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SOILS, SEC 3 • REMEDIATION AND MANAGEMENT OF CONTAMINATED OR DEGRADED LANDS • RESEARCH ARTICLE

Remediation of metal-contaminated mine tailings by the application of organic and mineral amendments

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Abstract

Purpose Tailings are generally characterized by severe physicochemical conditions that limit the establishment of vegetation. The present study aimed to select suitable combinations of organo-mineral amendments to improve the physicochemical, biochemical, and biological properties of spolic technosols, highly contaminated with metals.

Materials and methods Several substrates were prepared by mixing mine tailings (MT) of an abandoned mining area with non-contaminated agricultural soil (anthrosol), green waste compost, lime, and rock phosphate at different rates: S1 — 50% of MT + 50% of agricultural soil; S2 — S1 + 3% of lime ($CaCO_3$); S3 — S1 + 6% of rock phosphate; S4 — S1 + 10% of compost; S5 — S1 + 10% of compost + 3% of lime; S6 — S1 + 10% of compost + 6% of rock phosphate. Untreated MT and agricultural soil were analyzed immediately, and 8 months after incorporating the amendments.

Results and discussion Heterotrophic microorganisms were not recovered from untreated MT due to the highly acidic pH and available metal concentrations. However, the addition of organo-mineral amendments ameliorated the tailings' characteristics by increasing pH, conductivity, total organic carbon, and available P levels. Moreover, after 8 months, heterotrophic microorganisms were recovered from those substrates and dehydrogenase activity was enhanced. The incorporation of agricultural soil and green waste compost mixed either with lime (S5) or rock phosphate (S6) was the most effective treatment.

Conclusions Both S5 and S6 mixtures successfully reduced the environmental risk posed by tailings, suggesting the potential use of these amendments for the remediation of pyrite mines.

Keywords Kettara mine · Metals · Amendments · Acid mine drainage · Microbial populations · *In situ* remediation

1 Introduction

Located northwest of Marrakech, Kettara mine was extensively exploited from 1938 to 1982. It constitutes the main abandoned deposit of pyrrhotite and copper within the Jebilets massive (Hibti et al. 1999). After mine closure, tailings were deposited open air in the adjacent areas, exposed to weather events, including wind erosion. Moreover, the oxidation of metal sulfide in tailings ponds has contributed to the contamination of the surrounding environment, mainly through leaching (Boularbah et al. 2006a, b; El Khalil et al. 2008; El Hamiani et al. 2010). Indeed, according to previous studies, this abandoned mine constitutes a problematic source of toxic elements, namely Cu and Zn, which can be transferred to the vegetation and surface and ground water (Boularbah et al. 2006a, b; El Khalil et al. 2008; El Hamiani et al. 2010). This poses a real health risk to populations, especially for children, residing in the vicinity of this mine

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due to the biomagnification of metals through the consumption of contaminated vegetables and/or direct ingestion and inhalation of dust and soil particles (Zheng et al. 2007; Zhuang et al. 2009; El Hamiani et al. 2015).

In addition to the high concentration of metals, sulfide mine wastes are often characterized by low organic matter and nutrient content, highly acidic pH, and poor physical structure (Wong 2003; Mendez et al. 2008), which may hinder plant growth (Boularbah et al. 2006a; Sheoran et al. 2010; Benidire et al. 2021) and negatively impact biological processes in soils (Benidire et al. 2020, 2021; Tembo et al. 2006; Navarro et al. 2008). Therefore, lack of vegetation cover is frequent in mining sites, which explains why these areas are highly prone to erosion, leading to an increase in the dispersion of pollutants and a decrease of the natural resilience of contaminated ecosystems (Nicolau and Asensio 2000; Singh et al. 2016).

The development of sustainable strategies for pollution control in abandoned mines is of utmost importance to reduce harmful impacts on the neighboring environment and on human health. The most commonly used strategies for the remediation of anthropogenically contaminated areas are those related to physical and chemical techniques, since they require less time for treatment. Nevertheless, these methods are expensive and generally environmentally disruptive, damaging soil structure and biodiversity (Pérez-de-Mora et al. 2006; Burges et al. 2018; Lacalle et al. 2020). In contrast, alternative *in situ* remediation techniques, such as the application of organic and/or mineral amendments, are gaining momentum (Lwin et al. 2018; Nejad et al. 2018; Liu et al. 2018). Amelioration of soil conditions by the addition of amendments results from a decrease of the mobility and bioavailability of metallic pollutants (Gao et al. 2020; Lwin et al. 2018; Palansooriya et al. 2020; Pardo et al. 2014), which occurs through the increase of pH and several adsorption, precipitation, and complexation reactions (Lwin et al. 2018; Peng et al. 2009; Palansooriya et al. 2020). In addition, amendments may facilitate the settlement of a stable vegetative cover on such degraded areas by enhancing the chemical and biological processes that occur in the soils (Lwin et al. 2018; Pérez-de-Mora et al. 2006; Madejón et al. 2006; Benidire et al. 2021). The most frequently used amendments to ameliorate physicochemical properties of mining soils can be either organic materials such as compost, manure, biosolids, sewage sludge, and biochar (Ahmad et al. 2017; Beesley et al. 2014) and/or inorganic substrates such as lime and mineral rock phosphate (Holland et al. 2018; Bade et al. 2012). For instance, Al-Lami et al. (2019) showed that the addition of biosolids contributed to neutralizing the pH and increasing the levels of organic carbon and nutrients, as well as the cation exchange and water retention capacity of Pb/Zn/Cu-contaminated mine tailings. Likewise, Gao et al. (2020) reported that the incorporation of a mixture of biochar, non-contaminated soil, peat, and manure significantly increased the pH and fertility of Pb/Zn ore tailings, and reduced the bioavailability of Zn, Pb, Cd, and As.

Nevertheless, the success of this strategy is often dependent on the composition of amendments (raw materials), on type and severity of the contamination, and ultimately on soil properties (Lwin et al. 2018; Palansooriya et al. 2020), and it is extremely important to define the best combination of amendments for each particular case. The aim of this work was to evaluate the suitability of organo-mineral amendments, applied singly or in combination, to improve the physicochemical, biochemical, and biological properties of tailings from the Kettara pyrrhotite mine to foster *in situ* soil remediation and, ultimately, the revegetation of such highly contaminated areas.

2 Material and methods

2.1 Mine tailings, amendments, and substrates

Mine tailings (MT) were collected from an abandoned sulfide mine located in Kettara village (31°52′25.2″N 8°10′42.0″W Fig. 1), near Marrakech (Morocco). According to the World Reference Base (WBR) for Soil Resources, these tailings can be classified as spolic technosols (IUSS WG WRB 2015). The extraction of pyrrhotite from Kettara mine resulted in huge amounts of residues, which were deposited over an area of 16 ha (Boularbah et al. 2006a; El Hamiani et al. 2010). This mine was abandoned many years, no barriers implemented to prevent the dispersion of contaminants to the surrounding areas, and no remediation strategies have been applied.

Due to the high acidity, low fertility, and poor physical structure (absence of aggregates and low organic matter) of mine tailings (Boularbah et al. 2006a), they were firstly mixed with an agricultural soil (1:1 w/w) in order to obtain a substrate which could support plant growth and development. The agricultural soil used in this experiment is an anthrosol (IUSS WG WRB 2015), sampled from three randomly selected points in a non-contaminated area (metal concentration within European standards) located at Souihla, near Marrakech (El Khalil et al. 2013) (Fig. 1). The physicochemical properties were as follows: pH 7.5 ± 0.080; electrical conductivity (mS cm^{-1}) 0.46 ± 0.056; total organic carbon (%) 1.3 ± 0.19; available P (mg g^{-1}) 0.26 ± 0.036. The agricultural soil and tailings used in this study were air dried for 2 weeks and sieved to 2 mm. Then, substrate S1 (MT + agricultural soil) was mixed in different proportions with organic (green waste compost) and/or mineral (lime-$CaCO_3$, rock phosphate) amendments in order to improve structure, decrease metals' availability,

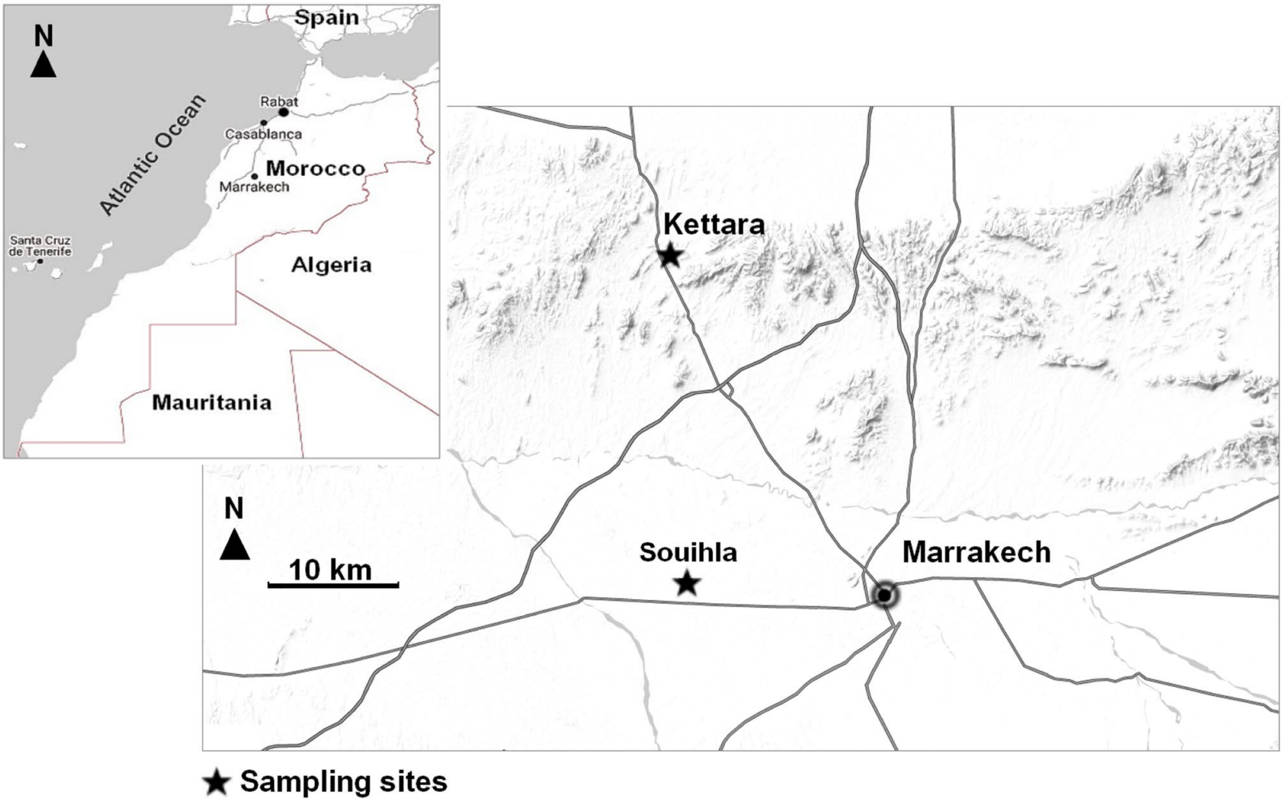

Fig. 1 Location of sampling sites of mine tailings and agricultural soil

and increase soil organic matter and nutrient content (Benidire et al. 2021; Holland et al. 2018; Xiao et al. 2017). As shown in Table 1, five different treatments were tested: S1 — 50% of MT + 50% of agricultural soil; S2 — S1 + 3% of lime; S3 — S1 + 6% of rock phosphate; S4 — S1 + 10% of compost; S5 — S1 + 10% of compost + 3% of lime; S6 — S1 + 10% of compost + 6% of rock phosphate. The compost was provided by the Laboratory of Microbial Biotechnologies, Agrosciences and Environment of the Science Faculty of University Cadi-Ayyad, while rock phosphate was obtained from wastes of a phosphate mine. Physicochemical properties of both amendments are described in Appendix 1 – supplementary material.

Table 1 Composition of subtracts obtained by the incorporation of organo-mineral amendments to mine tailings

Substrates	Composition
Control (C)	100% of agricultural soil
MT	100% of mine tailings
S1	50% of mine tailings + 50% of agricultural soil
S2	S1 + 3% of lime ($CaCO_3$)
S3	S1 + 6% of rock phosphate
S4	S1 + 10% of compost
S5	S1 + 10% of compost + 3% of lime ($CaCO_3$)
S6	S1 + 10% of compost + 6% of rock phosphate

2.2 Experimental design

Experiments were conducted in pots containing 300 g of substrates for 8 months under controlled temperature (30 °C) in a randomized design, with three replicates per treatment. All substrates were watered with deionized water and maintained at 50% of water holding capacity. Composite substrate samples were collected at the beginning, immediately after the incorporation of amendments (T0), and at the end of the experiment, 8 months latter (T8), and divided into two fractions; the first one was stored at 4 °C for biological analysis, while the other was air-dried for physicochemical analysis.

2.3 Substrate analysis

2.3.1 Physicochemical analysis

The pH and electrical conductivity (EC) were determined in substrate/water suspensions (1:2.5 and 1:5 (w/v) for pH and EC, respectively) by the glass electrode method (NF ISO 10390). Total nitrogen (TN) was determined by the Kjeldahl method (NF ISO 11261, AFNOR 1995), while total organic carbon (TOC) was analyzed by dichromate oxidation and titration with ferrous ammonium sulfate (Blakemore et al. 1972). Available P was measured on bicarbonate extracts by the molybdenum blue method as described by Olsen and Sommers (1982). Pseudo-total and extractable metal (Cu, Zn, Pb) concentrations were determined by atomic absorption spectrometry (AAS) (Model Perkin Elmer 400, USA) following digestion of samples with aqua regia, and extraction with 0.01 M $CaCl_2$ (1:25 (w/v)), respectively, according to standard methods (NF EN ISO 11 466). The Pollution Index (PI) was calculated for each metal *i* as the ratio between the metal concentration (C_i) in a soil sample and its reference value (S_i) according to Wu et al. (2015):

$$PI_i = \frac{C_i}{S_i}$$

The S_i values for Cu, Zn, and Pb were based on the Canadian Soil Quality Guidelines for the Protection of Environmental and Human Health for residential/parkland land use (mg kg^{-1}): Cu = 63; Zn = 200; Pb = 140. Contamination classes: $PI \leq 1$ = no contamination; $1 < PI \leq 3$ = slight contamination; $3 < PI \leq 5$ = moderate contamination; $PI > 5$ = severe contamination.

2.3.2 Germination toxicity test

The phytotoxicity of substrates was estimated using a germination inhibition test according to the AFNOR X 31–201 protocol. Seeds of three plant species, namely, alfalfa (*Medicago sativa* L.), barley (*Hordeum vulgare* L.), and soft wheat (*Triticum aestivum* L.), were used. Germination tests were prepared by placing 50 seeds in Petri dishes (10 cm) containing 40 g of each substrate. A control was prepared by placing 50 seeds in Petri dishes containing Whatman paper moistened with deionized water instead of substrate. Petri dishes were incubated at 28 °C for 7 days in darkness and 50% relative humidity. All trials were conducted in triplicate. Germination inhibition (GI) was calculated according to the following formula:

$$GI = \left[\frac{(A - B)}{A}\right] * 100\ \%$$

where A is the mean seed germination in the control and B is the mean seed germination in the substrates.

2.3.3 Enzymatic activities

Two soil enzymes were analyzed as biochemical parameters to evaluate the efficiency of the remediation process. Dehydrogenase (DHA) was used as an index of overall microbial activity, while acid phosphatase (AP) was analyzed due to its strong relationship with the P cycle. DHA activity was determined according to Alef (1995) by using 2,3,5-triphenyltetrazoliumchloride (TTC) as the electron acceptor. The reduction of TTC to triphenyl formazan (TPF) was measured spectrophotometrically at 490 nm, and the enzyme activity expressed as µg TPF g dry $soil^{-1}$ 24 h^{-1}. AP activity was measured according to a modified method of Tabatabai and Bremner (1969), after incubation of substrate samples with *p*-nitrophenyl phosphate disodium (27 g l^{-1}) in a 0.5 M modified universal buffer (pH 4.5) at 37 °C for 60 min. The formation *p*-nitrophenol (PNP) was determined spectrophotometrically at 405 nm and the enzyme activity expressed as µg PNP g dry $soil^{-1}$ h^{-1}.

2.3.4 Enumeration of culturable heterotrophic microorganisms

To evaluate the effects of organo-mineral amendments on the abundance of microorganisms in the different substrates, the total counts of heterotrophic bacteria (HB), fungi, and actinomycetes were determined by spread-plating serial dilutions on media: Tryptic Soy Agar (TSA, Fluka) plus cycloheximide (50 mg l^{-1}), Potato Dextrose Agar (PDA, Difco) plus chlortetracycline (30 mg l^{-1}), and International Streptomyces Project 2 (ISP2) supplemented with nalidixic acid (100 mg l^{-1}) and cycloheximide (50 mg l^{-1}), respectively. Three replicates were made for each sample and media. Plates were incubated at 30 °C and bacterial and fungi colonies were counted after 3 and 7 days, respectively. For actinomycetes enumeration, plates were incubated at 25 °C for 7–14 days. Data were expressed as colony-forming units (CFU) per g of dry weight of substrate.

2.4 Statistical analysis

All statistical analyses were carried out with the statistical program SPSS (IBM, Armonk, NY, USA, version 26.0.) Results were analyzed through analysis of variance (one-way and two-way ANOVA), and the statistical significance of differences ($P < 0.05$) between means was determined by Tukey's post hoc test. A correlation matrix between physicochemical, biochemical, and biological parameters was calculated. The significance level reported ($P < 0.01$ and $P < 0.05$) is based on Pearson's correlation coefficients.

3 Results

3.1 Substrate physicochemical analyses

Physicochemical properties of mine tailings changed significantly ($P < 0.001$) after the addition of different amendments (Table 2). Mine tailings showed a very low pH (2.7) which was significantly ($P < 0.001$) increased by the addition of the

Table 2 Physicochemical properties of different substrates immediately (T0) and 8 months (T8) after the incorporation of amendments

Time	Substrate	pH	EC (mS cm^{-1})	TOC (%)	P Olsen (mg g^{-1})
T0	**C**	7.5 ± 0.08^{a}	0.46 ± 0.056^{b}	1.28 ± 0.193^{b}	0.26 ± 0.036^{ab}
	MT	2.7 ± 0.04^{f}	2.14 ± 0.416^{a}	0.26 ± 0.021^{d}	0.06 ± 0.012^{d}
	S1	4.7 ± 0.03^{e}	0.59 ± 0.042^{b}	0.66 ± 0.015^{c}	0.16 ± 0.080^{bc}
	S2	5.8 ± 0.35^{c}	0.54 ± 0.008^{b}	0.64 ± 0.020^{c}	0.14 ± 0.017^{bc}
	S3	5.6 ± 0.08^{c}	0.43 ± 0.004^{b}	0.60 ± 0.021^{c}	0.25 ± 0.004^{ab}
	S4	5.2 ± 0.06^{d}	0.53 ± 0.010^{b}	2.62 ± 0.031^{a}	0.25 ± 0.001^{ab}
	S5	6.4 ± 0.12^{b}	0.31 ± 0.006^{b}	2.53 ± 0.042^{a}	0.30 ± 0.015^{a}
	S6	6.4 ± 0.16^{b}	0.39 ± 0.005^{b}	2.56 ± 0.050^{a}	0.36 ± 0.069^{a}
		***F = 274.663	***F = 48.763	***F = 560.343	***F = 17.036
T8	**C**	7.4 ± 0.14^{a}	$0.45\pm0.061^{b,c}$	1.66 ± 0.046^{c}	0.40 ± 0.074^{b}
	MT	2.6 ± 0.02^{c}	2.48 ± 0.228^{a}	0.25 ± 0.031^{f}	0.05 ± 0.006^{d}
	S1	5.2 ± 0.10^{d}	0.60 ± 0.033^{b}	0.95 ± 0.050^{e}	0.22 ± 0.083^{c}
	S2	$6.5\pm0.45^{b,c}$	$0.50\pm0.023^{b,c}$	0.83 ± 0.061^{e}	0.20 ± 0.028^{c}
	S3	6.4 ± 0.55^{c}	$0.43\pm0.004^{b,c}$	$1.14\pm0.091^{d,e}$	$0.33\pm0.013^{b,c}$
	S4	5.4 ± 0.07^{d}	$0.52\pm0.011^{b,c}$	2.81 ± 0.035^{b}	0.36 ± 0.001^{b}
	S5	7.1 ± 0.17^{ab}	0.30 ± 0.002^{c}	2.93 ± 0.076^{b}	0.32 ± 0.011^{bc}
		7.3 ± 0.03^{a}	0.35 ± 0.014^{c}	3.17 ± 0.142^{a}	0.56 ± 0.027^{a}
		***F = 106.171	***F = 217.716	***F = 661.989	***F = 39.951
		***FT = 49.921	NSFT = 0.754	***FT = 227.550	***FT = 48.164
		***FA = 286.941	***FA = 178.611	***FA = 1212.325	***FA = 53.680
		***FTxA = 5.365	NSFTxA = 1.551	***FTXA = 11.000	**FTxA = 4.178

Results are expressed as mean ± SD ($n = 3$). A two-way ANOVA was performed to determine the influence of time (T0, immediately after the incorporation of amendments; T8, 8 months after the incorporation of amendments) and amendments (control (C), 100% of agricultural soil; MT, 100% of mine tailings; S1, 50% of mine tailings + 50% of agricultural soil; S2, S1 + 3% of lime ($CaCO_3$); S3, S1 + 6% of rock phosphate; S4, S1 + 10% of compost; S5, S1 + 10% of compost + 3% of lime ($CaCO_3$); S6, S1 + 10% of compost + 6% of rock phosphate) on physicochemical properties of substrates (pH; EC, electrical conductivity; TOC, total organic carbon; P Olsen, available phosphorous). Results are shown with the test statistic for each case (T, time; A, amendments; TxA, time x amendments) and as: NS, non-significant at the level $P > 0.05$; *significant at the level $P < 0.05$; ** significant at the level $P < 0.01$; *** significant at the level $P < 0.001$. A one-way ANOVA was performed to determine the influence of amendments on physicochemical properties of substrates for each time. Means for different treatments in each column with different letters are significantly different from each other according to Tukey test. The test results are shown with the test statistic and as: NS, non-significant at the level $P > 0.05$; *significant at the level $P < 0.05$; ** significant at the level $P < 0.01$; *** significant at the level $P < 0.001$. For pH, the F values of one-way ANOVA are $F = 274.663$ ($P < 0.001$) and $F = 106.171$ ($P < 0.001$) for T0 and T8, respectively. For EC, the F values of one-way ANOVA are $F = 48.763$ ($P < 0.001$) and $F = 217.716$ ($P < 0.001$) for T0 and T8, respectively. For TOC, the F values of one-way ANOVA are $F = 560.343$ ($P < 0.001$) and $F = 661.989$ ($P < 0.001$) for T0 and T8, respectively. For P Olsen, the F values of one-way ANOVA are $F = 17.036$ ($P < 0.001$) and $F = 39.951$ ($P < 0.001$) for T0 and T8, respectively

agricultural soil (S1), the rise being even more pronounced with the application of organo-mineral amendments, reaching pH values up to 6.4 and 7.3 at T0 and T8, respectively. Mine tailings amended with a combination of agricultural soil, compost and lime (S5) or agricultural soil, compost and rock phosphate (S6) showed the highest pH increases. Indeed, after 8 months, both substrates showed pH values similar to those found in the control (non-contaminated agricultural soil). The incorporation of organo-mineral amendments significantly ($P < 0.001$) decreased EC of mine tailings. However, no significant ($P > 0.05$) differences were observed between sampling times (T0 and T8). For all substrates, mean values of TOC at the second sampling (T8) were significantly ($P < 0.001$) higher than at the beginning (T0), except for untreated mine tailings. However, the highest increases (up to 105%) were observed at T0 in all substrates supplemented with compost (S4, S5, S6). Available P levels determined in all amended substrates were significantly ($P < 0.001$) higher than in mine tailings. The incorporation of both compost and rock phosphate (S6) proved to be most effective treatment to increase the concentrations of available P, especially at the end of the experiment (T8).

In both sampling periods, pseudo-total Cu, Zn, and Pb concentrations were very high in untreated mine tailings, but tended to decrease by an average of about 70%, 40%, and 50%, respectively, in all amended substrates (Table 3). This

Table 3 Pseudo-total metal concentrations in substrates immediately (T0) and 8 months (T8) after the incorporation of amendments and pollution index (PI). Control (C), 100% of agricultural soil; MT, 100% of mine tailings; S1, 50% of mine tailings + 50% of agricultural soil; S2, S1 + 3% of lime ($CaCO_3$); S3, S1 + 6% of rock phosphate; S4, S1 + 10% of compost; S5, S1 + 10% of compost + 3% of lime ($CaCO_3$); S6, S1 + 10% of compost + 6% of rock phosphate. *dl*, detection limit

Time	Substrate	Pseudo-total metal concentrations (mg kg − 1 dry soil)			Pollution Index (PI)*		
		Cu	Zn	Pb	Cu	Zn	Pb
T0	**C**	5.79	58.87	< dl	0.1	0.3	0.0
	MT	1666.95	288.75	102.63	26.5	1.4	0.7
	S1	505.73	152.10	53.62	8.0	0.8	0.4
	S2	432.90	149.90	49.19	6.9	0.7	0.4
	S3	460.81	165.90	56.13	7.3	0.8	0.4
	S4	469.54	173.20	47.59	7.5	0.9	0.3
	S5	389.28	173.75	45.41	6.2	0.9	0.3
	S6	415.24	165.40	48.48	6.6	0.8	0.3
T8	**C**	6.15	58.79	< dl	0.1	0.3	0.0
	MT	1648.13	291.88	99.82	26.2	1.5	0.7
	S1	521.35	156.45	55.64	8.3	0.8	0.4
	S2	499.20	147.80	48.23	7.9	0.7	0.3
	S3	494.20	176.30	57.36	7.8	0.9	0.4
	S4	381.08	165.80	48.44	6.0	0.8	0.3
	S5	399.00	175.85	44.81	6.3	0.9	0.3
	S6	366.91	159.95	48.98	5.8	0.8	0.3

*Calculated based on Canadian Soil Quality Guidelines for the Protection of Environmental and Human Health for residential/parkland land use

tendency was accompanied by a decrease of PI in amended substrates. However, despite the overall abatement observed, PI for Cu remained very high in all treated substrates, them being still classified as severely contaminated.

Overall, the concentrations of Cu- and Zn-extractable forms in treated substrates tended to be higher than in the control (non-contaminated agricultural soil), but lower than those found in untreated mine tailings (Table 4). Cu- and Zn-extractable concentrations in mine tailings showed a rising trend over time; however, an opposite tendency was observed in amended substrates, with the lowest values being recorded in substrate S6, followed by S5 and S2.

3.2 Germination toxicity test

Germination inhibition (GI) of seeds of three plant species in different substrates, 8 months after the incorporation of organo-mineral amendments, is shown in Fig. 2. A complete inhibition of seed germination of all tested species was observed in untreated mine tailings, while in agricultural soil (control) GI was lower than 2.5%. The addition of agricultural soil to mine tailings (S1) significantly ($P < 0.001$) reduced GI (on average by 50%) in all tested species. This decrease was further pronounced when mine tailings were supplemented with the different combinations of organo-mineral amendments. The germination of soft wheat and barley seeds in substrates S2, S3, S4, S5, and S6 was poorly affected (≈GI < 3%). The highest sensitivity was observed for alfalfa, since germination remained quite

Table 4 $CaCl_2$-extractable metal concentrations in substrates immediately (T0) and 8 months (T8) after the incorporation of amendments. Control (C), 100% of agricultural soil; MT, 100% of mine tailings; S1, 50% of mine tailings + 50% of agricultural soil; S2, S1 + 3% of lime ($CaCO_3$); S3, S1 + 6% of rock phosphate; S4, S1 + 10% of compost; S5, S1 + 10% of compost + 3% of lime ($CaCO_3$); S6, S1 + 10% of compost + 6% of rock phosphate. *dl*, detection limit

Time	Substrate	$CaCl_2$-extractable metal concentrations (mg kg^{-1} dry soil)		
		Cu	Zn	Pb
T0	**C**	0.12	< dl	< dl
	MT	24.01	3.27	< dl
	S1	0.39	0.18	< dl
	S2	0.18	0.37	< dl
	S3	0.21	1.20	< dl
	S4	0.30	0.58	< dl
	S5	0.10	< dl	< dl
	S6	0.09	0.01	< dl
T8	**C**	0.13	< dl	< dl
	MT	43.01	4.62	< dl
	S1	0.44	0.06	< dl
	S2	0.17	< dl	< dl
	S3	0.30	0.01	< dl
	S4	0.33	0.01	< dl
	S5	0.09	< dl	< dl
	S6	0.05	< dl	< dl

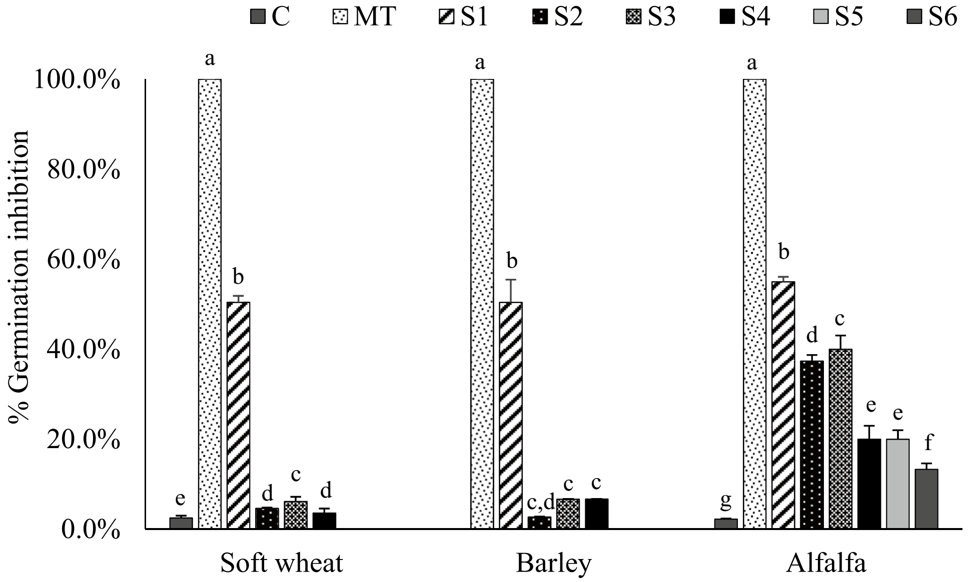

Fig. 2 Germination inhibitory rates (%) of soft wheat, barley, and alfalfa seeds grown in different substrates 8 months after the incorporation of amendments. The error bar represents the SD ($n = 3$). A one-way ANOVA was performed to determine the influence of amendments (control (C), 100% of agricultural soil; MT, 100% of mine tailings; S1, 50% of mine tailings + 50% of agricultural soil; S2, S1 + 3% of lime ($CaCO_3$); S3, S1 + 6% of rock phosphate; S4, S1 + 10% of compost; S5, S1 + 10% of compost + 3% of lime ($CaCO_3$); S6, S1 + 10% of compost + 6% of rock phosphate) on germination rates of each plant species. Means for the different treatments with different letters are significantly different from each other ($P < 0.05$) according to Tukey's test. The F values of one-way ANOVA are $F = 9504.555$ ($P < 0.001$), $F = 1292.567$ ($P < 0.001$), and $F = 1270.560$ ($P < 0.001$) for soft wheat, barley, and alfalfa, respectively

affected even after the addition of organo-mineral amendments to mine tailings.

3.3 Biochemical analyses

The activity of DHA and AP in substrates immediately (T0) and 8 months (T8) after the addition of amendments is shown in Fig. 3a, b respectively. Results show that DHA activity was not detected in untreated mine tailings, whereas in agricultural soil (control) it reached values of about 65 µg TPF g dry soil^{-1} 24 h^{-1}. The addition of agricultural soil and organo-mineral amendments led to an increase of DHA activity in mine tailings, especially 8 months (T8) after the incorporation. Indeed, mine tailings amended with agricultural soil plus compost and rock phosphate (S6) showed a DHA activity similar to that observed in control soil (≈103 µg TPF g dry soil^{-1} 24 h^{-1}), followed by substrates S5 and S3 (64 and 33 µg TPF g dry soil^{-1} 24 h^{-1}, respectively).

Contrary to that observed for DHA, the activity of AP, in the first sampling time (T0), was significantly higher in non-amended mine tailings than in the agricultural soil. The effect of amendments addition on AP activity seems to be dependent upon the nature of the mixture used. In lime-containing substrates (S2 and S5), the activity of this enzyme was quite low if compared to the other substrates. Moreover, the activity of AP in non-treated and treated mine tailings decreased with the time. This decrease was particularly evident in substrates S2 and S6.

3.4 Enumeration of culturable microorganisms

The effect of organo-mineral amendments on microbial counts is presented in Fig. 4. No heterotrophic bacterial counts were recovered from untreated mine tailings (Fig. 4a). However, immediately (T0) after the incorporation of agricultural soil and of organo-mineral amendments, the numbers of heterotrophic bacteria increased significatively (up to 0.2×10^6 CFU g^{-1} dry weight substrate). After 8 months, this increase was further pronounced both in control and in amended mine tailings, with the highest value (58×10^6 CFU g^{-1} dry weight substrate) being recorded in agricultural soil, compost and rock phosphate-containing substrate (S6).

As for the bacteria, fungi populations were not detected in untreated mine tailings. However, a significant increase in the number of fungi was observed in treated substrates for both sampling periods (Fig. 4b). The treatments that performed best were those containing agricultural soil combined with compost and lime (S5) and compost and rock phosphate (S6), with values of 0.49×10^5 and 0.64×10^5 CFU g^{-1} dry weight substrate, respectively.

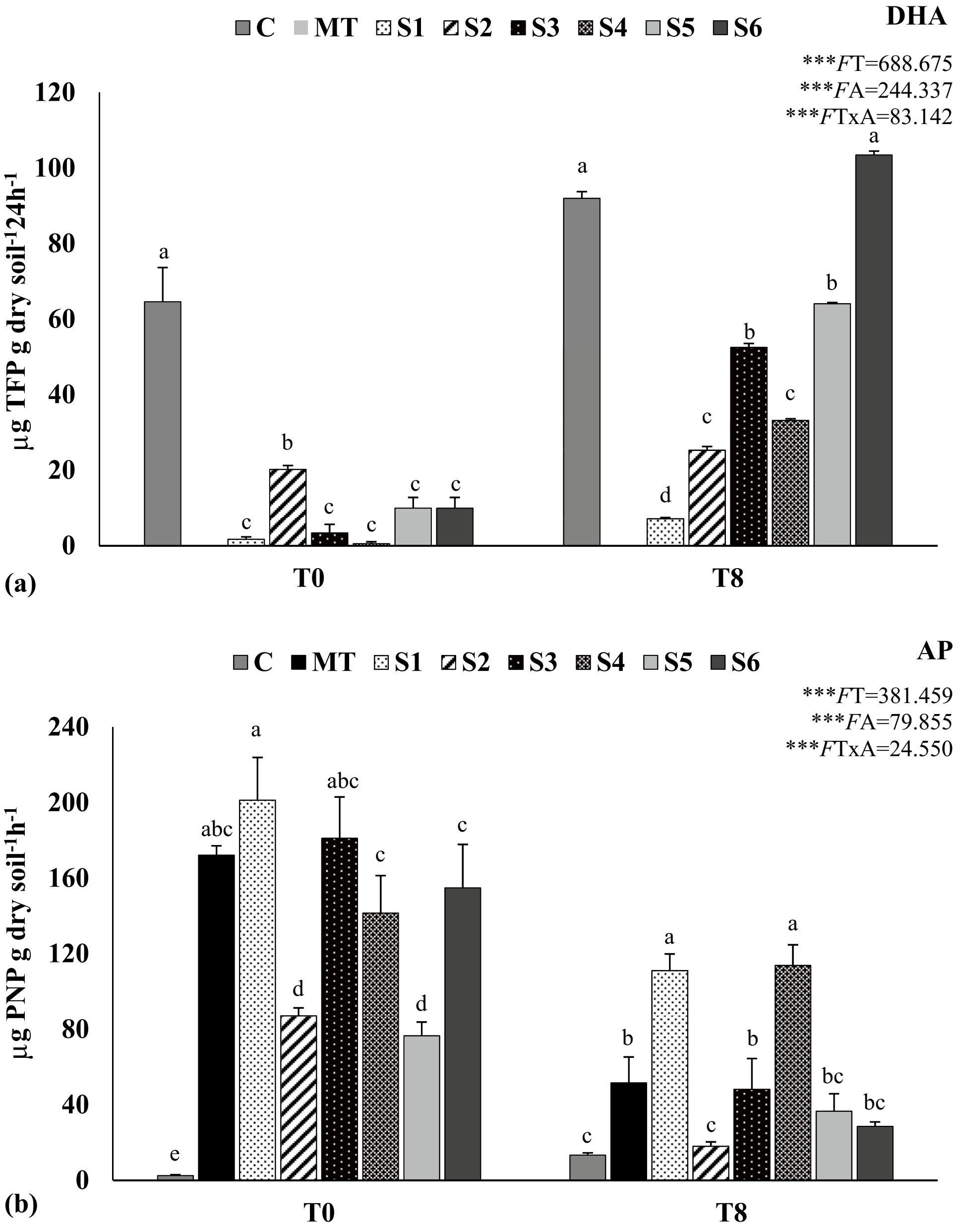

Fig. 3 Dehydrogenase (DHA) (**a**) and acid phosphatase (AP) (**b**) activities in different substrates immediately (T0) and 8 months (T8) after the incorporation of amendments. The error bar represents the SD ($n = 3$). A two-way ANOVA was performed to determine the influence of time (T0, immediately after the incorporation of amendments; T8, 8 months after the incorporation of amendments) and amendments (control (C), 100% of agricultural soil; MT, 100% of mine tailings; S1, 50% of mine tailings + 50% of agricultural soil; S2, S1 + 3% of lime ($CaCO_3$); S3, S1 + 6% of rock phosphate; S4, S1 + 10% of compost; S5, S1 + 10% of compost + 3% of lime ($CaCO_3$); S6, S1 + 10% of compost + 6% of rock phosphate) on DHA and AP activities. Results are shown with the test statistic for each case (T, time; A, amendments; TxA, time x amendments) and as: NS, non-significant at the level $P > 0.05$; *significant at the level $P < 0.05$; ** significant at the level $P < 0.01$; *** significant at the level $P < 0.001$. A one-way ANOVA was performed to determine the influence of amendments on DHA and AP activities in substrates for each time. Means for different treatments for each time with different letters are significantly different from each other according to Tukey's test. For DHA activity, the F values of one-way ANOVA are $F = 106.383$ ($P < 0.001$) and $F = 200.254$ ($P < 0.001$) for T0 and T8, respectively. For AP activity, the F values of one-way ANOVA are $F = 53.495$ ($P < 0.001$) and $F = 48.793$ ($P < 0.001$) for T0 and T8, respectively

The addition of organo-mineral amendments and the incubation time (8 months) had a significant ($P < 0.001$) effect on actinomycetes enumeration (Fig. 4c). Non-amended mine tailings did not support the growth of actinomycetes; however, the initial incorporation of amendments slightly increased the numbers of these microorganisms in substrates

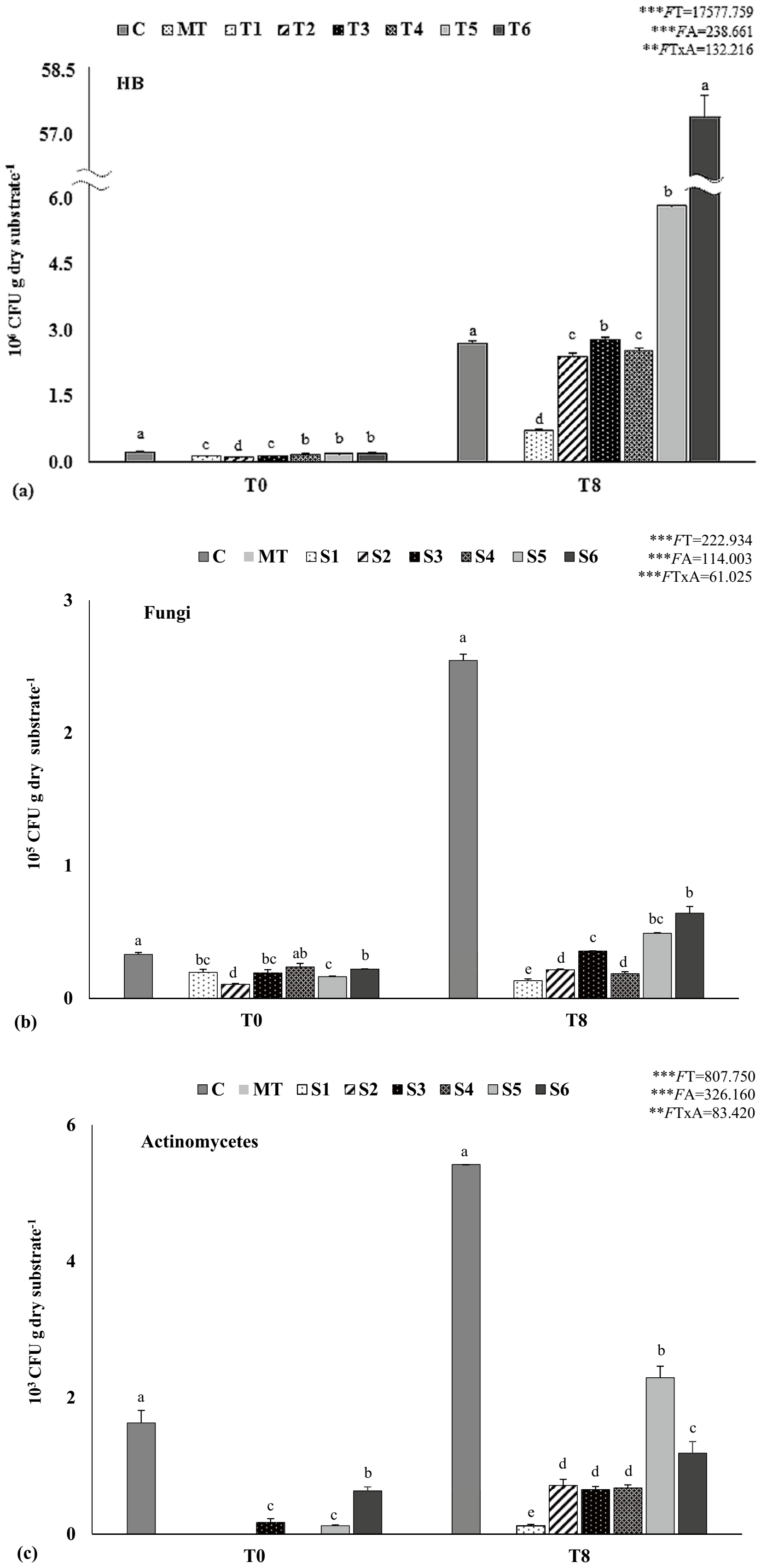

Fig. 4 Enumeration of heterotrophic bacteria (HB) (**a**), fungi (**b**), and actinomycetes (**c**) in different substrates immediately (T0) and 8 months (T8) after the incorporation of amendments. The error bar represents the SD ($n = 3$). A two-way ANOVA was performed to determine the influence of time (T0, immediately after the incorporation of amendments; T8, 8 months after the incorporation of amendments) and amendments (control (C), 100% of agricultural soil; MT, 100% of mine tailings; S1, 50% of mine tailings + 50% of agricultural soil; S2, S1 + 3% of lime ($CaCO_3$); S3, S1 + 6% of rock phosphate; S4, S1 + 10% of compost; S5, S1 + 10% of compost + 3% of lime ($CaCO_3$); S6, S1 + 10% of compost + 6% of rock phosphate) on DHA and AP activities. Results are shown with the test statistic for each case (T, time; A, amendments; TxA, time x amendments) and as: NS, non-significant at the level $P > 0.05$; *significant at the level $P < 0.05$; ** significant at the level $P < 0.01$; *** significant at the level $P < 0.001$. A one-way ANOVA was performed to determine the influence of amendments on DHA and AP activities in substrates for each time. Means for different treatments for each time with different letters are significantly different from each other according to Tukey's test. For bacteria, the *F* values of one-way ANOVA are $F = 169.886$ ($P < 0.001$) and $F = 8420.389$ ($P < 0.001$) for T0 and T8, respectively. For fungi, the *F* values of one-way ANOVA are $F = 56.034$ ($P < 0.001$) and $F = 138.199$ ($P < 0.001$) for T0 and T8, respectively. For actinomycetes, the *F* values of one-way ANOVA are $F = 157.262$ ($P < 0.001$) and $F = 366.138$ ($P < 0.001$) for T0 and T8, respectively

S3, S5, and S6. After 8 months, actinomycetes populations reached their maxima in substrates S5 (2.29×10^3 CFU g^{-1} dry weight substrate) and S6 (1.19×10^3 CFU g^{-1} dry weight substrate).

4 Discussion

Incorporating organo-mineral amendments to Kettara mine tailings ameliorated its physicochemical, biochemical, and biological properties, restoring essential soil functions which allowed plant germination, pivotal for the successful establishment of vegetation cover in these highly contaminated areas.

The application of alkaline mineral amendments, such as lime, is a common practice to neutralize the pH of metal-contaminated acidic mining wastes (Dybowska et al. 2006; RoyChowdhury et al. 2015; Liu et al. 2018; Pardo et al. 2017). However, in the present work, despite the fact all amendments raised the pH of mine tailings, the higher increases were observed in substrates containing agricultural soil, green waste compost and lime (S5) or rock phosphate (S6), where pH reached values close to neutrality. This could be explained by the high content of $CaCO_3$ in lime-containing substrates, and by the base cations present in compost (Walker et al. 2004; Naramabuye and Haynes 2006; Mensah and Frimpong 2018; Qaswar et al. 2020). Several authors have also highlighted the importance of incorporating organic materials to improve soil quality and repair the biological functions of environmentally degraded soils (Pérez-de-Mora et al. 2005; 2006; Diacono and Montemurro 2011; Larney and Angers 2012; Gil-Loaiza et al. 2016), such as the case of Pb/Zn-contaminated mining sites (Galende et al. 2014) and of other multi-element-contaminated areas (Beesley et al. 2010). In this study, the substrates incorporating green waste compost showed on average 3–4 times higher TOC levels than substrates amended individually with lime or rock phosphate. These results are particularly important in case of Kettara mine wastes, since they are characterized by very low TOC levels. The amount of available P in mine tailings was also significantly increased by the addition of organo-mineral amendments, in particular in the agricultural soil, green waste compost, and rock phosphate-containing substrate (S6), indicating that the incorporation of the organic material had an additive effect to the lone incorporation of rock phosphate. In fact, according to Li et al. (2020), the addition of green waste compost to a metal-contaminated soil remarkably increased the levels of available P even at low application rates ($< 10\%$). Similar results were obtained by Farrel and Jones (2010) in a highly acidic metal-contaminated soil.

Our results showed that the mixture of non-contaminated agricultural soil had a dilution effect on pseudo-total concentrations of Cu, Zn, and Pb, and the incorporation of organo-mineral amendments tended to further reduce the concentrations of these metals in all substrates. Despite this general abatement in metal contamination, according to the Canadian Soil Quality Guidelines, pseudo-total Cu concentrations in all amended substrates (366.91 to 521.35 mg Cu kg^{-1}) were still above the limit recommended for agricultural, parkland, commercial, and industrial land uses (63–91 mg Cu kg^{-1}), while pseudo-total Zn and Pb concentrations were below the recommended limits for all land uses. Moreover, Cu and Zn concentrations in amended substrates were above the target values (36 mg Cu kg^{-1} and 140 mg Zn kg^{-1}) reported in Dutch Standards. This scenario is in accordance with the PI determined for Cu, indicating that mine tailings are severely contaminated by this metal even after the incorporation of organo-mineral amendments.

The analysis of pseudo-total metal concentrations is a consensual indicator that provides information on soil enrichment by metallic elements, but it is not enough to estimate their availability and mobility and their consequent impact on ecological processes (Boularbah et al. 2006a; El Khalil et al. 2008; Daldoul et al. 2019; Massas et al. 2013). The analysis of metal extractable fractions has been used for ecological risk assessment of contaminated sites, especially concerning the remediation of abandoned mines (Clemente et al. 2003; Esshaimi et al. 2013; Benidire et al. 2020). In the present study, results showed that $CaCl_2$-extractable Cu and Zn concentrations tended to be lower in substrates with higher pH (S2, S5, and S6). Indeed, previous studies have shown that the increase of a pH unit leads to a decrease of metal solubility in soil by a factor of 100 (Fageria et al. 2002). Therefore, the increase of pH in Kettara mine tailings seems to have a strong influence on the reduction of extractable Cu and Zn concentrations in all amended substrates, as corroborated by the negative correlations obtained between pH and Cu ($r = -0.793$, $P < 0.01$) and Zn ($r = -0.839$, $P < 0.01$) extractable concentrations (Appendix 2 – supplementary material). Similar results were obtained in other studies where the input of green waste compost and/or alkaline mineral amendments reduced metal availability in mine tailings (van Herwijnen et al. 2007; Beesley et al. 2010; Bade et al. 2012). Moreover, Cu and Zn extractable concentrations were also negatively correlated with available P ($r = -0.510$, $P < 0.05$ and $r = -0.553$, $P < 0.05$, respectively; Appendix 2 – supplementary material), suggesting that the release of P present in the compost and/or in rock phosphate contributed to the chelation of metals, increasing their immobilization in substrates (Li et al. 2020). Therefore, the decline of metal availability in soils may contribute to enhancing its health and quality, as the biochemical and biological activity is in large part influenced by the available forms of metals, rather than by their total concentrations (Zhang et al. 2013; Lee et al. 2020).

The negative influence of available metals on DHA activity has been reported by several authors (Maliszewska-Kordybach and Smreczak 2003; Wiatrowska et al. 2015; Łukowski and Dec 2018). In this work, Cu and Zn available concentrations impacted negatively the overall microbial activity of untreated mine tailings, as proven by the absence of DHA activity. However, the incorporation of organo-mineral amendments ameliorated the physicochemical properties of mine tailings, leading to an enhanced activity of this enzyme. This improvement was mainly observed after 8 months, reflecting the importance of the substrates' maturation, the greatest increase being found in substrate S6 which combined agricultural soil, compost, and rock phosphate. This finding can be explained by the microbial biomass present in the added compost, and by the increasing levels of available P provided by the rock phosphate, which certainly enhanced microbial growth (Duong et al. 2013). Indeed, a strong positive correlation ($r = 0.685$, $P < 0.01$; Appendix 2 – Supplementary material) was observed between available P content and DHA activity. In addition, DHA activity was also positively correlated with pH ($r = 0.718$, $P < 0.01$), whereas a negative, but not significant, correlation with the concentrations of extractable Cu and Zn forms was observed. Positive correlations between DHA activity and soil pH have also been found by other authors (Jiang et al. 2003; Benidire et al. 2021), which may be explained by the contribution of pH to the overall microbial activity, since it influences microorganisms' survival and growth (Wolińska and Stępniewska 2012).

A different trend was observed for the acid phosphatase activity, since its values were significantly lower in agricultural soil and in most amended substrates if compared to those found in mine tailings. In addition, an overall reduction was observed in the activity of this enzyme after 8 months. This decrease can be explained by the rise of pH ($r = -0.560$, $P < 0.05$) and/or by the increase of available P in most amended substrates resulting from rock phosphate dissolution and/or from the conversion of insoluble phosphate into soluble P forms by phosphate solubilizing microorganisms. Indeed, several studies reported that phosphatase activity is negatively regulated by available P in soil (Olander and Vitousek 2000), and is highly dependent on soil pH (Herbien and Neal 1990; Šarapatka et al. 2004).

Given the crucial role of microorganisms on soil functions, evaluating changes on microbial communities may provide important information on the efficacy of remediation techniques on the restoration of soil quality of anthropogenically degraded areas (Sharma et al. 2010; Epelde et al. 2009a, b). In this work, none of the microbial groups analyzed was detected in untreated mine tailings, reflecting the harsh conditions and the poor microbiological status of this highly contaminated substrate. However, the addition of amendments had a positive effect on microbial counts, especially after 8 months, indicated by the general improvement of the physicochemical properties of mine tailings, including reduced metal contamination. Despite no significant correlations being observed between microbial counts and the extractable Cu and Zn concentrations, the greater increases in microbial abundance were observed in substrates containing agricultural soil, compost and lime or rock phosphate, which showed at same time the lowest metal availability. Interestingly, the heterotrophic bacterial counts in these substrates surpassed the numbers found in the agricultural soil (control), highlighting the powerful effect of the combined use of mineral and organic amendments on soil microbiota. The microbial biomass already existing in organic amendments, and the addition of carbon/nutrient-rich materials, may also explain the results obtained for the abovementioned substrates. In fact, the twofold effect of organic amendments was also already reported by other authors (Pérez-Piqueres et al. 2006; Tripathy et al. 2014; Strachel et al. 2017).

In this study, the mitigation of the harsh conditions of mine tailings by the incorporation of amendments was corroborated by the results obtained for the germination tests. Indeed, the GI decreased abruptly in all amended substrates, with the best results being obtained for substrates S5 and S6. Likewise, Wang et al. (2018) also reported higher germination rates in Cd-contaminated soils supplemented with vermicompost and biochar.

5 Conclusion

The application of different combinations of organo-mineral amendments improved the physicochemical properties of acidic Kettara mine tailings, which allowed the restoration of some pivotal soil functions, such as those related to nutrient cycling, habitat for microbial communities, and support of plant systems. The substrates incorporating agricultural soil and green waste compost combined either with lime (S5) or with rock phosphate (S6) successfully reduced the environmental risk posed by these mine tailings. The increase of pH and fertility levels, and of microbial activity, accompanied by the reduction of total and extractable Cu and Zn concentrations, suggests the suitability of both mixtures of amendments to remediate the acidic tailings of Kettara mine. Notwithstanding, the establishment of field trials to further evaluate the effectiveness of these amendments' combinations to support plant growth is of utmost importance.

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Author contribution Benidire Leila designed the study, performed the experiments, and wrote the manuscript. Pereira Sofia contributed substantially to the statistical analysis of the data, the writing and revising of the manuscript. Castro Paula provided critical feedback and helped shape the final manuscript. Boularbah Ali conceived the original idea and was in charge of overall direction and planning of this work.

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Declarations

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Competing interests The authors declare no competing interests.

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