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Attenuation of Acid Mine Drainage in a Coal Waste Deposit in Southern Brazil and the Prospect of Transitioning from Active to Passive Treatment

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Abstract

Capão da Roça, located in the municipality of Charqueadas, is one of the few areas of coal tailing deposits at the surface within the State of Rio Grande do Sul, Brazil that generates acid mine drainage (AMD). Over the course of 2007, the landfill was characterised in detail, and an active treatment plant involving pH neutralisation and metal precipitation operations was implemented to meet emission standards for mine water. In that year, based on the sulphur mass balance, it was estimated that the process of AMD generation would last for approximately two decades. The objective of this work was to study the temporal evolution of the parameters of the raw AMD. The effluent was analysed for 17 years on a monthly basis in regard to pH, acidity, metals (Fe, Al, and Mn), and sulphates. The results indicated an increase in pH (from 2.1 to 4.7), a decay in the concentration of metals (from 177.8 to 0.1 mg L^{-1} for iron, 29.0 to 0.1 mg L^{-1} for aluminium, and 3.1 to 0.6 mg L^{-1} for manganese), sulphates (from 2023 to 307 mg L^{-1}), and acidity (from 539.5 mg $CaCO_3$ L^{-1} to 3.96 mg $CaCO_3$ L^{-1}), which were adjusted to a first-order kinetic model in agreement with observations at some other mining sites. Over the years, the active lime neutralisation–precipitation treatment system has been efficient in treating the effluent. Today, most water quality parameters already meet emissions standards; however, the AMD treatment plant is still necessary to prevent pH fluctuations and to reduce the concentrations of manganese. For this reason, a transition from an active to a passive treatment system was considered. Pilot scale studies confirmed that channels filled with gravel-size limestone or slag enable the neutralisation/increase in the pH of the effluent and remove residual amounts of some metals, resulting in an effluent with no level of toxicity to the microcrustacean *Daphnia magna*.

Keywords: mine water; temporal evolution; effluent management

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Academic Editor: Carlito Tabelin

Received: 25 August 2025
Revised: 7 October 2025
Accepted: 9 October 2025
Published: 11 October 2025

Citation: Keller, F.S.; Boff, C.; Silva, D.; Grigorieff, A.; Weber, C.C.; Weiler, J.; Schneider, I.A.H. Attenuation of Acid Mine Drainage in a Coal Waste Deposit in Southern Brazil and the Prospect of Transitioning from Active to Passive Treatment. *Minerals* **2025**, *15*, 1068. https://doi.org/10.3390/min15101068

1. Introduction

Acid mine drainage (AMD), also known as acid rock drainage (ARD), remains one of the most concerning environmental problems arising from mining operations. It forms as the result of the chemical oxidation of metal sulphides in the presence of atmospheric air and water, and their relationships with the surrounding environment [1–3]. Many methods for the mitigation of AMD have recently been developed from various perspectives [4–23].

When considering approaches to controlling AMD, its evolution and attenuation over time are a particular topic of concern for mine water management, since the longevity of mine water pollution forms the basis for decision making. This is a complex topic, as it is affected by geographical, geological, and climatic conditions as well as the mining process [24]. Furthermore, despite AMD lasting decades or centuries, many sites have not been systematically monitored in order to provide a consistent dataset to enable the assessment of the evolution processes involved. In particular, there is a special lack of studies in tropical regions.

Efforts have been made to monitor, understand, and model the water quality over time in the areas surrounding mining sites, including deep underground coal mines [25–29], decommissioned open cast coal mines [30], coal waste rock piles [31], and open pits and ore tailings from polymetallic sulphides [32–36]. Some aspects already appear aligned, such as the first-order decay model for acidity, metal, and sulphate concentrations, and the concept of first flush applied to water percolation in deep coal mines.

Another important aspect concerns the transition from active to passive treatment systems. Active systems require continuous operation, such as a chemical treatment plant, while passive systems operate without strict control, such as engineered channels or wetlands. The design and performance of various passive systems have been the subject of several review articles [4,5,22] and field applications [35,36]. Some studies have considered the use of open channels with limestone [37,38]. Some studies have considered the use of open channels with limestone [39–41], steel slags [42–46], or even available neutralising materials (e.g., low grade ores, fly ash, and concrete wastes) [47], and a highly detailed summary concerning diverse materials is provided in ref. [5]. In essence, AMD is neutralised and oxidised by the flow of water through an open channel filled with an alkaline material, which causes the precipitation of metal hydroxides. Armouring by Fe-bearing minerals (such as goethite and lepidocrocite) represents the main drawback, as the efficiency of the system decreases [48,49]. To avoid the phenomenon of coating, it is recommended that the system operates at high flow rates, with slopes of around 20%. Aside from armouring, passive techniques are inefficient or only partially effective for the removal of geochemically mobile pollutants like arsenic, selenium, boron, sulphate, manganese, zinc, and copper [47].

In the southern states of Brazil, AMD is associated with coal mining and takes place on a huge scale in the carboniferous region of Santa Catarina state and on a lesser scale in the states of Rio Grande do Sul and Paraná. This paper addresses the evolution of a small waste deposit in the State of Rio Grande do Sul, in the county of Charqueadas, known as Capão da Roça, which was an area of waste disposal for residues from coal preparation in the region of Baixo Jacuí, Rio Grande do Sul, during the 1970s and 1980s.

Figure 1 shows a timeline of the actions that have been carried out at this waste site. Coal processing was carried out in dense medium cyclones to be used in the direct reduction process at the Aços Finos Piratini Co. Steel Company (Rio Grande Do Sul, Santa Catarina, Brazil, no longer in operation). Part of the processed coal was also sent to a nearby thermoelectric plant. Coal was initially supplied by Poço Otávio Reis, an underground mine in the municipality of Charqueadas, and later by the Recreio surface mine, located in the municipality of Butiá, both in Rio Grande do Sul state, Brazil [50]. Coal preparation was carried out at the metallurgical plant, so the waste deposited at Capão da Roça was exclusively the material unsuitable for the metallurgical process or for burning in the thermoelectric plant. The activity of coal waste deposition ended in 1988, and restoration of the area began to a small extent in 1999. In 2006, with the aim of predicting AMD generation, the volume of waste was measured and the materials were characterised in terms of their net potential generation of acid. Based on the sulphate concentration in the acid drainage, it was estimated that AMD would continue to be generated for approximately two decades. In 2007, action was taken to mitigate AMD generation: the tailings pile was covered, drainage was contained and redirected, and a water treatment plant was established. The covering for the pile consisted of a layer of bottom ash from a nearby thermoelectric power plant and a 40 cm layer of soil. The ash had a slightly alkaline characteristic, and it was hoped that this would neutralise the acidity, which ultimately proved unsuccessful. The AMD was retained in a dam, and the effluent quality parameters were analysed before and after treatment, from 2008 onwards.

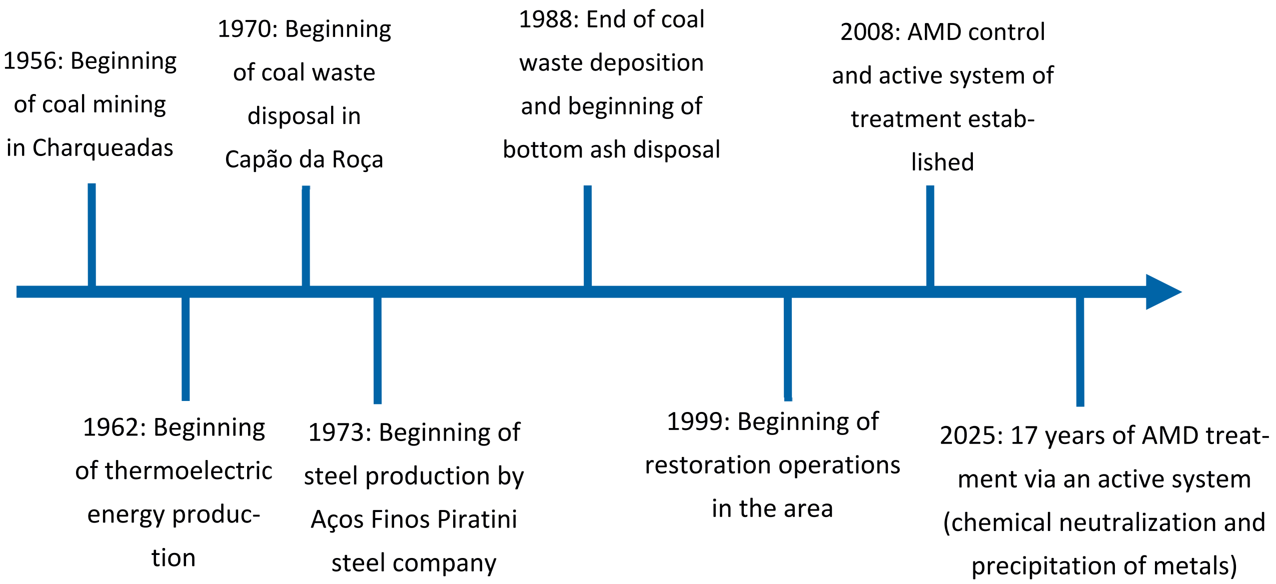

Figure 1. Timeline of the main actions carried out at the Capão da Roça waste site.

The aim of this study was to assess the evolution of the characteristics of AMD over time at a coal tailing dump in Capão da Roça, Brazil. We also considered the possibility of transitioning the effluent treatment from an active treatment via chemical neutralisation/metal precipitation to a passive treatment system involving channels containing limestone or iron steel slag. The study was conducted using a monitoring database throughout the AMD attenuation process and under the operational conditions of the treatment plant. It is hoped that the results presented here for a relatively small-scale waste dump site, in terms of both the volume of material and time, will assist in the management of coal mining waste and AMD in other regions of Brazil and the world, particularly in regions with hot and humid climates.

2. Materials and Methods

2.1. Study Area

The coal waste deposit is situated in the county of Charqueadas, Rio Grande do Sul state, Brazil, with coordinates 29°57′17″ S and 51°37′31″ W, approximately 30 m above sea level (Figure 2). The material originated from coal preparation at Leão-Butiá Gondwana Coal Fields, with particle sizes of up to 4.1 mm. It is ranked as a subbituminous coal, with a high ash content and sulphur content varying from 0.3% to 11.5%. A detailed characterisation of the regional geochemical and petrological considerations and trace elements can be found in work by Kalkreuth et al. [51]. The climate is subtropical and humid, with relatively warm summers and cool winters (the hottest months being January and February, max 31 °C and min 21 °C, the coldest months being June and July, max 20 °C and min 10 °C, on average). Average rainfall is about 1350 mm per year and is generally well-distributed throughout the year. Details of the local weather conditions can be found in ref. [52]. Regarding the geomorphology, it is a plain region, without pronounced slopes. The coal deposit occupies an area of 37.1 ha, with the thickness of the coal tailings reaching 3 m, which gives an approximate volume of 1,150,000 m^3 and a waste mass of 1,500,000 metric tons (considering a bulk specific weight of 1.3 t m^{-3}). Water flow occurs through the entry of rainwater and subsurface water, which is stored downstream in a nearby reservoir with an area of 3 ha and pumped daily to the effluent treatment plant. Based on the pumping flow rate, the average detention time in the reservoir has been estimated as two months.

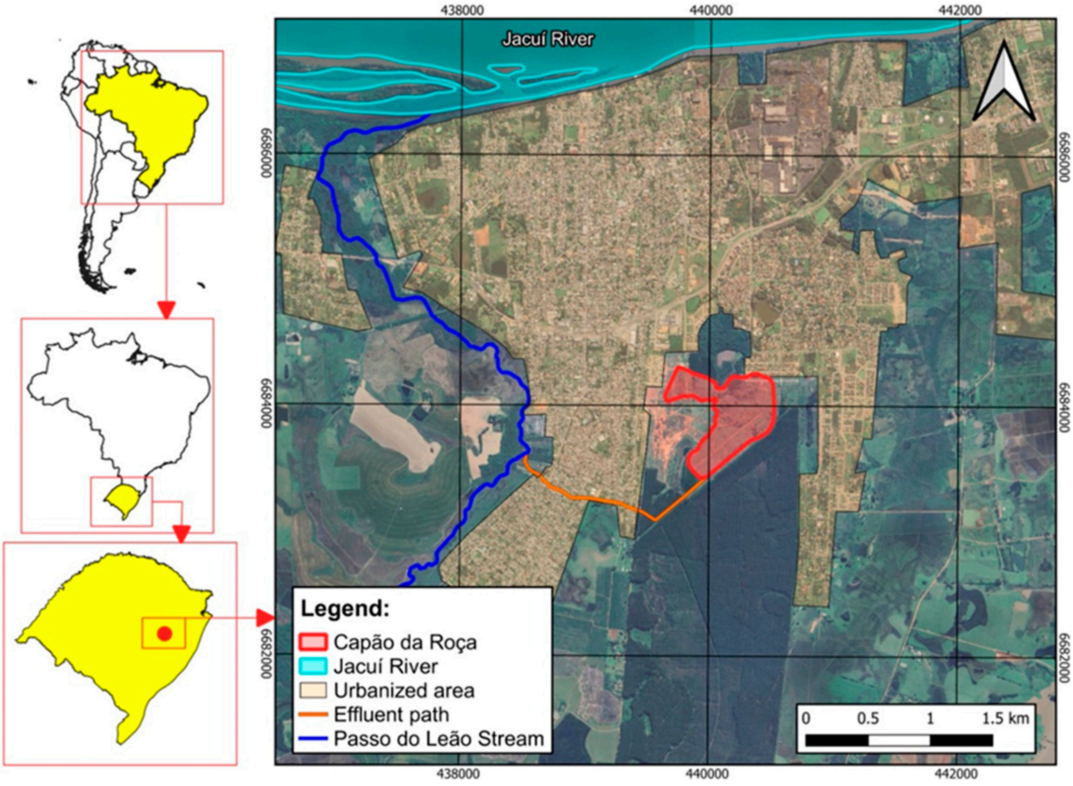

Figure 2. Location of the Capão da Roça coal waste deposit in Rio Grande do Sul, Brazil.

2.2. Materials Characterisation

Tailings samples were collected and a materials analysis was performed based on three samples of coal waste and three samples of the bottom ash and topsoil used to recover the coal waste mass.

The bulk density (or apparent density) and porosity were calculated following ASTM D167 [53]. The total sulphur content was determined using a sulphur analyser (LECO S144-DR, Criciúma, Brazil). The acid–base accounting (ABA) method EPA 1994 [54] was used to predict AMD generation. If sulphide minerals are present, there is potential for acid generation, whereas if caustic minerals are present, then there is potential for acid neutralisation. This test calculates the acid generation potential (AP) for the sample and also measures its neutralisation potential (NP). The AP was determined from the total sulphur content (wt.%), based on the predominance of pyritic sulphur (>80%). Stoichiometric balance shows that one mole of $CaCO_3$ is required for neutralisation of one mole S_{total} (Equation (1)).

$$AP\left(kgCaCO_3t^{-1}\right) = \left(\frac{1000}{32}\right) \times TotalSulphur(wt.\%) \tag{1}$$

The NP was determined by applying acid digestion to consume the neutralising minerals (1 h digestion at 90 °C), followed by titration with NaOH until a pH of 7.0 was reached (Equation (2)).

$$NP\left(kgCaCO_3t^{-1}\right) = \left([HCl], gg^{-1}ofsample\right| \times \left(\frac{50}{36}\right) \times 1000\right) \tag{2}$$

These data were used to calculate the net neutralisation potential (NNP) (Equation (3)).

$$NNP\left(kgCaCO_3t^{-1}\right) = NP - AP \tag{3}$$

Calculation of the acid generation potential took into consideration the following criteria [54,55]:

NNP < -20 kg $CaCO_3$ t^{-1} indicates acid generation;

NNP > +20 kg $CaCO_3$ t^{-1} indicates no acid generation;

-20 kg $CaCO_3$ t^{-1} < NNP < +20 kg $CaCO_3$ t^{-1} means there is no clear indication as to whether or not acid generation will occur.

2.3. Historical Data Evaluation and Mathematical Modelling

A historical survey of analytical data was carried out in order to identify the main pollutants and their concentrations in the raw acid drainage in Capão da Roça. This survey was based on technical reports provided by Copelmi Mineração Ltda. Mining Company, over the period February 2008 to May 2025, with monthly measurements of pH, acidity, metals (total iron, aluminium, and manganese), and sulphates of the effluent at the inlet of the acid water treatment plant.

A mathematical model was created based on these data, with the concentrations of metals and sulphate adjusted as function of time. This regression was performed numerically using Minitab® Statistical Software Version 22.3.0 to obtain a generic expression as follows:

$$C(t) = C_0 \times e^{-kt} \tag{4}$$

where

C—concentration as function of time (mg L^{-1});

C_0—initial concentration (mg L^{-1});

k—rate constant ($days^{-1}$ or $years^{-1}$);

t—time (in days or years).

2.4. Active Treatment

The acid drainage collection basin and the effluent treatment plant are shown in Figure 3. The treatment plant has been operating at a flow rate of 40 m^3 day^{-1}, 20 h per day, since February 2008. The pH is first raised to 8.7 by adding an alkaline reagent (calcium hydroxide, $Ca(OH)_2$) to precipitate the metals as hydroxides. This is followed by the addition of anionic polyacrylamide for flocculation and the settling of the flocs in ponds. Raw and treated effluent has been collected monthly and prepared for analysis, with the aim of meeting the emission standards established by the CONSEMA resolution 355/2017 [56] for the release of effluents into water bodies in the regional context. The results obtained for the control parameters required by the aforementioned resolution were grouped into different operating periods, to allow for the evaluation over time and to count the cases of non-compliance.

Figure 3. Photographs of the Capão da Roça coal waste site taken in 2010, Charqueadas county, Rio Grande do Sul, Brazil: (1) coal waste deposit, (2) dam for acidic water retention, (3) laboratory, (4) preparation of reagents in containers, (5) mixing tanks, (6) baffled flow unit for the formation of flocs, (7) settling ponds, and (8) results of the chemical neutralisation process shown in an Imhoff cone.

2.5. Passive Treatment Test

In 2022, studies involving passive treatment in open channels were carried out at the bench scale (Figure 4). Two drainage systems were created, one with calcitic limestone and one with a laden furnace slag from a steel plant. The main characteristics of the materials are listed in Table 1, and the X-ray pattern of both materials can be found in the Supplementary Material (Figures S1 and S2). The drainage systems were made from PVC gutters 12.5 cm in diameter, 1.5 m in length, and with a slope of 0.2%. Acidic water from the Capão da Roça site was added at a controlled rate of 0.1 L min^{-1}, providing a detention time of approximately 20 min, with the flow controlled by valves located downstream and upstream (Figure 4). In this experiment, 5 kg of alkaline material (limestone or slag) was used in each channel, which occupied approximately one quarter of the channel section. The water column was kept at 5.5 cm above the bottom of the gutter, so that all particles were submerged, providing a bed volume of approximately 7 L along the entire channel. Tests were conducted by feeding the system with acidic water with a volume corresponding to 10 times the bed volume. The consumption of limestone and slag was calculated based on the acidity and flow rate of the effluent and the neutralisation potential of the materials.

Figure 4. Acid drainage treatment experiments involving open channels with limestone (**left**) and slag (**right**) at the bench scale.

Table 1. Characteristics of the limestone and steel slag used in the drains.

Parameter	Limestone	Slag
Particle size distribution (mm)	9–25	9–25
Bulk density—ρ (t m^{-3})	1.45	1.78
Void ratio—e (%)	50	41
Elemental analysis carried out by FRX (%)		
CaO	56.05	36.31
SiO_2	nd	19.92
Fe_2O_3	0.07	18.7
MgO	1.46	11.59
Al_2O_3	nd	6.95
MnO	0.03	4.94
TiO_2	0.02	0.83
P_2O_5	0.01	0.34
Na_2O	nd	nd
K_2O	nd	nd
Neutralisation potential (kg $CaCO_3$ t^{-1})	954.8	731.8
Crystalline compounds detected by XRD	calcite (99.6%) quartz (0.4%)	wustite (63.4%) magnetite (22.1%) hematite (14.5%)

nd—not detected.

2.6. Chemical and Toxicological Analysis

The raw and treated water were analysed throughout the study for pH, acidity, metals (Fe, Al, and Mn), sulphate content, and toxicity to the micro crustacean *Daphnia magna*. The pH of the suspension was measured using a bench pH meter (AKSO model 86505, São Leopoldo, Brazil). Metal analyses were carried out by sample preservation with HNO_3 and using an issuing optical spectrophotometer with inductively couple plasma (Perkin Elmer model Optima 8300, Porto Alegre, Brazil). The sulphate content was measured via turbidimetry. All analyses followed the procedures described in the publication "*Standard Methods for the Examination of Water and Wastewater*" [57].

Acute toxicity tests for *Daphnia magna* were performed in triplicate, in accordance with the guidelines for testing chemicals using *Daphnia* sp. OECD 202 [58], using the statistical technique EPA 821-R2-012 [59]. This test assesses the immediate toxic effects of a sample on the mobility of this microcrustacean species, and is based on the exposure of organisms to a series of dilutions of the sample over 48 h. The effect on the organisms is expressed as a loss of mobility. The result was calculated by a statistical procedure and represented by the concentration effect on 50% of the population (EC_{50}-48 h).

3. Results and Discussion

3.1. Coal Waste Characterisation

The characteristics of the coal waste, bottom ash, and natural soil in the Capão da Roça coal waste deposit are given in Table 2. The waste has an average pyritic sulphur content of 1.29%, giving an average AP of 39.6 kg $CaCO_3$ t^{-1}. The neutralisation potential of 0.3 kg $CaCO_3$ t^{-1} is almost non-existent, resulting in a negative net acidity generation potential (NNP) of −39.3 kg $CaCO_3$ t^{-1}, indicative of acid generation. The bottom ash and the topsoil used to cover the coal waste do not have sufficient neutralisation potential to change this condition.

Table 2. Characteristics of the coal waste at the Capão da Roça deposit and the top layers of material in 2007.

Material	Sample	Total Sulphur (wt.%)	AP (kg $CaCO_3$ t^{-1})	NP (kg $CaCO_3$ t^{-1})	NNP (kg $CaCO_3$ t^{-1})
Coal waste	1	0.52	16.2	1.0	−15.2
	2	1.79	56.0	0.0	−56.0
	3	1.56	46.7	0.0	−46.7
	Average	1.29	39.6	0.3	−39.3
Bottom Ash	1	-	-	26.8	26.8
	2	-	-	21.1	21.1
	3	-	-	31.3	31.3
	Average	-	-	26.4	26.4
Covering soil	1	0.12	3.8	0	−3.8
	2	0.13	4.0	0	−4.0
	3	0.07	2.2	0	−2.2
	Average	0.11	3.3	0	−3.3

3.2. Temporal Evolution of AMD Parameters

Since the effluent under study originated from a coal waste deposit, the parameters monitored were pH, metals (iron, aluminium, and manganese), sulphate, and acidity.

The evolution of pH over time is shown in Figure 5. CONSEMA resolution number 355/2017 [52] states that the pH of an effluent must be between five and nine. It can be seen that in the initial years, the pH fluctuated between 2.0 and 3.5, with a gradual increase over time, meaning that pH adjustment was required up to the year 2022 for effluent release into the receiving water body. In 2023 and 2024, some samples had already reached a circumneutral pH, an effect that was mainly associated with the loss of strength of the AMD generation process. However, it should also be noted that in 2023 and 2024, historic floods occurred in the state of Rio Grande do Sul, which may also have contributed to the rise in pH [60,61].

Figures 6–8 show the evolution in the concentrations of iron, aluminium, and manganese over time, which undergo exponential decay. It is worth mentioning that the emission limits for wastewater discharge into water bodies are 10 mg L^{-1} for iron and aluminium, and 1 mg L^{-1} for manganese. With a few exceptions, the reference value for iron emissions has been reached since 2016 and, in the case of aluminium, since 2013. Manganese has undergone a significant decline, although the values still fluctuate substantially above and below the threshold value established by legislation.

The decline of elements can be associated with multiple factors. It can be mentioned that the pyrite oxidation process, metal's solubilisation from the waste rock mass in acidic media, and the reactions of metals in aqueous media, is strongly influenced by the medium pH. The formation of insoluble hydroxides of iron, aluminium, and manganese occurs at pH values above 3.5, between 5 and 9, and above 9 [62], respectively, and it plays an important role. In can be seen in the graphs, that in recent years, water pH is in a proper condition for iron precipitation, nearby for aluminium, and apart for manganese, which explains the absence of iron and the remaining amounts of aluminium and manganese in the raw AMD.

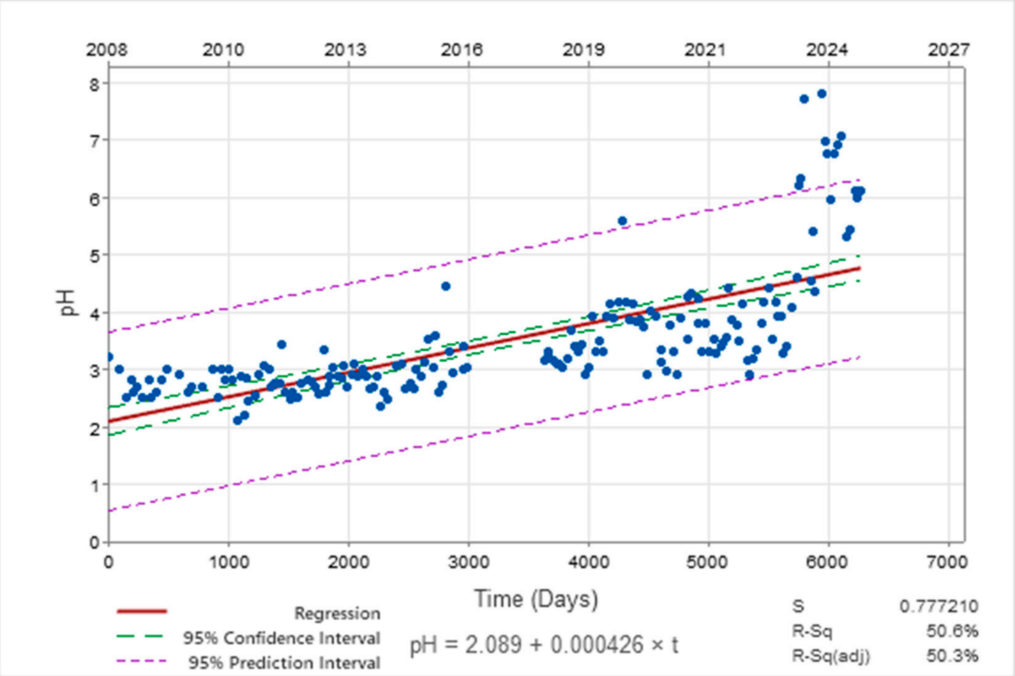

Figure 5. Evolution of downstream pH (measured values as blue dots) at the Capão da Roça coal waste site.

Like iron, sulphate ions are a direct product of pyrite oxidation, although they are more soluble in water. Sulphate concentration has also decreased over time (Figure 9), indicating that pyrite oxidation is occurring and the acidification process is losing intensity. During the period studied here, sulphate concentrations ranged from 3581 mg L^{-1} to 54 mg L^{-1}, with average values of around 2000 mg L^{-1} in 2008, rising to 400 mg L^{-1} after 2020. It is estimated that 5420 tons of sulphate were leached during this time, corresponding to the oxidation of 2820 tons of pyrite. The variation can be partly explained by seasonal trends, which affect bacterial activity, rainfall, and evaporation rates.

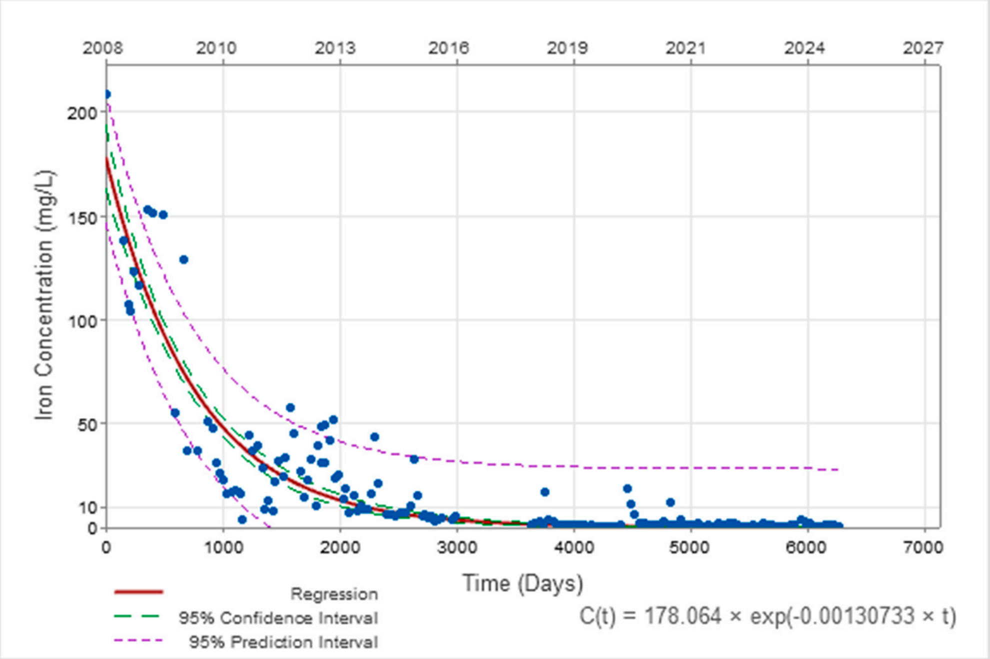

Figure 6. Downstream total iron concentration (measured values as blue dots) at the Capão da Roça coal waste site.

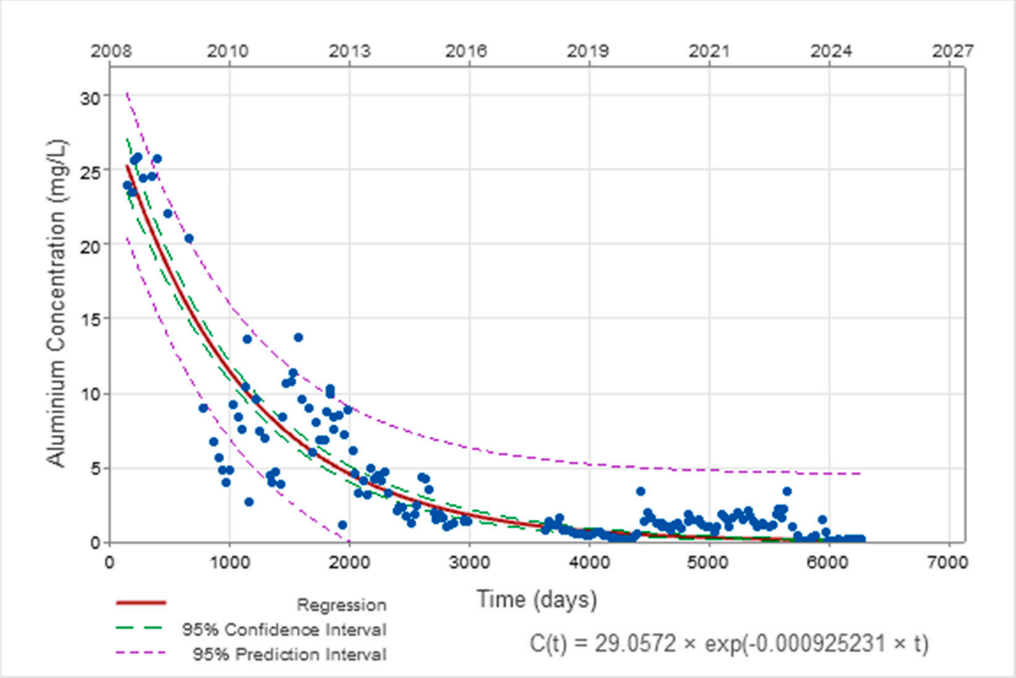

Figure 7. Downstream total aluminium concentration (measured values as blue dots) at the Capão da Roça coal waste site.

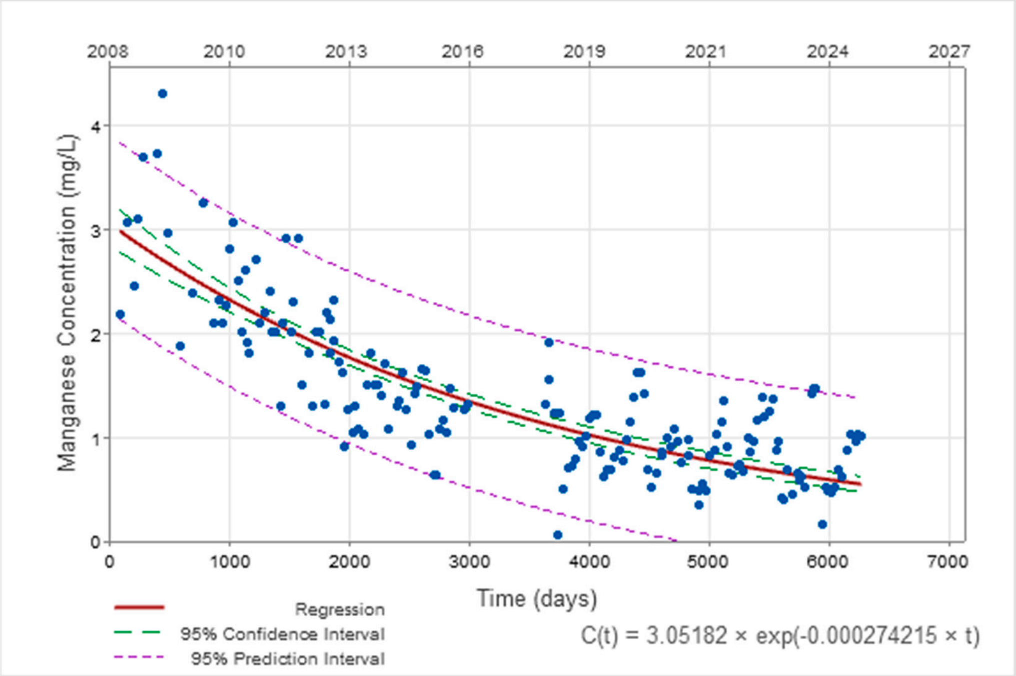

Figure 8. Downstream total manganese concentration (measured values as blue dots) at the Capão da Roça coal waste site.

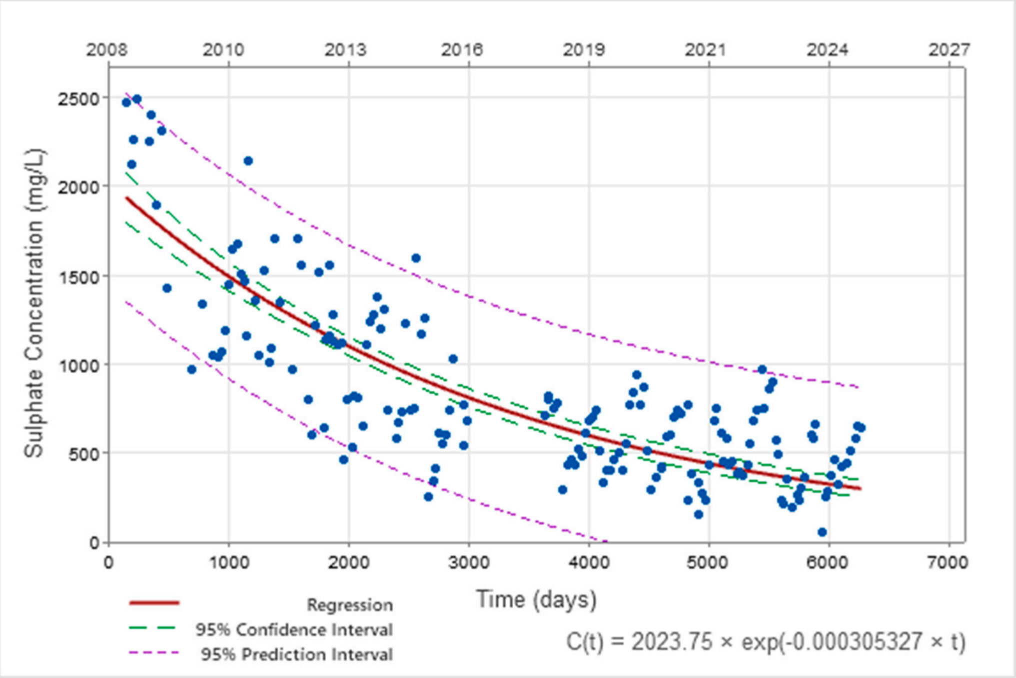

Figure 9. Downstream sulphate concentration (measured values as blue dots) at the Capão da Roça coal waste site.

The increase in pH and the decrease in the concentration of metals over time promoted a reduction in the total acidity of the effluent (Figure 10). The average consumption of the neutralising reagent, which in 2008 corresponded in $Ca(OH)_2$ to the equivalent of 550 mg of $CaCO_3$ L^{-1}, decreased to no more than 100 mg of $CaCO_3$ L^{-1} after 2020, with typical values in more recent years ranging from 10 to 30 mg of $CaCO_3$ L^{-1}. The main practical benefits were reductions in the cost of the alkalising reagent and the volume of sludge generated.

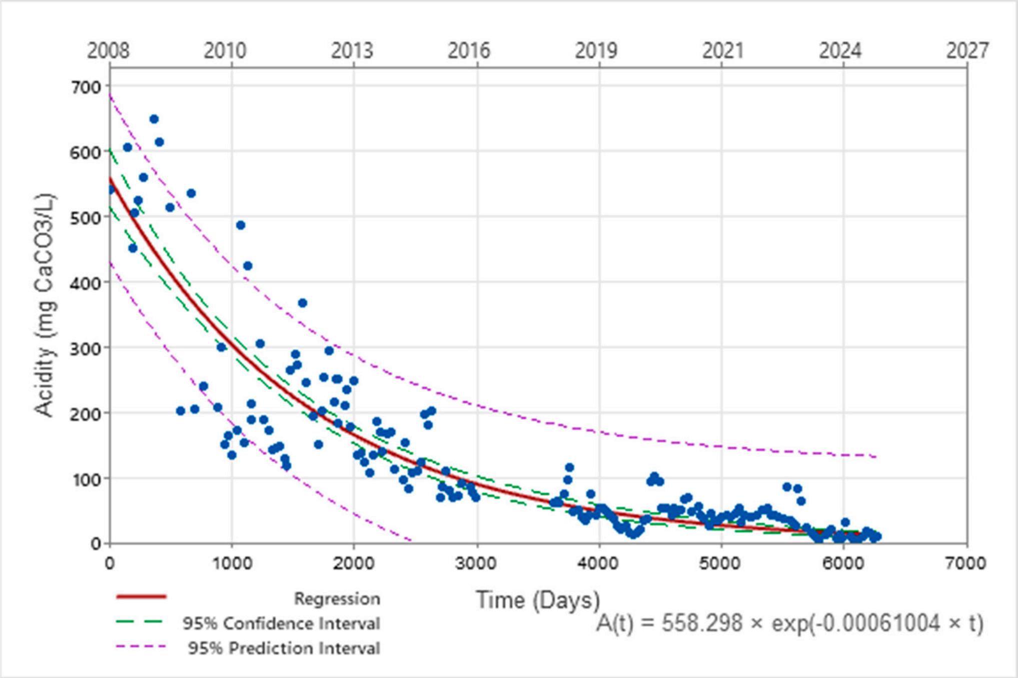

Figure 10. Downstream acidity of the drainage (measured values as blue dots) at the Capão da Roça coal waste site.

Table 3 shows the results of a mathematical regression based on a first-order kinetic decay model for iron, aluminium, manganese, sulphate, and acidity, in days and years. The value of C_0 in the equation represents the first value measured in 2008, when the coal waste site was reconfigured. However, the AMD generation process has been under way since 1988, suggesting that the concentration values may have been even higher. k is the rate of decay, which was greater at Capão da Roça than in almost all the other works [26–29], with values six to twenty-eight times larger for iron and four to nine times higher for sulphate. This may be because the other sites were all underground mines or disposal sites in temperate regions, whereas Capão da Roça is a coal waste dump site at the surface, and is more exposed to surface water.

Table 3. Results of a mathematical regression based on a first-order kinetic decay for iron, aluminium, manganese, sulphate, and acidity, in days and years.

Parameter	Exponential Regression	Standard Error
Iron	$C(t) = 178.06.e^{-0.0013\cdot t}$ t in days $C(t) = 178.06.e^{-0.48\cdot t}$ t in years	$C_0 = 7.66$ k = 0.000068 (t in days) k = 0.025 (t in years)
Aluminium	$C(t) = 29.06.e^{-0.00093\cdot t}$ t in days $C(t) = 29.06.e^{-0.34\cdot t}$ t in years	$C_0 = 1.19$ k = 0.000042 (t in days) k = 0.015 (t in years)
Manganese	$C(t) = 3.05.e^{-0.00027\cdot t}$ t in days $C(t) = 3.05.e^{-0.10\cdot t}$ t in years	$C_0 = 0.11$ k = 0.000015 (t in days) k = 0.0055 (t in years)

Table 3. *Cont.*

Parameter	Exponential Regression	Standard Error
Sulphates	$C(t) = 2023.75.e^{-0.00031 \cdot t}$ t in days $C(t) = 2023.75.e^{-0.11 \cdot t}$ t in years	$C_0 = 79.41$ k = 0.000017 (t in days) k = 0.0064 (t in years)
Acidity	$A(t) = 558.30.e^{-0.00061 \cdot t}$ t in days $A(t) = 558.30.e^{-0.22 \cdot t}$ t in years	$C_0 = 22.45$ k = 0.000031 (t in days) k = 0.011 (t in years)

Some authors have added a constant C_b, representing the background concentration value, to the regression to give $C(t) = C_0.e^{-k.t} + C_b$. In other words, this is the value to which the equation tends as time goes to infinity. In this case, it seems clear that the concentrations of iron and aluminium tend to zero, although the values for manganese and sulphates are still unclear.

3.3. Transition from Active to Passive Treatment

The effluent originating from the percolation of water through the dump waste has been treated by chemical precipitation and sedimentation since 2008. Based on the evolution of the ARD, we can divide the treatment into three distinct phases:

(i) From 2008 to 2020 (from day 1 to day 4699): needed pH adjustment and removal of metals Fe, Al, and Mn (Table 4);
(ii) From 2021 to 2023 (from day 4700 to day 5794): needed pH adjustment and removal of Mn (Table 5);
(iii) From 2024 to present (from day 5798 to day 6180): needed the occasional adjustment of pH and removal of Mn (Table 6).

It can be seen that active treatment has always proven effective, with few or no nonconformities, especially in recent years. However, over the last five years, the raw effluent has begun to show very low levels of iron and aluminium, requiring only pH adjustment and a reduction in the manganese levels; in fact, even the pH and manganese levels were often within the discharge standards. However, uncertainty over these two parameters means that operation of the active treatment plant is required. Under such conditions, however, when the concentrations of Fe^{3+} and Al are below 1 mg L^{-1}, a transition to passive treatment systems could be considered [5].

Table 4. Regression results of active treatment at Capão da Roça coal waste site from 2008 to 2020.

Parameter	Raw Wastewater, n = 129			Treated Wastewater, n = 129			Emissions Standards
	Day 1	Day 4699	NC	Day 1	Day 4699	NC	
pH	2.1	4.1	119	5.6	7.5	16	5.0–9.0
Fe (mg L^{-1})	177.8	0.4	58	2.1	0.1	1	<10.0
Al (mg L^{-1})	29.0	0.4	19	0.9	0.1	1	<10.0
Mn (mg L^{-1})	3.1	0.8	95	0.4	0.1	3	<1.0
Sulphates (mg L^{-1})	2023.1	482.2	-	972.2	500.0	-	-

n = sample size, NC—cases of non-compliance.

Table 5. Regression results of active treatment at Capão da Roça coal waste site from 2021 to 2023.

Parameter	Raw Wastewater, n = 36			Treated Wastewater, n = 36			Emissions Standards
	Day 4700	Day 5794	NC	Day 4700	Day 5794	NC	
pH	4.1	4.6	36	7.5	7.9	0	5.0–9.0
Fe (mg L^{-1})	0.4	0.1	0	0.1	0.1	0	<10.0
Al (mg L^{-1})	0.4	0.1	0	0.1	0.1	0	<10.0
Mn (mg L^{-1})	0.8	0.6	9	0.1	0.1	0	<1.0
Sulphates (mg L^{-1})	481.9	345.0	-	500.0	390.0	-	-

n = sample size, NC—cases of non-compliance.

Table 6. Regression results of active treatment at Capão da Roça coal waste site in 2024 and 2025.

Parameter	Raw Wastewater, n = 16			Treated Wastewater, n = 16			Emissions Standards
	Day 5795	Day 6525	NC	Day 5795	Day 6525	NC	
pH	4.6	4.7	2	7.9	8.0	0	5.0–9.0
Fe (mg L^{-1})	0.1	0.1	0	0.1	0.1	0	<10.0
Al (mg L^{-1})	0.1	0.1	0	0.1	0.1	0	<10.0
Mn (mg L^{-1})	0.6	0.6	6	0.1	0.1	0	<1.0
Sulphates (mg L^{-1})	345.0	306.7	-	389.9	351.2	-	-

n = sample size, NC—cases of non-compliance.

In 2020, the first effort was made by Silva et al. [41] to treat effluent from Capão da Roça in a pilot scale closed-loop open-channel system as function of time (up to 60 min), using calcitic and dolomitic limestones, associated or not with natural or functionalised zeolites. This approach successfully increased the pH (increased alkalinity) and removed the residual amounts of iron, aluminium, and manganese present in the effluent in concentrations of 8.2 mg L^{-1}, 2.5 mg L^{-1}, and 0.8 mg L^{-1}, respectively. Some degree of armouring of the limestone particles was observed.

Two years later, with the effluent showing lower concentrations of metals, especially iron, a second experiment was carried out in which the bed of the open channel was composed of calcitic limestone or steel slag, as described above. Table 7 summarises the results obtained for the pH, Fe, Al, and Mn concentrations, as well as toxicity results for *Daphnia magna*. The complete analysis of the raw effluent, the effluent treated after flowing through the channel filled with limestone and the channel filled with laden furnace slag from a semi-integrated steel plant can be found in Table S1 of the Supplementary Material. It can be observed that there was an increase in pH and a decrease in iron and aluminium levels. Although the manganese levels in this specific raw wastewater sample were low, it can be observed that in the slag channel, some decrease in the concentration of this metal was caused by raising the pH further than the limestone channel. Finally, the raw effluent, which still had some toxicity to the test organism, was classified in ecotoxicological terms as nontoxic after passing through the beds. In addition, considering a flow rate of 40 m^3 day^{-1} (20 h per day), the acidity of the AMD in 2023 of 20.6 mg $CaCO_3$ L^{-1} (calculated from the equation in Table 3), and taking into account the neutralisation potential of limestone and slag (Table 1), a consumption of 21.6 kg $month^{-1}$ of limestone and 28.1 kg $month^{-1}$ of slag is estimated if the system works without operational problems (e.g., armouring of limestone or slag pebbles).

Table 7. Results of passive AMD treatment with open channels of limestone and steel slag for an AMD sample collected in 2022 and the emission standards defined by the local environmental agency for the release of liquid effluents in the Capão da Roça area.

Parameter	Raw AMD	After Limestone Channel	After Slag Channel	Emissions Standards
pH	3.4	7.5	8.2	5.0–9.0
Fe (mg L^{-1})	0.7	0.2	0.1	15
Al (mg L^{-1})	2.0	0.3	0.5	10
Mn (mg L^{-1})	0.7	0.7	0.6	1.0
CE_{50}-48 h (%)	66.81	>100	>100	
Interpretation	Toxic	Nontoxic	Nontoxic	

Thus, there is a strong indication that a passive treatment system based on limestone or slag channels, whether associated with zeolites or not, could guarantee the quality of the effluent. This treatment configuration would require less operational control and lower energy consumption, resulting in reduced greenhouse gas emissions among other benefits [23]. As a final observation, it is worth highlighting that in the case of slag, it must be ensured that the material comes from a safe source and is free from any harmful components [63].

4. Conclusions

Previous analyses of coal waste at Capão da Roça demonstrated the potential for AMD generation, and despite being covered with alkaline coal ash and topsoil, the water acidification process continued. Based on a historical survey of the site, it was possible to demonstrate that the effluent was acidic from 2008 to 2023; however, in 2024 and 2025, the acidity was lowered to such levels that the pH was raised, and the presence of metals was minimal. The main reason for this was the attenuation of the pyrite oxidation process, which provided an elevation of the pH to values sufficient for the precipitation of iron and close to that for the precipitation of aluminium; although the extreme rainfall events that occurred in 2023–2024 may also have had an influence.

During the 17-year monitoring period, the iron, aluminium, manganese, and sulphate content and the acidity were well modelled by first-order decay phenomena, thus validating the work of prior authors. However, the rate constant observed in this work was higher for the dissolution of iron and sulphates, both products of pyrite oxidation, for which there could be two reasons: (a) this location is a surface coal waste deposit, which is more susceptible to oxidant conditions, and (b) it is subject to warmer climate conditions than the sites of most of the previously published data. Although these parameters have been highlighted, factors such as waste deposit size, porosity, water retention time, pyrite content, mineral composition, and the absence of toxic elements, among other factors, were also relevant to stablish the AMD generation rate. The results indicated an increase in pH (from 2.1 to 4.7) and a decay in the concentration of metals (from 177.8 to 0.1 mg L^{-1} for iron, 29.0 to 0.1 mg L^{-1} for aluminium, and 3.1 to 0.6 mg L^{-1} for manganese), sulphates (from 2023 to 307 mg L^{-1}), and acidity (from 539.5 mg $CaCO_3$ L^{-1} to 3.96 mg $CaCO_3$ L^{-1}).

The active treatment of raw AMD with pH correction, precipitation, and metal hydroxide sedimentation is still in operation due to the uncertainty in pH and final manganese concentration, although the concentration levels of iron and aluminium reliably meet the emission standards. Tests involving the transition of the active system to open channels with limestone or slag pebbles were successful, and this may offer a safe alternative process with less need for operational control, lower energy demand and a more integrative approach for the ecological and functional restoration of the area.

Supplementary Materials: The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/min15101068/s1, Figure S1: Mineralogical analysis by XRD of the limestone sample; Figure S2. Mineralogical analysis by XRD of the slag sample; Table S1. Characteristics of AMD in terms of pH, Eh, acidity, sulfates, and elemental analysis in 2022.

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

Funding: This work was supported by the National Council for Scientific and Technological Development (CNPq)—314880/2020-8, the Coordination for the Improvement of Higher Education Personnel (CAPES)—88887.007050/2024-00, and COPELMI Mineração.

Data Availability Statement: The data presented in this study are available within the article and its supplementary materials.

Acknowledgments: The authors would like to thank UFRGS, CNPq, CAPES, and COPELMI Mineração for their financial support in the development of this research.

Conflicts of Interest: Daniela Silva, Alexandre Grigorieff, and Cristiano Corrêa Weber are employees of Copelmi Mineração Ltda. The content of this paper reflects the views of the scientists and not those of the company.

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