the most metabolizable organic-C [33]; it includes low-molecular-weight hydrophilic organic matter from mineralization processes, excluding microbial biomass [33].

Carbon dioxide (CO_2) evolution or oxygen consumption rates, which can be monitored continuously or semi-continuously, indicate microbial activity and are widely used to assess soil quality [34]. Laboratory respiration measurements [35] have proven effective for evaluating the success of Technosol construction.

In the context of a semiarid Mediterranean landscape, this study evaluates the effectiveness of rehabilitation activities on newly created Technosols using saline materials and other residual products in a degraded area of the Mequinenza municipality affected by mining activities. The general objective is to assess the potential for successful ecological restoration of degraded saline mine spoils, in semiarid environments, and in alignment with the European Union 2024 Nature Restoration Law [6]. The specific objectives are as follows:

- Quantify the effects of surface slurry application as mulch on plant establishment, runoff water, soil erosion, and soil mineral nitrogen (N).
- Evaluate soil cover and the establishment of salt-adapted plant species as indicators of successful revegetation.
- Measure changes in key soil parameters, including soluble organic carbon (C) and microbial activity, as indicators of soil quality improvement.

Our working hypotheses are as follows:

- Salt-adapted plants will successfully establish in the experimental areas due to the ameliorative properties of the Technosol substrate and mulch materials, leading to reduced soil erosion and nutrient loss.
- Rehabilitation activities will enhance soil quality, as indicated by increases in soluble organic carbon (the most dynamic fraction of soil organic matter) and microbial activity (measured as soil respiration).

This research provides valuable data and practical methodologies for restoring degraded saline environments using mine spoils. It addresses critical knowledge gaps in ecological restoration strategies for semiarid regions, contributing to the broader objectives of nature restoration under the European Union framework.

2. Materials and Methods

2.1. Study Site Description

The mine area is located in northeastern Spain (Figure 1), 5 km SE from the city of Mequinenza, and straddles the regions of Aragon and Catalonia (UTM coordinates: 31T X: 279232 Y: 4581397; geographical coordinates: 41°21′21″ N, 0°21′23″ E). The climate is semiarid Mediterranean. Climate data were obtained from the Mequinenza meteorological station (Zaragoza, Spain). The mean annual rainfall is 375 mm, and the annual average temperature is 14.1 °C. Spring, summer, autumn, and winter average temperatures are 12.6, 23.1, 15.0, and 5.6 °C, respectively.

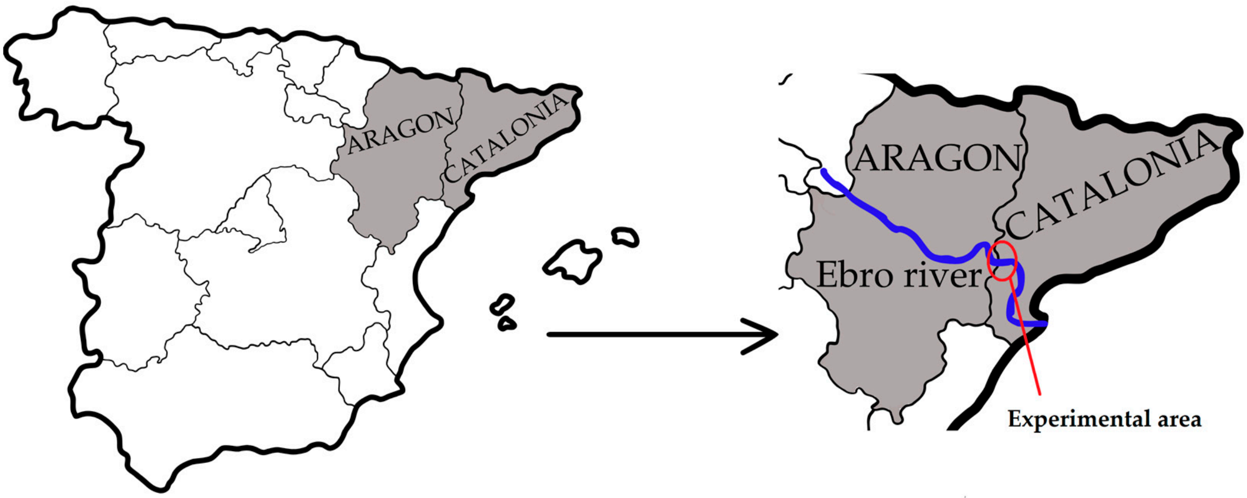

Figure 1. Location map of the experimental area in northeastern Spain.

2.2. Experimental Design and Layout

The substrate used in this rehabilitation process was composed of different materials (Table 1). The first component was a residual lignite powder accumulated in settling ponds. These ponds were used to separate lignite from denser materials by flotation, allowing water to evaporate naturally and leaving sedimented lignite powder. The particle size of the lignite powder was similar to that of lime mineral particles. A 10 cm layer of lignite powder was mixed with 370 m^3 ha^{-1} of pig slurry and 9 Mg ha^{-1} of cereal straw, applied in five separate applications. The second material consisted of mine spoils, primarily derived from marlstone deposits in the area, with 89% of the material by weight comprising additional colluvial coarse fragments. The third component was sewage sludge. These materials were combined in a volume ratio of 1:3:1, respectively. This ratio was used based on previous experiments in the reclamation of marginal lands with sludges [36]. From these studies, the mineral material intended to be mixed with sludges (in our case, the 1:3 substrate) should contain a minimum of 20% mineral particles with an apparent diameter of less than 2 mm, along with a clay content of about 5%. Finally, we maximized the proportion of sludge because the spoils are saline, with very low fertility, and the rehabilitation procedure must be carried out in a single step, ensuring its sustainability over time. The chemical characteristics of the fine fraction (<2 mm in diameter) of the resulting substrate are presented in Table 1.

In an area with a slope gradient of 30%, six plots were established. Substrate was deposited on the slope to create the plot area. Each plot had a surface area of 78 m^2 (13 m long, 6 m wide), with a thickness of 0.25 m. These plots were provided by the mining company; therefore, their dimensions respond to the types and sizes determined by the machinery they were using to shape the final reliefs of the spoil banks.

Under field conditions, it was observed that the lignite fine (powder) material, due to its silty texture, tended to become compacted. This compaction posed a potential challenge for plant emergence in the new substrate. To address this issue, a mulch treatment was introduced and evaluated. It involved the surface application (or lack thereof) of pig slurry. Slurry was chosen because it was abundantly available in the area and had the ability to form small, scattered crusts when its solid fraction dried on the surface. This process also increased the already important surface roughness. Three blocks (experimental replicates) were established, with each block consisting of two plots. In one randomly selected plot per block, an equivalent of 91.6 m^3 ha^{-1} of slurry (Table 1) was applied to the surface using a pipe. The rate was associated with the maximum amount of slurry that can be applied with the available machinery in the area, and with a single application. The exact amount specified in the text was measured a posteriori. We weighed the contents of the slurry tank before and after application.

Table 1. Chemical and physical properties of the components of the substrate (lignite powder, mine spoils, pig slurry, and sewage sludge) and of the final fine fraction (diameter < 2 mm) of the substrate used for spoil bank rehabilitation. Chemical characteristics (over dry matter) of pig slurry applied on the surface, as a mulch treatment, are included.

Characteristics	**Lignite Powder**	**Mine Spoils**	**Pig Slurry in Substrate**	**Sludge**	**Substrate**	**Pig Slurry on Surface**
Dry matter (over fresh matter, g kg^{-1})	–	–	39	498	13.5	34
pH (potentiometry, 1:2.5)	7.6	7.5	8.5	8.5	7.5	8.7
Electrical conductivity 1:5 (dS m^{-1} at 25 °C, soil:water)	2.2	2.4	5.9	1.8	5.2	5.9
Electrical conductivity (saturated soil extract, dS m^{-1} at 25 °C)	6.3	6.3	–	–	–	–
Total organic carbon (calcination at 550 °C) (g kg^{-1})	–	–	–	291	69.8	–
Total—N (Kjeldahl) (g N kg^{-1})	2.1	2.8	114	51.3	–	143
Organic—N (Kjeldahl) (g N kg^{-1})	–	–	37	33.2	–	34
NH_4—N (Kjeldahl distillation) (g kg^{-1})	–	–	77	18.1	–	109
Total phosphorous (acid digestion) (g P kg^{-1})	–	–	14	21	–	12
Phosphorous (Olsen, mg P kg^{-1})	3	2	–	–	>80	–
Total potassium (acid digestion) (g K kg^{-1})	–	–	110	30	–	89
Potassium (NH_4OAc, mg K kg^{-1})	42	33	–	–	233	–
Sand ($0.05 < ø < 2$ mm, g kg^{-1})	166	623	–	–	329	–
Silt ($0.002 < ø < 0.05$ mm, g kg^{-1})	633	276	–	–	560	–
Clay (<0.002 mm, g kg^{-1})	201	101	–	–	111	–
$CaCO_3$ equivalent (Bernard calcimeter, g kg^{-1})	510	520	–	–	540	–
Calcium (acid digestion, g Ca kg^{-1})	–	–	–	61.5	–	–
Soil water retention (m^3 m^{-3}; in fine fraction <2 mm)	–	–	–	–	–	–
−33 kPa	–	–	–	–	0.32	–
−1500 kPa	–	–	–	–	0.13	–

The six plots were identified by the block number (1, 2, 3) and the final substrate treatment: with slurry (SS) or without slurry (SL) applied on the surface.

2.3. Chemical Laboratory Analysis

The first evaluation phase of the constructed Technosols focused on their chemical properties. This phase was conducted 2 years after the plots were established and spanned from May to November 2002. In May, an initial soil sampling was carried out. Composite soil samples were collected from 10 different points within each plot, at a depth of 0–0.2 m. The fine soil fraction (particle diameter < 2 mm) was analyzed for electrical conductivity (EC, 1:5 soil:water, w/v) and mineral-N content (nitrates). Nitrate (NO_3^-) content was measured by extracting 20 ± 0.5 g soil with 50 mL of 1 mol L^{-1} KCl. These extracts were analyzed using a continuous flow autoanalyzer (Seal Autoanalyzer 3, Seal Analytical, Germany). A second soil sampling was carried out in November of the same year, during which salinity was additionally assessed using the saturated paste method. For each sample, the soil water content at saturation was measured in the laboratory. The electrical conductivity (ECe) and pH (pHe) of the saturated extracts were analyzed. Cations and anions in the saturated extracts were measured by capillary electrophoresis [37]. Concentrations of soluble bicarbonates were determined by titration using phenolphthalein and methyl orange as indicators [38]. The sodium adsorption ratio (SAR) of the saturated extract was calculated as the sodium (Na) concentration divided by the square root of one half of the sum of calcium (Ca) and magnesium (Mg) concentrations, with all ions expressed in millimoles of charge per liter. Furthermore, during the November sampling, a volume of soil was excavated to a depth of 0.25 m in each plot to measure bulk density. The excavated hole (~8 L in volume) was refilled with water using a plastic bag to determine its volume. The excavated soil was oven-dried at 40 °C for 48 h to account for the presence of gypsum in the substrate. The total dry soil mass, along with the proportions of the fine fraction (particles < 2 mm) and the coarse fraction (particles > 2 mm), was obtained. The coarse fraction was further divided in two size classes: particles > 2 mm to 20 mm, and particles > 20 mm to 60 mm. These data were used to calculate the analyzed mineral N content (as nitrates) in the fine fraction (in May and November) per hectare for the top 0.25 m of soil.

2.4. Field Methods

Field measurements of runoff water, soil erosion, and vegetation cover by spontaneous vegetation were performed from May to November 2 years after rehabilitation. In May, a metallic Gerlach box [39] (1 m in length, 0.2 m in width, 0.2 m in height) was installed at the bottom of each plot to collect surface runoff water and sediments over a seven-month period (until November). Since the length of the box was 1 m and the slope length was 13 m, the total area of sediment and water collection was 13 m^2. The Gerlach box consisted of a channel placed flush with the ground, with one lateral edge perfectly aligned to the surface. The collector included a drain connected to a 50 L capacity tank to store water and sediments carried by runoff. The collector was protected by a partially open lid that prevented rainwater from directly entering the tank. Samples from the Gerlach box were collected after each rainfall event. The collected runoff water was analyzed for volume, electrical conductivity, pH, and nitrate concentration, while the eroded sediments were weighed. Additionally, in May, six circles, each measuring one square meter, were delineated within each plot. Three of them were located in the middle of the upper part of each plot, and the other three were located in the middle of the lower part. In each circle, the percentage of vegetation cover was assessed using the Folk diagram [40]. Vegetation cover measurements were taken on 9 May, 23 May, 16 July, 9 August, and 15 October. Wild plant species within the circles and their specific soil cover were also quantified.

2.5. Biological Laboratory Analysis

In the second phase, conducted 4 years after plot establishment, the evaluation of the Technosol quality prioritized soil biological parameters. It was focused on the analysis of soluble organic-C through new samplings conducted on 3 February and 27 April, as well as microbial activity analyzed from 29 May samplings. In February and April, composite soil samples were collected from three different points at a depth of 0–0.2 m in each plot. However, only two blocks were sampled. Soil water content was determined from subsamples using a forced-air oven due to the presence of gypsum. Samples from February were air-dried, and the fine fraction (particle diameter < 2 mm) was analyzed for water-soluble organic-C following the method in [41]. Twenty grams of the fine fraction were weighed into a 250 mL polycarbonate centrifuge tube and extracted with 200 mL of deionized water by shaking for 24 h. The extracts were centrifuged at 10 K rpm for 25 min and filtered through 0.45 μm membranes. Samples obtained in April were not dried; instead, the fresh sieved fraction (<2 mm diameter) was analyzed immediately after sampling, although soil water content was quantified as previously explained. Extraction was performed by shaking 12.5 g of soil with 50 mL of 0.5 M K_2SO_4 for 30 min at 200 rpm. The resulting extracts were filtered as described earlier. For both analytical methods, the extracts were stored at 4 °C and analyzed the following day. Total oxidizable C in the extracts was quantified using $K_2Cr_2O_7$ (1N) as an oxidizing agent in an acidic medium (H_2SO_4, concentrate), following the method in [42]. Excess $Cr_2O_7{}^{-2}$ was titrated with $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$, with orthophenanthroline as indicator. The amount of $Cr_2O_7{}^{-2}$ reduced during the reaction was assumed to be equivalent to the organic-C in the extracts. As a reference, the fine lignite and a fine fraction of mine spoil fractions used in the development of the substrate were also analyzed for organic-C content. In the samples collected on 29 May, a soil fraction (particle diameter < 6 mm) from each plot was reserved for microbial activity evaluation (soil respiration) using a manometric methodology described in [43,44]. Incubation at room temperature was initiated the following day in respirometer jars (1 L volume), and oxygen consumption was monitored daily for 1 week (until 3 June). Oxygen consumption data were converted to CO_2-C release. The lignite and the fine mine spoil fractions (diameter < 6 mm) used to create the Technosol substrate were also incubated. Prior to incubation, the moisture content per unit weight of moist soil was determined, and all samples were incubated at 65% of their water holding capacity.

2.6. Statistical Analysis

The data were statistically analyzed by using the statistical package SAS (v9.4) [45].

When sampling time was included in the analysis, the SAS System MIXED procedure [46] was selected. The use of the Akaike information criterion (AIC) [47] was chosen to compare the relative goodness-of-fit among non-nested candidate models. The AIC performed better when mulch treatment and sampling time effects were treated as fixed, while block was treated as random. We tested the homogeneity of variances and the normality of the distributions. Multiple comparisons of least squares means for the main effects and interactions were performed using the LSMEANS option.

When differences related to sampling time were not considered, analysis of variance (ANOVA) was performed. Separation of means was conducted with the Duncan Multiple Range Separation test ($\alpha = 0.05$). Linear equations were obtained and compared using the SAS REG procedure.

3. Results

The classification of the new Technosols according to IUSS Working Group WRB [48] is Hyperspolic Technosol (Siltic, Calcaric, Carbonic, Gypsiric, Ochric, Salic, Skeletic).

One of its characteristics is the high content of coarse fragments. The average values (by weight) and the standard deviation (±SD) were 62.3% (±4.6). Of these fragments, 34.4% (by weight) had an apparent diameter less than 20 mm, while the remainder fell within the range of 20 to 60 mm. The resulting mean bulk density (±SD), including coarse fragments, was 1597 kg m^{-3} (± 67).

3.1. Chemical Characteristics

Two years after establishment, all plots showed high salinity and NO_3^--N content (Figure 2), but there were no significant differences linked to slurry mulch treatment or sampling months (Appendix A, Table A1). In May, salinity (EC, 1:5) of the fine material ranged from 4.0 (1SL) up to 6.0 dS m^{-1} (2SL). The average mineral N (± standard deviation), expressed for the 0.25 m depth, was 1497 (±354) kg NO_3^--N ha^{-1}. In November, the salinity ranged from 4.1 (2SS) up to 5.2 dS m^{-1} (3SS), while the average mineral N was 928 (±711) kg NO_3^--N ha^{-1}.

In November, the characterization of the extract from saturated soil samples did not show significant differences between treatments (Table 2; Tables A2–A4) for any of the parameters studied.

The relationship between the sum of cations and the electrical conductivity of the saturated soil extract ranged between 11.4 and 15.1. The analysis of anions and cations (Table 2) showed that nitrates were dominant, along with other soluble salts of chloride, such as NaCl, $CaCl_2$, and $MgCl_2$.

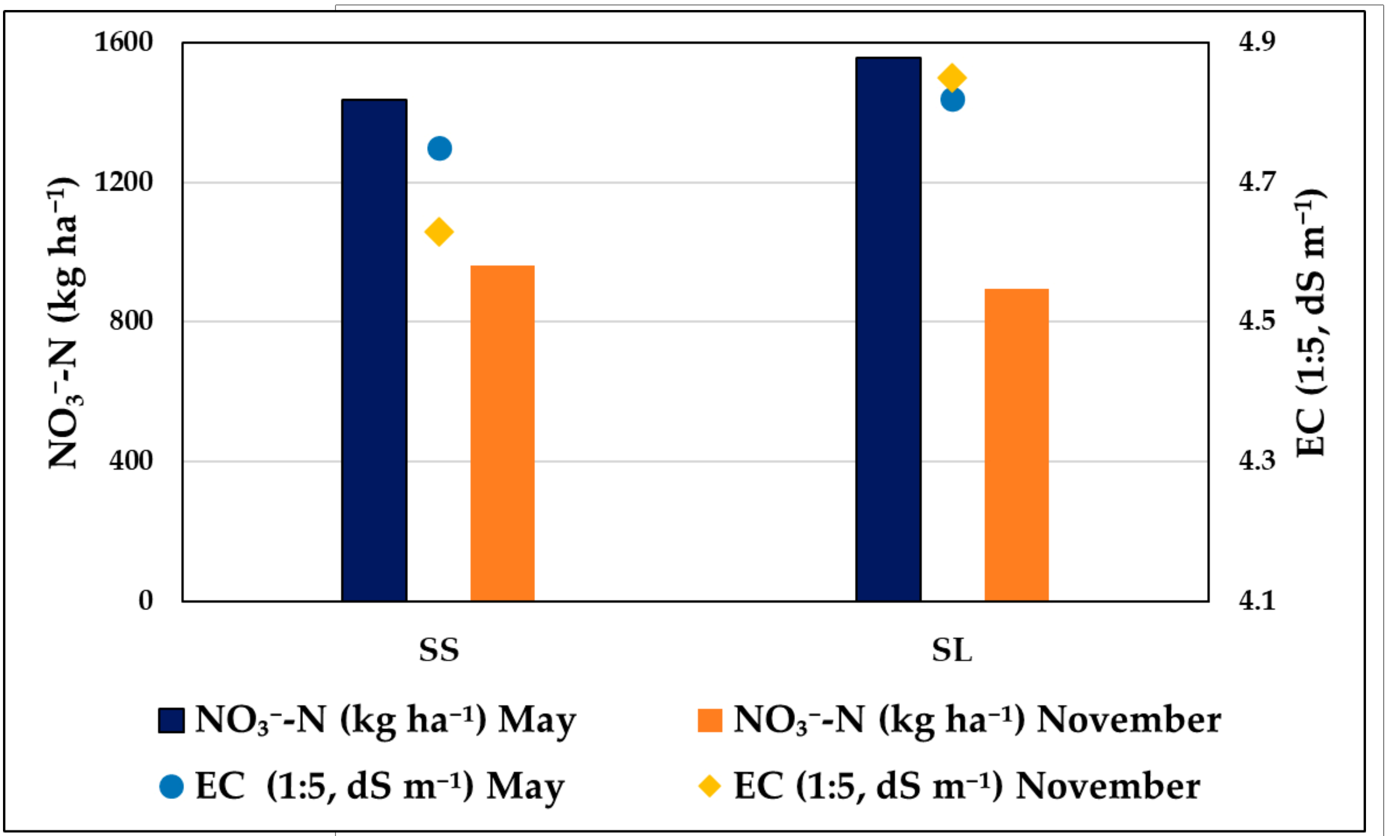

Figure 2. Average of mineral nitrogen (NO_3^--N) of the whole soil for 0.25 m depth, in May and November samplings, for the two studied treatments (slurry application over the surface (SS) or not (SL)), and after 2 years of plot establishment. Salinity average (electrical conductivity, EC 1:5) of the fine soil fraction (diameter < 2 mm) is also included.

Table 2. Average values [1] of water content in the saturated paste (Hs), pH (pHe) and electrical conductivity (ECe) of the extract from saturated soil paste (ECe), cation and anion concentrations in the same extract according to the different treatments: with (SS) or without (SL) slurry applied on the surface as a mulch. The mean value of the sodium adsorption ratio (SAR) is included.

Treatment	Hs	pHe	ECe	NH_4^+	K^+	Ca^{2+}	Na^+	Mg^{2+}	Cl^-	SO_4^-	NO_2^-	NO_3^-	HCO_3^-	SAR
	($g\ kg^{-1}$)		($dS\ m^{-1}$)					($mmol^c\ L^{-1}$)						($mmol^c\ L^{-1})^{0.5}$
SL	366.5	7.0	27.0	2.8	8.9	156.0	61.4	122.9	55.1	35.0	7.9	199.6	9.0	5.2
SS	384.3	7.0	26.6	4.2	9.7	170.4	62.2	124.6	59.8	35.3	8.8	220.1	9.3	5.1

[1] Analyses were performed 2 years after plot establishment, in November, and in the fine soil fraction (diameter < 2 mm). Carbonate content in the extract was absent.

3.2. Runoff Water, Nitrates, Erosion, and Vegetation Cover

Seven rain events were recorded during the study period, occurring on 31 May, 14 June, 11 July, 12 July, 15 August, 28 August, and 14 October, with associated rainfall amounts of 47, 38, 4, 10, 7, 51, and 38 mm, respectively. No statistical differences ($p > 0.05$) were found in the volume, EC, and nitrate content of the runoff water, nor in the amount of eroded sediments (Table 3). During the study period and averaging across both treatments (SL and SS), the total averages of runoff water, mineral N, and soil loss due to erosion were 1.3 L m^{-2}, 3.1 kg NO_3^-–N ha^{-1}, and 299 kg ha^{-1}, respectively.

Table 3. Averages (± standard deviation, SD) of runoff water volume, its conductivity (EC), and nitrate content according to the different treatments (Treat.): with (SS) or without (SL) slurry applied on the surface as a mulch, 2 years after plot establishment [1]. Data include the weight of eroded materials and are presented for different daily rainfall events (values in brackets).

Variable	Treat.	31 May (47 mm)	14 June (38 mm)	11 July (4 mm)	12 July (10 mm)	15 August (7 mm)	28 August (51 mm)	14 October (38 mm)
Runoff (mL m^2)	SL	325 (±57.8)	342 (±37.0)	7 (±0.0)	177 (±146)	7 (±0.0)	248 (±107)	209 (±128)
	SS	192 (±54.3)	342 (±74.0)	7 (±0.0)	71 (±61.1)	7 (±0.0)	256 (±44)	62 (±49.2)
	Means	251 b	342 a	7 d	124 c	15 d	252 b	136 c
EC ($dS\ m^{-1}$)	SL	5.4 (±2.7)	6.0 (±4.5)	7.0 (±1.6)	3.7 (±1.5)	4.8 (±2.5)	3.6 (±1.0)	3.0 (±0.2)
	SS	4.6 (±2.1)	3.0 (±0.5)	5.1 (±3.1)	2.3 (±1.4)	4.1 (±1.6)	2.1 (±0.4)	2.8 (±0.4)
	Means	4.8 ab	4.5 ab	6.0 a	3.0 b	4.6 ab	2.8 b	2.9 b
NO_3^- ($mg\ L^{-1}$)	SL	1560 (±1015)	2393 (±1897)	1220 (±1133)	907 (±793)	124 (±173)	269 (±247)	131 (±207)
	SS	1278 (±887)	2589 (±2727)	702 (±991)	558 (±632)	124 (±109)	137 (±180)	155 (±211)
	Means	1493 ab	2490 a	816 bc	733 bc	197 c	203 c	143 c
Erosion ($g\ m^{-2}$)	SL	12.8 (±13.1)	18.2 (±11.7)	0.5 (±0.5)	3.6 (±5.6)	0.1 (±0.0)	40.3 (±41.4)	4.4 (±5.2)
	SS	12.8 (±10.2)	17.6 (±10.7)	0.5 (±0.4)	1.1 (±1.3)	0.3 (±0.2)	9.2 (±4.2)	0.2 (±0.2)
	Means	12.7 ab	17.9 a	0.5 b	2.3 b	0.2 b	24.8 a	2.3 b

[1] Mean values from rainfall events with different letters are significantly different according to the LSD test ($p < 0.05$). No significant interactions existed between treatments and rainfall events for any of the studied variables.

Three main plant species were identified: *Atriplex halimus*, *Atriplex hastata*, and *Bassia scoparia*. Some individuals of *Limonium hibericum* Erben, *Salsola vermiculata*, and *Sonchus tenerrimus* were also present. Vegetation cover ranged from 12% in May up to 82% in August (Figure 3). No significant differences were associated with the slurry application as a mulch. Differences in soil plant cover and *Bassia scoparia* cover were related to time (Table A1).

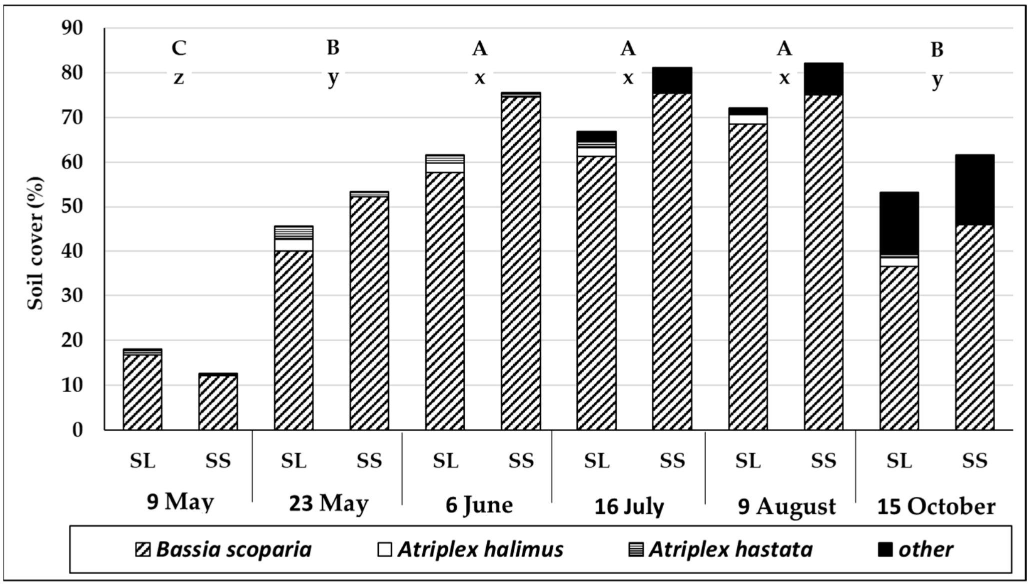

Figure 3. Evolution of soil cover after 2 years of plot establishment, from May to October, and for the three main species identified, and according to both treatments: with (SS) or without (SL) slurries applied on the experimental plots. Mean values of total soil cover or *Bassia scoparia* soil cover for the different sampling dates, marked with different letters at the top of the columns, were significantly different according to the LSD test ($p < 0.05$): (i) "A", "B", or "C" for total soil plant cover and (ii) "x", "y", or "z" for *Bassia scoparia* soil cover.

3.3. Water Extractable Organic-C and Soil Respiration

After 4 years of plot establishment, no significant differences ($p > 0.05$) were found between slurry mulch treatments in the quantity of organic-C extracted with water or potassium sulfate (Table A6). However, values in the created Technosols tended to be higher than those in the lignite powder or in the original mine spoils (Table 4).

Table 4. Total water-extractable organic-C (WEOC) in February, and total extractable organic-C with potassium sulfate (PSEOC) in April (±standard deviation, SD), 4 years after plot establishment. The samples were obtained from the fine lignite, the fine fraction of mine spoils, and the final substrates of the rehabilitated plots (with (SS) or without (SL) slurry applied over the surface).

	WEOC		PSEOC	
Sample	**(g kg^{-1})**	**SD**	**(g kg^{-1})**	**SD**
SL	0.19	0.07	0.34	0.07
SS	0.32	0.19	0.32	0.05
Lignite	0.16	-	0.15	-
Mine spoils	0.11	-	0.20	-

Microbial activity expressed as accumulated respiration over a one-week period (Figure 4) was described using linear equations (Table 5). The comparison of parameters from these equations (Table A7) revealed significant differences in the daily accumulated basal respiration rate between all materials, except between SS and SL. Fine lignite (powder) exhibited a significantly different rate compared to mine spoils, SS, and SL, but without significant differences in its intercept value. Mine spoils also had a significantly different rate than SS and SL, along with a significantly different intercept value in its linear equation.

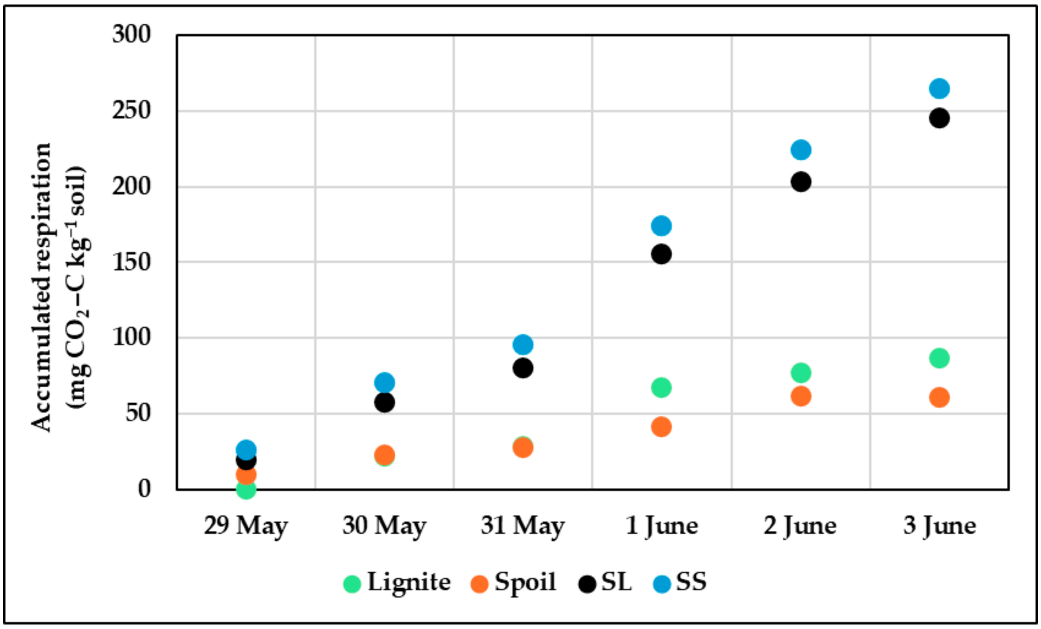

Figure 4. Evolution of the accumulated respiration over a one-week period for the fine lignite (powder), the mine spoils, and the Technosol with (SS) or without (SL) pig slurry applied on the surface and after 4 years of plot establishment.

Table 5. Linear equations describing the accumulated respiration (mg CO$_2$-C kg^{-1} soil) over time (day) and over a one-week period, for lignite, mine spoils, and the performed substrates with (SS) or without (SL) slurry applied on the surface 4 years earlier.

Material	Equation	Significance	R^2
Lignite	$y = -16.99 + 18.26$ time	0.0007	0.96
Mine spoils	$y = -1.17 + 10.99$ time	0.0008	0.96
SS	$y = -30.79 + 9.55$ time	<0.0001	0.98
SL	$y = -37.10 + 46.87$ time	0.0001	0.98

4. Discussion

The percentage range of coarse fragments in the field plots and the associated bulk densities were linked to the observed variability (by weight and size) of coarse fragments in mine spoils.

Two years after the construction of the Technosols, average mineral N levels (Figure 2) were much higher than those found in agricultural plots under fallow conditions in semiarid systems [49], despite the presence of coarse fragments (62%, w/w). The surface addition of pig slurry did not increase mineral N availability, likely because pig slurries contain about 75% of their N content in a mineral form [50], which has a low residual effect over time [51].

The substrate used for the reclamation of the mine spoils was made from inexpensive, locally abundant materials. These substrates inherited many key features from the materials used: they were calcareous, gypsiferous, saline, and rich in mineral N.

According to the analysis of the saturation extracts, the substrate is saline, as the ECe is much higher than 4 dS m^{-1} (Table 2). For revegetation purposes, it is important to identify salts that are more soluble than gypsum, as these are responsible for osmotic effects. Additionally, quantifying the presence of chloride and sodium is crucial due to their toxic effects on some plant species and the impact of sodium on soil structure. However, unlike typical saline soils [52], chloride and sulfate anions are not the principal soluble anions, in this substrate. The predominant salts were associated with nitrates, followed by highly soluble chlorides. Salinity has been exacerbated by the restoration process, primarily through the mineralization of organic N and the oxidation of ammonium forms. However, it is important to note that mine spoils were already saline due to their chlorine salt content (Table 3). Nitrates, which are very soluble, are a significant contributor to the osmotic pressure in the developed soil. Future studies should explore using smaller amounts of sewage sludge per hectare, to prevent the excessive accumulation of nitrates. Magnesium chloride, a highly soluble salt (353 g L^{-1}), is also present and can cause plant toxicity. Its presence is inherent to the composition of the materials used. Being highly hygroscopic, it can absorb water from the air [52]. In contrast, gypsum (calcium sulfate dihydrate), with limited solubility (around 2.3 g L^{-1}), dissolves readily when preparing saturated paste extracts. This dissolution contributes to higher electrical conductivity values (1 to 3 dS m^{-1} higher) in gypsiferous soils compared to non-gypsiferous soils with similar conductivity at field capacity.

Plants grown in gypsiferous soils can tolerate EC values approximately 2 dS m^{-1} higher than the standard tolerance [53]. The high EC values obtained in the saturated paste extracts can largely be attributed to nitrate salts, magnesium chloride, and, to a smaller extent, calcium sulfate. As the Mg/Ca ratio increases, sodification of the soil might be promoted in some scenarios, which can cause the dispersion of clays [54]. However, in this case, gypsum present in the soil mitigates the negative effects of sodium on soil structure. From a rehabilitation perspective, the presence of gypsum should be considered a favorable characteristic of this substrate. This conclusion is supported by the low SAR values observed (Table 3).

The observed species in the rehabilitated area were *Atriplex hastata, Atriplex halimus, Bassia scoparia, Limoniun hibericum* Erben, *Salsola vermiculata*, and *Sonchus tenerrimus* (Figure 3). Among these, the most common plant is *Bassia scoparia*, which grows in disturbed environments such as roadsides and agricultural fields in the Mediterranean region [55]. It has a low dormancy period and germinates at the beginning of spring, giving it an advantage over other competitor plants [56], a characteristic that explains its predominance from May onwards. Furthermore, it has a salinity tolerance mechanism that prevents sodium accumulation regardless of soil conditions [57]. The other two most common species were *Atriplex hastata* and *Atriplex halimus*. They belong to the same family (*Chenopodioideae*) as

Bassia scoparia and *Salsola vermiculata*, and they are considered halophytes. The *Atriplex* genus has different mechanisms to tolerate salinity, such as ion exclusion, adjustment of the cell osmotic potential, and stability of the cell structure and function, allowing plants in this genus to tolerate high ECe values (Table 2). The germination of *Limonium hibericum* Erben and *Salsola vermiculata*, which have similar behaviors, is influenced by the interaction of salinity and temperature factors. When salinity decreases and temperatures range between 10–20 °C, germination increases, which explains their presence in spring. However, concentrations above 200 mM of NaCl and temperatures between 25–35 °C might constrain or even inhibit their germination. The main appearance of these species between the end of summer and the beginning of autumn coincides with periods when salinity values and temperatures tend to decrease (Figure 2), two factors that allow them to successfully germinate. In contrast, the germination of *Sonchus oleraceus* is not affected by temperature, light, soil salinity, or seed depth. Under optimal conditions, it has a low dormancy level, with germination above 90% at seed maturity, although 12–14% of the seeds can germinate in darkness. At a concentration of 320 mM NaCl and above, germination is completely inhibited. However, even in soils with high salinity, a proportion of the seeds can still germinate [58]. The presence of all the mentioned species, which are tolerant to salinity, supports the trend in European countries of avoiding non-native species for sustainable reclaimed mine sites [59].

The mineral-N content measured as nitrate remained steady, showing no statistically significant differences between the initial and the final values. Nevertheless, the quantified nitrogen was consistent with the typical levels of mineralized nitrogen found in semiarid Mediterranean zones, which generally represent around 1% of the organic matter [60]. The control of runoff and erosion through soil plant cover limited nitrogen losses out of the system to just 3.1 kg NO_3^-–N ha^{-1} (over a period of 7 months), a reasonably acceptable outcome. It is worth noting that the maximum daily precipitation recorded was 51 mm (Table 3), a significant amount given that 75 mm of precipitation corresponds to a 10-year return period.

The established slope of 30% further demonstrated the feasibility of controlling soil erosion within acceptable levels through vegetation establishment (Table 3), confirming previous findings [61]. However, it was not possible to completely control runoff water. To prevent such losses from the system, the usage of a perimeter drainage to redirect it would be advisable [62]. Soil erosion from May to October was 799 kg ha^{-1} for the SL treatment and 417 kg ha^{-1} for the SS treatment. Considering that this six-month period encompassed only parts of the peak spring and autumn rains, doubling these figures provides a reasonable estimate of annual erosion losses. Tolerable erosion is considered to be the maximum value that a field can endure while maintaining economic sustainability for crop production. In this case, even though crop production was not the primary focus of this study, it can serve as a reference value. According to [63], the tolerance values in a case like this are 2200 kg ha^{-1} $year^{-1}$. Therefore, the soil erosion in this case was not excessive and remained below the commonly accepted tolerance levels in cultivated fields.

Four years after the construction of the Technosols, soil quality parameters clearly distinguished these new soils from the original fine lignite (powder) and spoil materials used in their construction. However, no significant differences were observed due to the surface application of slurry. The new soil differed from the soil overburden materials, mainly because of its microbial activity, which resulted in a higher accumulated respiration over the period studied (Figure 4, Table 5), and which indicated, in the mid-term, no adverse effects of the created substrate on soil health. In fact, similar findings have been reported by [64], who described the enhancement of soil respiration when using composted sewage sludge on degraded soils. This characteristic coincides with [65], who noted that

Technosols show high levels of biological activity, which contributes to their favorable evolution. In our case, it did so despite their high salinity (Table 2).

The increase in soluble organic-C content 4 years after the construction of the soil can partially be explained by the addition of slurry. Pig slurry contains aliphatic structures, mainly polyalkyl [66], that might have a weak effect on long-term C sequestration but enhance evolution of soil organic carbon. Furthermore, its low C/N ratio [50] likely facilitated lignite decomposition/evolution. In contrast, sewage sludge favors C sequestration, but its impact should be better evaluated in the long term [67]. Some variability in the obtained results for soluble organic-C was detected (i.e., WEOC values, Table 4), likely due to the heterogeneous distribution of materials. Despite this variability, the increase in soluble organic-C within the new substrates (Table 4) was evident. Soluble organic-C is also considered a potential risk if it reaches water bodies and groundwater; however, in this study, revegetation demonstrated its ability to control runoff water and soil erosion at acceptable levels (Table 3). These indicators reinforce the validity, in terms of soil quality, of rehabilitation procedures using saline materials mixed with organic and the ability of the Technosols to function effectively within their immediate environmental context.

5. Conclusions

The use of pig slurry as a mulch had no significant effect on the studied parameters related to the sustainability of the rehabilitation procedures.

Salinity emerges as a crucial conditioning factor in soil rehabilitation, largely determined by the chemical characteristics of the waste materials used. In this context, adaptation to salinity conditions, in our case, proved to be essential to enhance the system's long-term sustainability.

The study reveals that, while there was a high availability of mineral N 2 years after the construction of the Technosols, the overall N content remained constant. This stability can be attributed to an effective absorption of available nitrogen by plants and reduced runoff and lixiviation losses.

The Technosols were successfully colonized by salt-tolerant species that have effectively acted as a barrier against soil erosion and N losses. This colonization underscores the beneficial role of the establishment of salt-tolerant vegetation for the successful rehabilitation of degraded soils in semiarid environments where marginal saline materials and organic fertilizers are generally used to construct Technosols.

The quantification of extractable organic-C and microbial activity as quality indicators, 4 years after the construction of the Technosols, reinforces the sustainability (mid-term evaluation) of the rehabilitation actions performed.

Despite the large amounts of materials needed to build these Technosols, mining companies in semiarid degraded environments can effectively address rehabilitation procedures with low-quality residual materials (such as the saline ones) plus raw organic fertilizers. This approach offers a cost-effective solution that supports the adoption of nature-based restoration practices while preserving high-quality materials for arable soils.

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Appendix A

Table A1. Test of fixed effects [1] for mineral-N content ($NO_3{}^-$-N, kg ha^{-1}) and soil electrical conductivity EC (1:5, dS m^{-1}) and according to treatments of slurry surface application and month sampling.

		$NO_3{}^-$-N			EC			Soil Plant Cover			*B. scoparia* Cover	
Source	**df**	**Den df**	***p***	**df**	**Den df**	***p***	**df**	**Den df**	***p***	**df**	**Den df**	***p***
Month	1	2	0.14	1	4	0.87	5	202	<0.0001	5	202	<0.0001
Treatment	1	4	0.87	1	4	0.87	1	1	0.4363	1	1	0.2655
Treatment × Month	1	4	0.78	1	4	0.78	5	202	0.3443	5	202	0.3488

[1] df, degrees of freedom for the factor; Den df, the denominator degrees of freedom. The mixed procedure of SAS type III test was used.

Table A2. Analysis of variance of humidity at saturation (Hs, g kg^{-1}), pH of the saturated soil paste (pHe), and electrical conductivity of the extract from saturated soil (ECe, dS m^{-1}).

		Hs		pHe		ECe	
Source	**df**	**AnovaSS**	***p***	**AnovaSS**	***p***	**AnovaSS**	***p***
Block	2	1036.86	0.42	0	0.9	117.49	0.76
Treatment	1	475.26	0.38	0	1	0.24	0.97

Table A3. Analysis of variance of cations (mmol^c L^{-1}) present in the soil saturated extract.

		$NH_4{}^+$		K^+		Ca^{2+}		Na^+	
Source	**df**	**AnovaSS**	***p***	**AnovaSS**	***p***	**AnovaSS**	***p***	**AnovaSS**	***p***
Block	2	13.7	0.7	14.29	0.51	13609.14	0.65	887.77	0.53
Treatment	1	2.8	0.72	0.96	0.76	312.48	0.89	0.8	0.97

Table A4. Analysis of variance of anions (mmol^c L^{-1}) present in the soil saturated extract.

		Cl^-		$SO_4{}^{2-}$		$NO_2{}^-$		$NO_3{}^-$	
Source	**df**	**AnovaSS**	***p***	**AnovaSS**	***p***	**AnovaSS**	***p***	**AnovaSS**	***p***
Block	2	833.82	0.72	36.4	0.35	6.8	0.68γ	22028.3	0.74
Treatment	1	33.6	0.88	0.13	0.92	1.3	0.71	628.32	0.9

Table A5. Test of fixed effects [1] for mineral-N content ($NO_3{}^-$, mg L^{-1}), soil electrical conductivity EC (dS m^{-1}), erosion (g m^{-2}), and runoff water (mL m^{-2}) and according to treatments of slurry surface application and rainfall events (time).

		$NO_3{}^-$			EC			Erosion			Runoff Water	
Source	**df**	**Den df**	***p***	**df**	**Den df**	***p***	**df**	**Den df**	***p***	**df**	**Den df**	***p***
Time	6	23.1	0.003	6	22.1	0.04	6	22.4	<0.012	6	22.1	<0.0001
Treatment	1	23	0.54	1	3.99	0.24	1	1.98	0.35	1	3.99	0.21
Treatment × Time	6	23.1	0.99	6	22.1	0.84	6	22.3	0.33	6	22.1	0.10

[1] df, degrees of freedom for the factor; Den df, the denominator degrees of freedom. The mixed procedure of SAS type III test was used.

Table A6. Analysis of variance of total water extractable organic-C (WEOC) in February, and total extractable organic-C with potassium sulfate (PSEOC) in April, 4 years after plot establishment.

		WEOC		**PSEOC**	
Source	**df**	**AnovaSS**	***p***	**AnovaSS**	***p***
Block	1	0.03496	0.28	0.00075	0.14
Treatment	1	0.01769	0.37	0.00055	0.44

Table A7. Comparison of the lineal functions described in Table 5. Comparisons were set up using a fictitious variable (z) and are based on the following equation: Respiration = $\alpha + (\gamma \times z) + (\beta \times \text{time}) + (\lambda \times \text{time} \times z)$. The adopted value for z is 0 or 1 for the first and the second material compared in each line of the table, respectively.

Comparison [1]	α	γ	β	λ
Lignite vs. Mine spoils	0.03	0.11	<0.0001	0.01
Lignite vs. SS	0.13	0.36	0.0001	<0.001
Lignite vs. SL	0.13	0.20	0.0001	<0.0001
Mine spoils vs. SS	0.90	0.05	0.0016	<0.0001
Mine spoils vs. SL	0.90	0.02	0.0019	<0.0001
SL vs. SS	0.01	0.72	<0.0001	0.56

[1] SS: Substrate with (SS) or without (SL) slurry applied on the surface.

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