

Division - Soil In Space and Time | Commission - Soil Genesis and Morphology

Changes in chemical properties of a coal minesoil over two decades of reclamation with perennial grasses in Southern Brazil

Maria Bertaso de Garcia Fernandez^{(1)*} , Gláucia Oliveira Islabão⁽²⁾ , Bruna Cortez Farias⁽²⁾ , Emanuelle Soares Cardozo⁽¹⁾ , Adão Pagani Junior⁽¹⁾ , Tainara Vaz de Melo⁽¹⁾ , Ana Paula Knapp⁽¹⁾ , Pablo Miguel⁽¹⁾ , Luiz Fernando Spinelli Pinto⁽¹⁾ , Otávio dos Anjos Leal⁽³⁾  and Lizete Stumpf⁽¹⁾ 

⁽¹⁾ Universidade Federal de Pelotas, Programa de Manejo e Conservação do Solo e da Água, Pelotas, Brasil.

⁽²⁾ Instituto Federal de Educação, Ciência e Tecnologia do Rio Grande do Sul, Pelotas, Brasil.

⁽³⁾ Institute of Bio- and Geosciences - Agrosphere (IBG-3), Forschungszentrum Jülich GmbH, Jülich, Germany.

ABSTRACT: Mineral coal is a major global source of electricity, and in Brazil, it is primarily used when hydroelectric generation is limited by low rainfall. The Candiota Mine, located in Rio Grande do Sul State, is the largest coal deposit in both Brazil and Latin America, with estimated reserves of 1 billion tons. Minesoils, or Technosols, formed at the Candiota Mine are highly susceptible to acid mine drainage due to the characteristics of the parent material and the practices used for topographic recomposition of the mined land. Enhancing the fertility parameters of these minesoils is a key challenge for successful revegetation and restoration. This study aimed to evaluate changes in acidity and fertility parameters in the topsoil of minesoils after 8.6 and 20 years of revegetation with perennial grasses. We hypothesized that correcting acidity and fertility immediately after minesoil formation would have a lasting positive impact on pH and fertility properties, allowing grass establishment and an increase in minesoil carbon content. The experiment was set up in 2003, with treatments consisting of perennial grasses used for revegetation. Soil samples were collected from the 0.00–0.10 m and 0.10–0.20 m layers after 8.6 and 20 years of revegetation. Soil pH, exchangeable calcium (Ca^{2+}), magnesium (Mg^{2+}), available potassium (K^+), aluminium (Al^{3+}), and total organic carbon (TOC) content were measured. Additionally, potential cation exchange capacity at pH 7.0 (CEC), base saturation (V%), and aluminium saturation (m%) were calculated. Soil pH in the 0.00–0.10 m layer remained statistically unchanged from 8.6 to 20 years of revegetation, regardless of treatment. However, V% decreased and this was accompanied by a sharp increase in m%, from 0.6 % at 8.6 years to 7.2 % at 20 years of restoration. The content of Mg^{2+} decreased significantly in *Hemarthria altissima* (0.00–0.20 m layer) and *Cynodon dactylon* (0.10–0.20 m layer). On average, TOC content increased by 141 % in the 0.00–0.10 m layer and 97 % in the 0.10–0.20 m layer from 8.6 to 20 years of restoration. After 20 years of restoration, minesoil pH, nutrients, and TOC content were classified as adequate, confirming our hypothesis of long-term benefits from acidity and fertility correction. On one hand, our results indicate the success of liming, fertilization, and revegetation practices in this area, which could serve as a guideline for minesoil restoration strategies. On the other hand, the increase in acidity and m% suggests that additional interventions may be required in the upcoming years of restoration.

Keywords: soil fertility, minesoil reclamation, acid mine drainage, organic carbon accumulation.

* **Corresponding author:**

E-mail: mariabgfernandez@gmail.com

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INTRODUCTION

Mineral coal is a major source of energy to generate electricity in several countries. According to the International Energy Agency, global demand for coal in 2023 was 2.5 % higher than the previous year, reaching 8,687 million tons (IEA, 2024). In Brazil, coal is an alternative energy source when hydroelectric generation is compromised by rainfall deficit. Candiota Mine, in the Rio Grande do Sul State, is the largest coal deposit in Brazil and Latin America, with an estimated reserve of 1 billion tons (CRM, 2024). The method of coal extraction adopted in the Candiota Mine is open-pit mining, which has already led to the degradation of about 800 ha to supply the local thermoelectric power plant, which holds an electric generation capacity of 350 MW (CRM, 2024).

Coal open-pit mining involves the removal of vegetation, soil and rocks overlying the coal, culminating in the destruction of the ecosystem (Fabbri et al., 2021). Topographic reconstitution of the land in the Candiota Mine domain includes filling in the mining pit with the rocks and remains of unextracted coal, forming the overburden layer. Topographic reconstruction is completed with the deposition of a topsoil layer on top of the overburden layer (Pinto et al., 2020). This process originates soils classified as Technosols (WRB, 2015) or minesoils (Sencindiver and Ammons, 2000). Revegetation of the minesoils at the Candiota Mine is carried out concurrently with mining, and generally uses annual or perennial grasses, taking into account the typical vegetation of the local Pampa Biome (CRM, 2024).

Minesoils at the Candiota Mine are not suitable for agricultural activities due to their susceptibility to further degradation. Among other factors such as fragile soil structure (Lucas et al., 2019), this is associated with the presence of mine waste fragments within the first 0.40 m of soil (Stumpf et al., 2018), which confer adverse chemical properties to cropping and revegetation in general, such as extreme acidity, and low fertility (Thakur et al., 2022; Zhang et al., 2022).

Extreme acidity in minesoils is often caused by acid mine drainage (AMD), which occurs when the mine waste containing sulphurous minerals, such as pyrite (FeS_2), is exposed to oxygen and water, forming sulfuric acid (Bitencourt et al., 2015). Acid mine drainage is a major environmental hazard in post-coal minesoils (Albert et al., 2022) and results in strong minesoil acidification and release of high concentrations of Fe, Mn and Al in the minesoils (Sarmiento et al., 2018; Zhang et al., 2023).

At the Candiota Mine, many minesoils are susceptible to AMD, given that many areas reconstructed between 1970 and 1990 have not yet received a layer of topsoil on top of the overburden layer. Other areas constructed between 1990 and 2000 have received a thin layer of topsoil, meaning that the waste material potentially generating AMD is very close to the soil surface. Notably, from the 2000s onwards, the minesoils at the Candiota Mine were constructed including a layer of clay between the topsoil and the overburden layer, aiming to prevent AMD (Bitencourt et al., 2015).

Restoring the fertility parameters is among the main challenges of reclamation of minesoils. Liming and the use of corrective fertilizers, which have already been used with success in agricultural soils (Brignoli et al., 2024), can also be used with the purpose of improving minesoil chemical properties, while taking into account minesoil texture and plant species chosen for revegetation. For example, Dias (2024) observed that the application of dolomitic limestone and NPK chemical fertilizer (12–24–12) in a chronosequence of minesoils with sandy clay loam topsoil, in Mozambique, helped in the establishment of tree species used in revegetation. At the beginning of the restoration process, the minesoils exhibited similar and strong acidity ($\text{pH} < 5.0$) until 1.4 years, while from 5.3 to 10.6 years, minesoils exhibited medium (5.14) and moderate (6.14) soil pH. In addition, the N, K, and P levels also increased significantly from 0.5 to 1.4 years of restoration, when their values increased, respectively, from 4.06 to 9.34 mg kg^{-1} for N; from 15.51 to

33.15 mg kg⁻¹ for P; and from 4.02 to 49.49 mg kg⁻¹ for K. From this initial period onwards, all macronutrients remained stable until 10.6 years of restoration.

Successful revegetation of minesoils may favor soil fertility in the long-term because of the cycling of nutrients and the increase of Soil Organic Carbon (SOC) stocks exerted by vegetation (Ahirwal et al., 2017). In China, at the country largest opencast coal mine, Zhang et al. (2022) report that minesoil chemical properties, as soil organic matter, total nitrogen, pH, and available phosphorus, progressively improved with revegetation age over a 20-year chronosequence. The oldest minesoils, from 20 years of restoration, achieved pH and Total Organic Carbon (TOC) values similar to those of the natural soil. Accordingly, a progressive increase of minesoil SOC stocks with the age of reclamation was reported for post-lignite minesoils in Germany revegetated with forest species (Matos et al., 2012) and agricultural land (Zhao et al., 2022). This is in line with Iskandar et al. (2022), who found increased SOC, nitrogen and phosphorus content after a decade of minesoils reclamation with fast-growing pioneer plants and herbaceous cover crops in Indonesia compared with initial contents.

At the Candiota coal mine, the research group on minesoils at the Federal University of Pelotas (UFPEl) installed an experimental area in 2003 with the aim of monitoring, over the long term, the restoration of physical and chemical properties of a minesoil revegetated with perennial grasses, which are already known to be promising in the restoration of soils degraded by agriculture. As far as we know, this field experiment is the only long-term study designed with statistical rigor and dedicated to monitoring the progress of the restoration of coal-mined soils in the Pampa Biome. In this context, this study aims to evaluate the dynamics of acidity and nutrients in a minesoil constructed in 2003 using clayey topsoil – originally an *Argissolo Vermelho* – over a period of 8.6 to 20 years of revegetation with perennial grasses. The hypothesis is that correcting the acidity and fertility immediately after the construction of the minesoil still has a positive impact on the development of the root system of the plants established to carry out its ecological restoration.

MATERIALS AND METHODS

Study area

The study was conducted within the Candiota Mine area, located in Rio Grande do Sul State, Southern Brazil (Figure 1). The region has a humid subtropical climate (Cfa) with an average annual temperature of 17 °C and annual precipitation of 1,400 mm, characterized by cold winters and hot summers (Alvares et al., 2013).

The topographic reconstitution of the mined area occurred in early 2003 and was conducted with the deposition of nearly 0.40 m of topsoil on the overburden layer. Topsoil used consisted mainly of the B horizon of the original soil of the pre-mining area, an *Argissolo Vermelho Eutrófico típico* (Santos et al., 2018) or Rhodic Lixisol (Clayic) (IUSS Working Group WRB, 2015), with 465.50 g kg⁻¹ of clay and low soil organic matter (SOM) content (11.5 g kg⁻¹) and pH in water of 5.6 (Stumpf et al., 2016).

In November/December 2003, the randomized block design experiment with four replications (4 × 5 m² plots) was installed (Figure 1). Before initiating revegetation of the minesoil with the perennial grasses, the minesoil was scarified to a depth of 0.15 m and received dolomitic limestone (10.4 Mg ha⁻¹), as well as 900 kg ha⁻¹ of a 5–20–20 fertilizer (45 kg N, 180 kg P₂O₅, and 180 kg K₂O), based on soil analysis results. Treatments consisted of three perennial grasses cultivated individually in the experimental plots: *Hemarthria altissima* (Poir.) Stapf & C.E. Hubbard, *Cynodon dactylon* (L.) Pers. cv. Tifton, and *Urochloa brizantha* (Hochst. ex A. Rich.) Stapf.

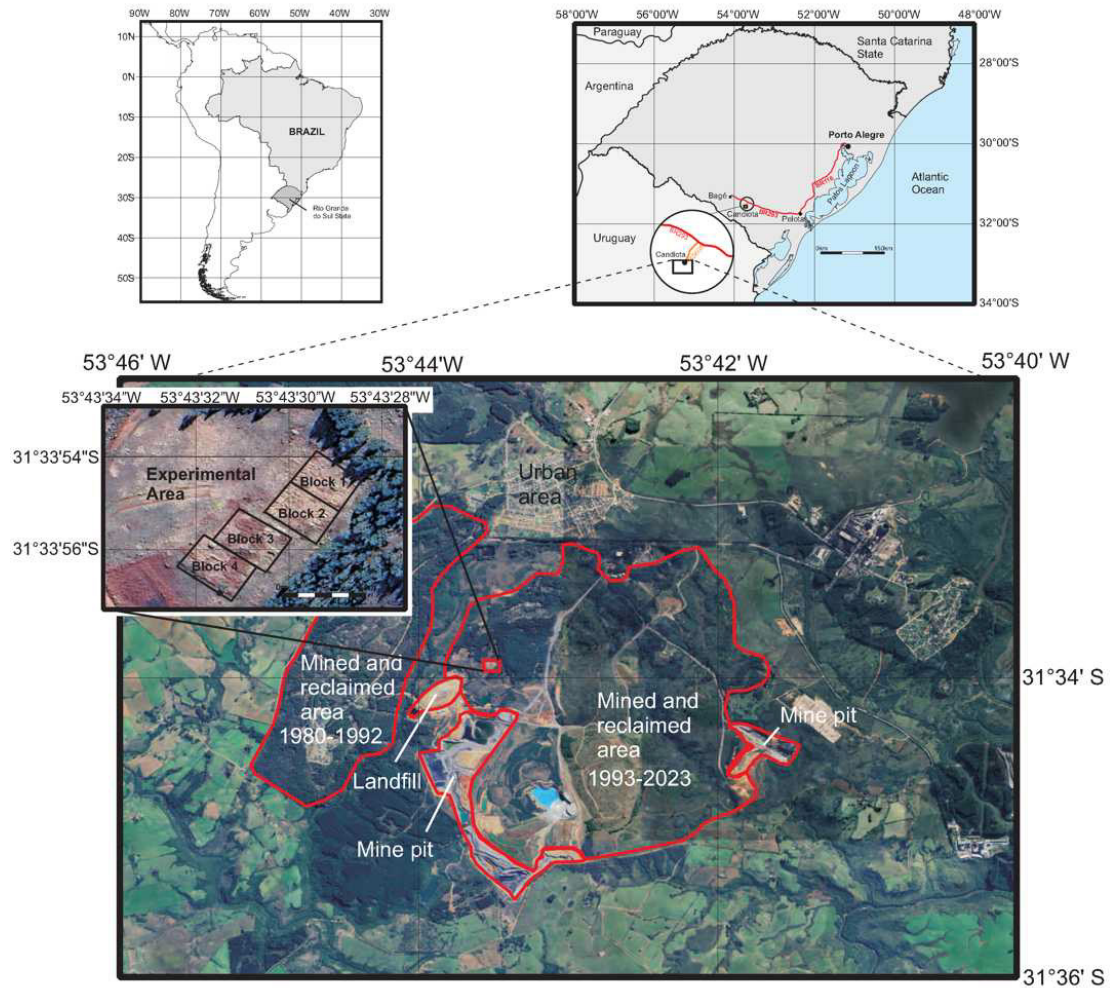


Figure 1. Experimental site location in the Candiota Coal Mine area in Southern Brazil. Source: Miguel et al. (2023).

Soil sampling and analyses

In October 2023, after 20 years of continuous minesoil revegetation, soil samples were collected at 0.00–0.10 and 0.10–0.20 m layers for the analysis of chemical soil properties. In total, 24 soil samples (3 treatments, 4 replicates, 2 layers) were collected with a shovel blade to determine the soil pH, calcium (Ca), magnesium (Mg), potassium (K), aluminum (Al), and total organic carbon (TOC) content.

Soil pH was determined using a 1:1 soil-to-water ratio. Exchangeable calcium (Ca^{2+}), magnesium (Mg^{2+}), and aluminum (Al^{3+}) were extracted with 1 mol L^{-1} KCl (Teixeira et al., 2017) and analyzed using atomic absorption spectrophotometry (Ca and Mg) and titration with NaOH (Al). Available potassium (K) was measured using the Mehlich-1 method and analyzed by flame photometry. Potential acidity was extracted with calcium acetate and determined by titration with NaOH. Sum of bases (SB) was calculated as $\text{Ca}^{2+} + \text{Mg}^{2+} + \text{K}^{+}$ content, and it was used to calculate the potential (pH 7.0) cation exchange capacity (CEC). The CEC was calculated as $\text{SB} + \text{Al}^{3+} + \text{H}^{+}$. Effective CEC (CECe) was calculated as $\text{SB} + \text{Al}^{3+}$, and it was used to calculate aluminum saturation (m%). The base saturation (V%) was calculated as $(\text{SB} \times 100)/\text{CEC}$, and the m% was calculated as $(\text{Al}^{3+} \times 100)/\text{CECe}$. These analyses and calculations were performed based on Teixeira et al. (2017).

Total organic carbon content was determined by the Walkley-Black combustion method, according to Teixeira et al. (2017). This method was applied because it is the official method used for TOC analysis of soil samples collected in the region of the experiment, according

to the Fertilization and Liming Manual for the States of Santa Catarina and Rio Grande do Sul (CQFS, 2016). Based on the average carbon content in SOM (58 %) (Stockmann et al., 2013), the TOC contents were multiplied by 1.724 and thereby converted to SOM content. The data obtained in the present study were compared with that obtained by Stumpf et al. (2016), when the experiment reached 8.6 years of implementation.

Statistical analysis

The data set passed the normality (Shapiro-Wilk) and homogeneity of variances (Levene) tests. Therefore, soil pH, V%, m%, CEC, and TOC content data were subjected to two-way repeated-measures analysis of variance (ANOVA), considering perennial grasses and restoration time as main factors. No significant interactions between grass and restoration time were observed for any of the properties. However, both factors –restoration time and grass species– were significant when considered individually. Therefore, statistical comparisons were performed separately for each factor: restoration time ($n = 12$; 3 grasses \times 4 replicates) and grass species ($n = 8$; 2 times \times 4 replicates). Means were compared by Tukey test at $p < 0.05$. One exception was $p = 0.07$ for TOC at 0.10–0.20 m, which was also considered significant ($p < 0.10$) and relevant for discussion.

One-way ANOVA was additionally performed (after normality and homogeneity of variances were confirmed) to reveal changes in the content of exchangeable Ca^{2+} and Mg^{2+} , as well as the available K^+ in each treatment from 8.6 to 20 years of restoration. In case ANOVA pointed out a significant effect of restoration time on these parameters, the averages within treatments were compared by Tukey test ($p < 0.05$).

Principal Component Analysis (PCA) was applied to the dataset to reveal shifts in the minesoil properties and their interrelations induced by the treatments (perennial grasses) over restoration time in each soil layer (0.00–0.10 and 0.10–0.20 m).

RESULTS

Perennial grasses and restoration time effect on soil chemical properties

Overall, all properties were affected by the factor “restoration time”, except for pH in the 0.00–0.10 m layer and m% in the 0.10–0.20 m layer (Table 1). The grass factor affected only CEC at 0.00–0.10 m layer (Table 1). Regarding Ca^{2+} , Mg^{2+} and K^+ content within treatments after 8.6 and 20 years, only Mg^{2+} changed significantly, regardless of soil layer (Figure 2a). These results for individual elements served mostly to aid in the interpretation of V% as a major fertility parameter.

In the 0.00–0.10 m layer, the minesoil pH ranged from 5.55 (*Urochloa brizantha* at 20 years) to 6.25 (*Hemarthria altissima* at 8.6 years), with no statistical differences among treatments or restoration times (Table 2). In contrast, V% showed a significant reduction over time, with mean values decreasing from 85.55 % at 8.6 years to 52.63 % at 20 years of restoration, considering the time averages ($n = 12$; Table 2). The inverse correspondence was observed for the time average m%, which increased from 0.59 % at 8.6 years to 7.18 % at 20 years of restoration (Table 2).

The CEC of the minesoil at 8.6 years of restoration in the 0.00–0.10 m layer varied over time, showing a significant increase from $10.97 \text{ cmol}_c \text{ dm}^{-3}$ at 8.6 years of restoration to $16.40 \text{ cmol}_c \text{ dm}^{-3}$ at 20 years, representing approximately a 1.5-fold increase. Among the grass species, a significant difference in CEC values was also observed, with *Urochloa brizantha* exhibiting the highest mean value ($16.14 \text{ cmol}_c \text{ dm}^{-3}$), which was approximately 1.3 times higher than that of the other grasses (Table 2).

Total organic carbon of the minesoil in the 0.00–0.10 m layer increased significantly during the restoration period, reaching more than twice its original value – from 9.04 g kg^{-1} at 8.6 years to 21.82 g kg^{-1} at 20 years – considering the time means. No additional significant differences were observed among treatments at this layer (Table 2).

Table 1. Two-way ANOVA p-values for the main factors grass type (Grass), time of restoration (Time), and their interaction (Grass*Time), for the different chemical properties of the soil evaluated at 0.00–0.10 and 0.10–0.20 m layer. Significant p-values are highlighted in bold letters

Soil properties	Grass	Time	Grass*Time
0.00–0.10 m			
pH	0.68	0.13	0.16
Base saturation (V%)	0.73	0.002	0.30
Aluminum saturation (m%)	0.19	0.04	0.17
Cation exchange capacity (CEC)	0.02	0.02	0.13
Total Organic Carbon (TOC)	0.15	0.01	0.89
0.10–0.20 m			
pH	0.66	0.01	0.57
Base saturation (V%)	0.63	0.006	0.57
Aluminum saturation (m%)	0.78	0.37	0.88
Cation exchange capacity (CEC)	0.50	0.02	0.77
Total Organic Carbon (TOC)	0.19	0.07	0.70

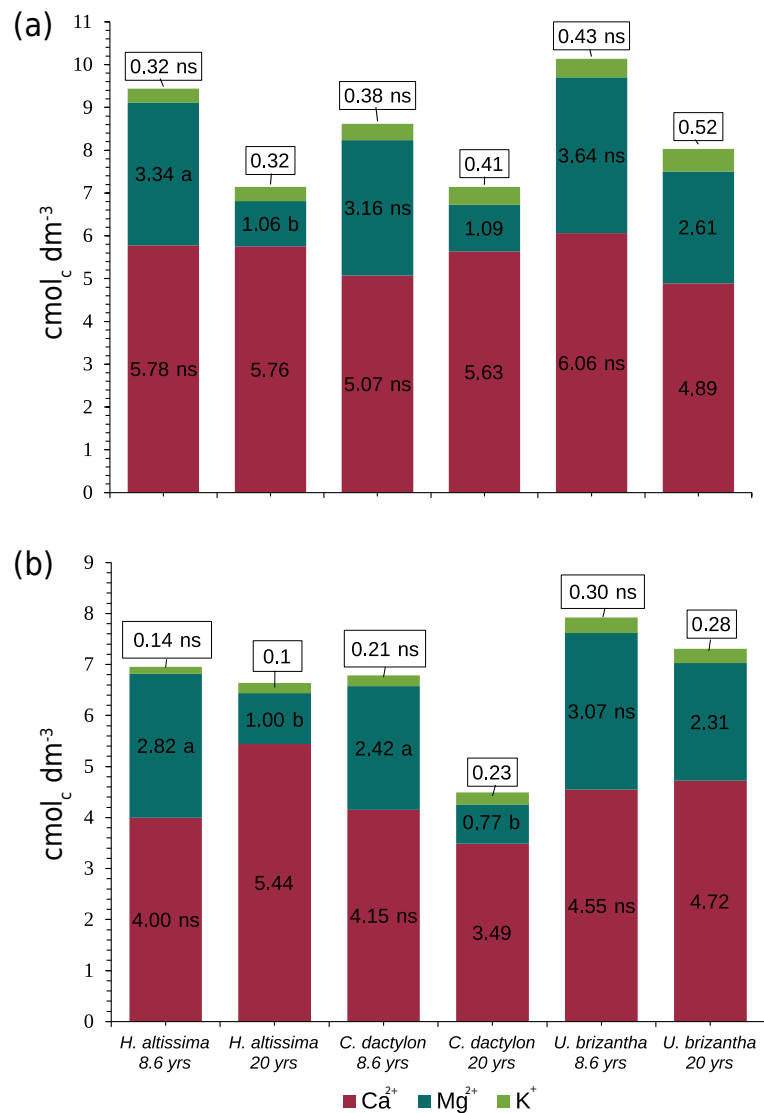


Figure 2. Content of exchangeable calcium, magnesium and available potassium in the 0.00–0.10 m (a) and 0.10–0.20 m (b) layer of a minesoil under restoration for 8.6 and 20 years with perennial grasses. Different letters for individual elements within treatment indicate statistical differences (Tukey test; $p < 0.05$) between 8.6 and 20 years of restoration; ns: not significant.

Table 2. Minesoil chemical properties in the 0.00–0.10 m layer at 8.6 and 20 years of restoration

Minesoil properties/Grasses	Time		Grass means
	8.6	20	
Soil pH			
<i>Hemarthria altissima</i>	6.25 ± 0.43 ^{ns}	5.87 ± 0.43 ^{ns}	6.06 ^{ns}
<i>Cynodon dactylon</i>	5.74 ± 0.35	5.88 ± 0.64	5.81
<i>Urochloa brizantha</i>	6.14 ± 0.50	5.55 ± 0.35	5.84
Time means	6.04 ^{ns}	5.77	
Base saturation (V%)			
<i>Hemarthria altissima</i>	88.88 ± 5.27 ^{ns}	58.16 ± 16.33 ^{ns}	73.52 ^{ns}
<i>Cynodon dactylon</i>	80.24 ± 9.04	53.77 ± 14.00	67.00
<i>Urochloa brizantha</i>	87.54 ± 8.69	45.96 ± 22.69	66.75
Time means	85.55 A	52.63 B	
Aluminum saturation (m%)			
<i>Hemarthria altissima</i>	0.37 ± 0.21 ^{ns}	4.74 ± 4.81 ^{ns}	2.56 ^{ns}
<i>Cynodon dactylon</i>	0.57 ± 0.38	3.64 ± 2.74	2.10
<i>Urochloa brizantha</i>	0.84 ± 0.76	13.18 ± 10.40	7.01
Time means	0.59 B	7.18 A	
Cation exchange capacity (CEC, cmol _c dm ⁻³)			
<i>Hemarthria altissima</i>	10.66 ± 0.93	13.95 ± 3.15	12.31 b
<i>Cynodon dactylon</i>	10.65 ± 1.78	14.54 ± 1.66	12.60 b
<i>Urochloa brizantha</i>	11.58 ± 1.26	20.70 ± 4.47	16.14 a
Time means	10.97 B	16.40 A	
Total Organic Carbon (TOC, g kg ⁻¹)			
<i>Hemarthria altissima</i>	8.40 ± 1.53 ^{ns}	21.09 ± 5.26 ^{ns}	14.74 ^{ns}
<i>Cynodon dactylon</i>	8.37 ± 1.97	20.09 ± 4.54	14.23
<i>Urochloa brizantha</i>	10.37 ± 2.71	24.28 ± 7.40	17.33
Time means	9.04 B	21.82 A	

Means of grasses (n = 4) followed by different lowercase letters within restoration time (column) or uppercase letters across restoration time (line) differ statistically according to Tukey's test. Time means (n = 12, line) or grass means (n = 8, column), followed by different uppercase letters differ statistically according to the Tukey test (p < 0.05). ns: not significant.

Unlike the upper layer, in the 0.10–0.20 m layer, the minesoil pH increased significantly from 5.29 to 5.81 between 8.6 and 20 years, considering the restoration time means (Table 3). Also, different from the upper layer, at the 0.10–0.20 m layer, the V% declined from 67.59 to 48.23 % considering the restoration time means (Table 3). In this layer the m% values ranged from 4.43 to 15.59 % without significant differences across treatments and restoration time (Table 3). The CEC increased significantly over the restoration period, rising from 10.9 cmol_c dm⁻³ at 8.6 years to 15.20 cmol_c dm⁻³ at 20 years – an increase of approximately 1.4 times, based on the restoration time means (Table 3). The TOC contents followed a similar trend, nearly doubling over the same period, from 6.35 to 12.50 g kg⁻¹, according to the restoration time means (Table 3).

Changes in Ca²⁺, Mg²⁺ and K⁺ content from 8.6 to 20 years of restoration in individual treatments

From 8.6 to 20 years of restoration, the Ca²⁺ content at the 0.00–0.10 m layer changed from 5.78 to 5.76 cmol_c dm⁻³ in *Hemarthria altissima*, from 5.07 to 5.63 cmol_c dm⁻³ in *Cynodon dactylon*, and from 6.06 to 4.89 cmol_c dm⁻³ in *Urochloa brizantha*, without statistical significance, regardless of treatment (Figure 2a). The same pattern was observed in the 0.10–0.20 m layer, where Ca²⁺ content across treatments and restoration time varied from 3.49 to 5.44 cmol_c dm⁻³, without statistical differences, regardless of treatment (Figure 2b).

Table 3. Minesoil chemical properties in the 0.10–0.20 m layer at 8.6 and 20 years of restoration

Minesoil properties/Grasses	Time		Grass means
	8.6	20	
Soil pH			
<i>Hemarthria altíssima</i>	5.34 ± 0.63 ^{ns}	6.11 ± 0.29 ^{ns}	5.73 ^{ns}
<i>Cynodon dactylon</i>	5.00 ± 0.56	5.65 ± 0.25	5.32
<i>Urochloa brizantha</i>	5.52 ± 1.17	5.68 ± 0.41	5.60
Time means	5.29 B	5.81 A	
Base saturation (V%)			
<i>Hemarthria altíssima</i>	67.31 ± 8.91 ^{ns}	56.08 ± 7.65 ^{ns}	61.69 ^{ns}
<i>Cynodon dactylon</i>	63.07 ± 5.49	40.28 ± 20.10	51.67
<i>Urochloa brizantha</i>	72.40 ± 20.29	48.34 ± 24.404	60.37
Time means	67.59 A	48.23 B	
Aluminum saturation (m%)			
<i>Hemarthria altíssima</i>	11.20 ± 12.91 ^{ns}	4.43 ± 4.55 ^{ns}	7.82 ^{ns}
<i>Cynodon dactylon</i>	15.59 ± 14.22	13.51 ± 11.87	14.55
<i>Urochloa brizantha</i>	12.40 ± 22.35	12.12 ± 17.01	12.26
Time means	13.07 ^{ns}	10.02	
Cation exchange capacity (CEC, cmol _c dm ⁻³)			
<i>Hemarthria altíssima</i>	10.34 ± 1.33 ^{ns}	13.27 ± 3.38 ^{ns}	11.80 ^{ns}
<i>Cynodon dactylon</i>	10.99 ± 3.53	14.76 ± 7.11	12.88
<i>Urochloa brizantha</i>	11.38 ± 1.85	17.57 ± 4.28	14.47
Time means	10.90 B	15.20 A	
Total Organic Carbon (TOC, g kg ⁻¹)			
<i>Hemarthria altíssima</i>	5.90 ± 0.18 ^{ns}	13.99 ± 9.84 ^{ns}	9.94 ^{ns}
<i>Cynodon dactylon</i>	4.81 ± 0.77	9.59 ± 2.09	7.20
<i>Urochloa brizantha</i>	8.35 ± 1.55	13.92 ± 5.25	11.13
Time means	6.35 B	12.50 A	

Time means (n = 12, line) followed by different letters differ statistically according to Tukey's test at p<0.05 (p<0.10 for TOC). ns: not significant.

In the 0.00–0.10 m layer, the content of exchangeable Mg²⁺ in *Hemarthria altíssima* treatment decreased significantly, dropping to nearly one-third of its value at 8.6 years (3.34 cmol_c dm⁻³) to 20 years (1.06 cmol_c dm⁻³) of restoration (Figure 2a). In contrast, in *Cynodon dactylon* and *Urochloa brizantha* treatments, the Mg²⁺ content remained statistically unchanged from 8.6 to 20 years of restoration (Figure 2a). In the 0.10–0.20 m layer, the Mg²⁺ content in *Hemarthria altíssima* and *Cynodon dactylon* treatments decreased significantly by 51 and 68 %, respectively, from 8.6 to 20 years of restoration, whereas in the *Urochloa brizantha* treatment, it remained statistically unchanged (Figure 2b). Overall, the K⁺ contents ranged from 0.14 to 0.52 cmol_c dm⁻³, and remained statistically unchanged from 8.6 to 20 years of restoration, regardless of treatment and soil layer (Figures 2a and 2b).

Principal component analysis of minesoil chemical attributes

Components 1 and 2 of PCA explained 70.43 and 14.51 %, respectively, of the variation in the dataset in the 0.00–0.10 m layer (Figure 3a). Component 1 clearly separated samples collected after 8.6 years of restoration to the left, along with pH and V% eigenvectors, whereas most samples collected after 20 years of restoration are positioned to the right of component 1 together with TOC, CEC and m% eigenvectors (Figure 3a). Overall, this PCA biplot reveals that minesoil acidity increased from 8.6 years to 20 years of restoration, and as a typical consequence of soil acidification, this was accompanied by an increase

in m% and a decrease in V%. Overall, this reflects the time elapsed since the single lime application at the start of the experiment. The TOC content increased from 8.6 to 20 years of restoration, which is known to explain the concomitant increase of CEC (Figure 3a). This shift in minesoil chemical properties was less remarkable in the *Cynodon dactylon* and *Hemarthria altissima* treatments, as revealed by the permanence of replicates of these treatments at the left side of component 1 after 20 years of restoration, which did not occur for *Urochloa brizantha* (Figure 3a). Notably, the TOC and CEC eigenvectors point out remarkably to *Urochloa brizantha* samples with 20 years of restoration, distinguishing this treatment from others with the same restoration time across component 2 (Figure 3a).

In the 0.10–0.20 m layer, the components 1 and 2 of PCA explained 54.45 and 33.35 %, respectively, of the variation in the dataset (Figure 3b). Unlike the upper layer, samples collected from the 0.10–0.20 m layer after 8.6 or 20 years of restoration were not clearly grouped on opposite sides of component 1. Overall, these observations indicate that similar shifts in minesoil chemical attributes occurred in the 0.10–0.20 m layer, but with lower intensity. The ordination of two replicates of *Urochloa brizantha* within the same PCA quadrant as TOC and CEC eigenvectors after 20 years of restoration resembles the chemical shifts observed in the upper layer in this treatment (Figures 3a and 3b).

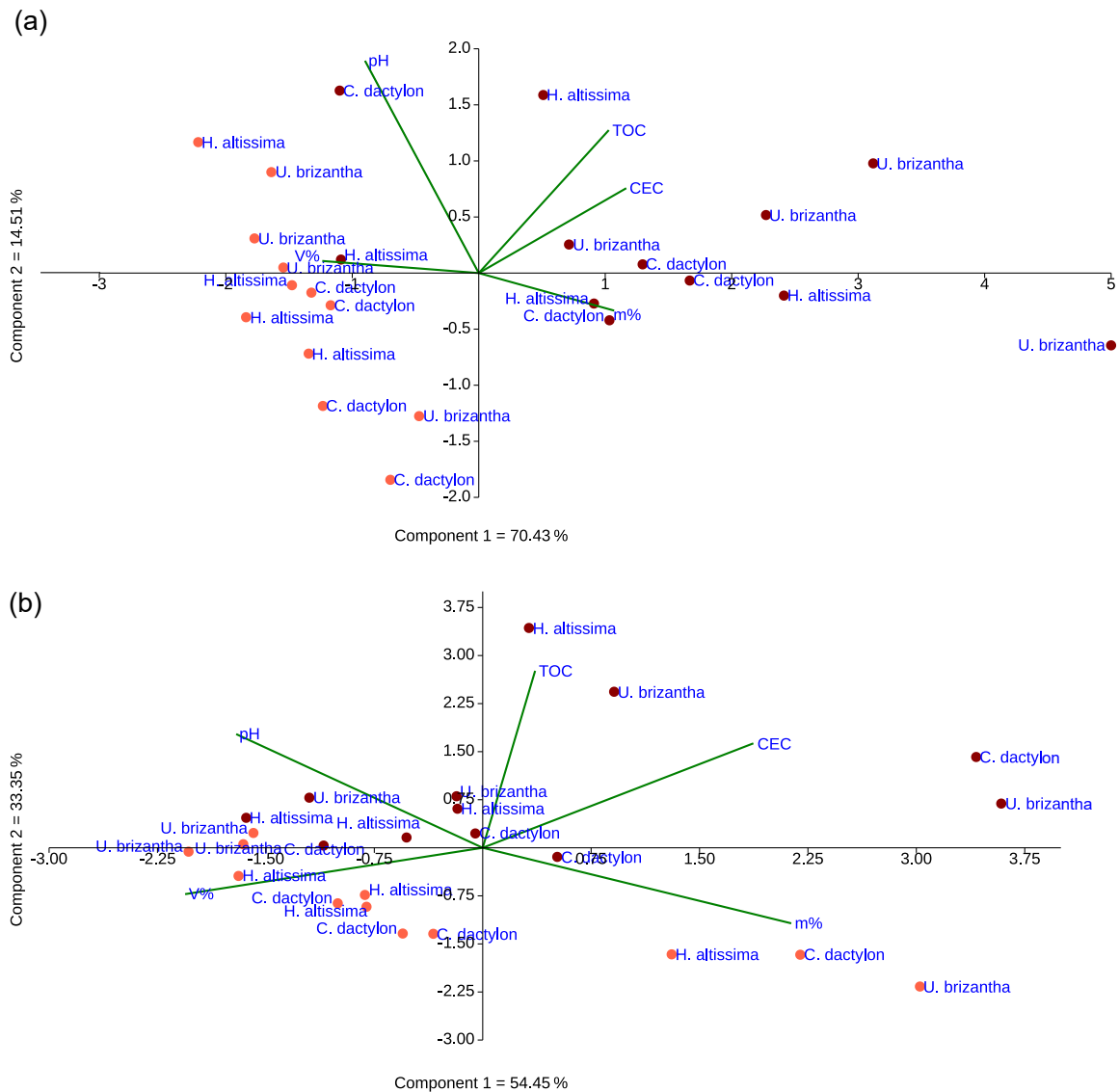


Figure 3. Principal Component Analysis integrating pH, base saturation (V%), aluminum saturation (m%), cation exchange capacity (CEC), and total organic carbon (TOC) content in the 0.00–0.10 m (a) and 0.10–0.20 m layer (b) at 8.6 (orange dots) and 20 years (brown dots) of restoration with perennial grasses (*Urochloa brizantha*, *Hemarthria altissima* and *Cynodon dactylon*).

DISCUSSION

Monitoring soil pH in mined areas is key to verifying the occurrence of acid mine drainage (AMD), which is influenced by the type of parent rock and overburden material added to the mining pit, as well as by the thickness of the topsoil deposited over the overburden layer (Bitencourt et al., 2015; Feng et al., 2019). Revegetation of post-coal minesoils that do not return to agricultural use is often conducted directly on the topsoil or overburden, without correction of minesoil acidity, expecting that plants will successfully adapt and promote the improvement of minesoil properties (Buta et al., 2019). On the other hand, when post-coal minesoils are destined for agriculture, corrections of acidity and fertility, along with initial restorative revegetation, have been shown to support satisfactory agricultural land use (Roy et al., 2023).

In our study area, inappropriate topographic recomposition of the land may lead to minesoil acidification because of the composition of the lithological profiles (Rio Bonito formation) (Albert et al., 2022). This is contrary to the alkaline pH of post-coal minesoils observed even after 60 years of revegetation in Germany, which is attributed to the characteristics of the lithological and reconstructed profile containing elevated carbonate contents (Zhao et al., 2022). In this context, the acidity correction performed in the experimental area before planting the perennial grasses was indispensable and proved to have long-term benefits.

Evidence of pH stability in surface soil and improvement in subsurface conditions was observed, with pH remaining constant from 8.6 to 20 years in the 0.00–0.10 m layer (Table 2), and increasing significantly in the 0.10–0.20 m layer (Table 3). This occurred despite the characteristics of the minesoil profile and an annual precipitation regime of 1,400 mm, which are likely to lead to soil acidification. According to the Fertilization and Liming Manual for the States of Santa Catarina and Rio Grande do Sul (CQFS, 2016), the average pH values observed in our study after 8.6 and 20 years of restoration (5.29–6.04) are slightly below or above the reference pH value for perennial summer grasses (>5.5). Particularly, the pH at the 0.00–0.10 m layer remained adequate, regardless of the restoration time, likely favoring root development and TOC increase over time (Table 2). Compared with this reference pH, the minesoil pH at the 0.10–0.20 m layer shifted from inappropriate to appropriate from 8.6 to 20 years of restoration. This is likely attributed to late root expansion below the 0.00–0.10 m layer, due to impeded compacted layers, as verified by Stumpf et al. (2016). This may have allowed for percolation of nutrients and liming reactions to correct pH in the subsoil (Wang et al., 2018; Feng et al., 2021). This long-term liming-assisted pH correction is corroborated by the maintenance of stable Ca^{2+} contents over time of restoration, regardless of treatment and soil layer (Figure 2). Additionally, it may be considered that stronger pH correction at a 0.10–0.20 m layer possibly occurred between 8.6 and 20 years of restoration, rather than only after 20 years, which was not captured in our study.

Domínguez-Haydar et al. (2019) studied a 20-year chronosequence of revegetated post-coal minesoils in Colombia and emphasized that maintaining minesoil pH within a recommended range is crucial to maintaining adequate contents of available nutrients for plants, thereby leading to successful revegetation and restoration of minesoil functions. In the present study, the tendency of decrease of Mg^{2+} contents after 20 years of restoration observed at 0.00–0.10 m and mainly at 0.10–0.20 m layer may be a response to the annual amendments of NPK fertilizer in the experimental area for a period of 18 years (2003 to 2021). Likely, the annual inputs of K^+ may displace Mg^{2+} from the cation exchange sites of the soil, making Mg^{2+} more prone to leaching. A similar relationship between K^+ and Mg^{2+} contents was reported by Thakur et al. (2022) in 20-year chronosequence of post-coal minesoil restoration in India. This was attributed to the restoration practices (i.e., fertilization), including predominant K^+ inputs combined with the acidity of the soil, which promotes H^+ retention in the soil cation exchange sites, thereby increasing Mg^{2+} susceptibility to leaching. According to Yan and Hou (2018), in the soil solution, these

two nutrients can compete for adsorption on mineral and organic particles with negative charges; therefore, the more K^+ , the less Mg^{2+} is retained in the soil.

Despite the decrease of Mg^{2+} contents over restoration time, the Mg^{2+} as well as the Ca^{2+} contents observed in our study are considered adequate ($Mg^{2+} = 0.7 \text{ cmol}_c \text{ dm}^{-3}$ and $Ca^{2+} \geq 3.4, \text{ cmol}_c \text{ dm}^{-3}$) according to the Fertilization and Liming Manual for the States of Santa Catarina and Rio Grande do Sul (CQFS, 2016). However, it is important to note that V% significantly decreased from 8.6 to 20 years of restoration, accompanied by a sharp increase of m% at the 0.00–0.10m layer (Table 2). This is likely associated with the decrease of pH in this layer (Table 2), which is known to increase Al^{3+} solubility and availability to plants, with possible restrictions to root growth (Mulazzani et al., 2024).

The relationship between pH and m% is clearly illustrated in the PCA biplot, in which the eigenvectors of pH and m% are ordered in opposite quadrants, meaning that m% increases when pH decreases (Figure 3), and monitoring changes in these properties can be key to deciding on the interventions needed in the coming years of restoration. The greater impact of m% on the surface is possibly due to several factors, such as the release of H from rainwater that infiltrates the topsoil and occupies the base spaces in the CEC (Takur et al., 2022), continuous fertilization with NPK on the surface of the area until the year 2021, which also promoted the release of H (Tkaczyk et al., 2020), and exudation of H by the root system of perennial grasses (Wang et al., 2018), which had greater volume in the 0.00–0.10m layer at 8.6 years of restoration (Stumpf et al., 2016) and increased TOC (Feng et al., 2021). Specifically, in this study, the increase in TOC was found to be positively correlated with m%, particularly in the 0.00–0.10 m layer (Figure 3).

The higher CEC in *Urochloa brizantha* compared with the other treatments at the 0.00–0.10 m layer likely reflects the tendency of higher TOC content in this treatment (Table 2). Organic carbon is the main component of SOM (Stockmann et al., 2013), which contains negatively charged functional groups. Thus, increasing TOC contents usually promotes an increase in CEC (Kebebew et al., 2022). In fact, this relationship between TOC and CEC in the studied minesoil is evident in the ordination of these eigenvectors within the same PCA quadrant, particularly highlighting the *Urochloa brizantha* treatment, especially in the 0.00–0.10 m layer (Figure 3). The remarkable increase of TOC content in *Urochloa brizantha* treatment compared with the other treatments (0.00–0.10 m layer) is probably associated with the capability of the plant to produce high biomass production aboveground (Baptistella et al., 2020) and belowground, as confirmed by higher root density in this treatment compared with the other (Stumpf et al., 2016). Moreover, Fernandez et al. (2023) and Oliveira et al. (2025) observed a greater abundance of mesofauna in this treatment, which may contribute to the incorporation of biomass into the soil and thus to the increase in TOC (Pandey et al., 2022).

Accordingly, Singh et al. (2023) reported an increase in TOC contents in response to the enhancement of biological activity in a minesoil in India after 10 years of restoration. In general, the TOC contents increased significantly over restoration time, regardless of treatment and soil layer (Tables 2 and 3). This finding aligns with those of Zhang et al. (2022) and Zhao et al. (2022), who reported a significant increase in TOC in chronosequences of minesoils in China and Germany, respectively. The time average SOM content observed in our study at the 0.00–0.10 m layer (37.6 g kg^{-1}) is classified as intermediate (26.0 to 50.0 g kg^{-1}), whereas that at the 0.10–0.20 m layer (21.6 g kg^{-1}) is classified as low ($\leq 25.0 \text{ g kg}^{-1}$) according to the CQFS (2016).

CONCLUSION

After 20 years of restoration with perennial grasses, the minesoil maintained an adequate pH and high levels of Ca^{2+} , Mg^{2+} and K, demonstrating the long-term benefits of lime application down to a depth of 0.20 m. It is likely that, these chemical soil conditions

enabled satisfactory aboveground biomass production and root system expansion down to a depth of 0.20 m, leading to a more than twofold increase in TOC content in the 0.00–0.20 m layer between 8.6 and 20 years of restoration, with no distinction among the grasses.

The controlled soil acidity and improved fertility of the minesoil over two decades of restoration are remarkable and suggest that the liming and fertilization practices adopted in our experiment are effective in promoting successful revegetation and could be applied to newly formed minesoils in Candiota. Furthermore, root growth and TOC increase along the minesoil profile may alleviate soil compaction, a typical critical challenge in minesoils, ultimately creating favorable conditions for the recovery of soil biological activity and ecological functions. This study can serve as a reference for future research that monitors changes in the chemical properties of minesoil influenced by grasses, supporting decisions on suitable species for revegetation. Evaluating changes in pH, base saturation, and total organic carbon content over time is essential for tracking minesoil recovery.

DATA AVAILABILITY

The datasets generated and analyzed during the current study are available from the corresponding author upon reasonable request.

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DECLARATION OF COMPETING INTEREST

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

AUTHOR CONTRIBUTIONS







Conceptualization:  Maria Bertaso de Garcia Fernandez (lead).

Data curation:  Bruna Cortez Farias (equal),  Gláucia Oliveira Islabão (equal),  Lizete Stumpf (equal),  Maria Bertaso de Garcia Fernandez (equal),  Adão Pagani Junior (equal) and  Tainara Vaz de Melo (equal).

Formal analysis:  Ana Paula Knapp (equal),  Gláucia Oliveira Islabão (equal) and  Maria Bertaso de Garcia Fernandez (equal).

Funding acquisition:  Lizete Stumpf (equal) and  Pablo Miguel (equal).

Investigation:  Lizete Stumpf (lead).

Methodology:  Adão Pagani Junior (equal),  Ana Paula Knapp (equal),  Bruna Cortez Farias (equal),  Gláucia Oliveira Islabão (equal),  Maria Bertaso de Garcia Fernandez (equal) and  Tainara Vaz de Melo (equal).





Project administration:  Luiz Fernando Spinelli Pinto (lead).

Resources:  Lizete Stumpf (lead).

Supervision:  Otávio dos Anjos Leal (equal) and  Pablo Miguel (equal).

Validation:  Gláucia Oliveira Islabão (lead).

Writing - original draft:  Lizete Stumpf (equal) and  Maria Bertaso de Garcia Fernandez (equal).

Writing - review & editing:  Emanuelle Soares Cardozo (equal),  Lizete Stumpf (equal),  Maria Bertaso de Garcia Fernandez (equal) and  Otávio dos Anjos Leal (equal).

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