

Leachability and plant availability of trace elements in Pb/Zn mine soil treated with different amendments

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ABSTRACT/RESUME

Abstract: A multi metal polluted soil from Jebel Ressay mining area (Southern of Tunisia) was treated with four cost-effective amendments (CaCO₃, phosphate rock, activated carbon, and exhausted olive cake ash). Lixivate from each treated and untreated soil were analyzed, and ryegrass-seed bioassays (*Lolium perenne* L.) were performed. The Cd, Pb and Zn leaching was reduced by all amendments mainly due to alkalinity increase. Phosphate rock addition to the soil reduced metal concentration in plant tissue. Additionally, the activated carbon amendment significantly decreased accumulation of Cd, Zn and Pb in the plant studied. The results obtained in this study suggest that the addition of CaCO₃ offered the best compromise as it successfully reduced both the leaching and the phytoavailability of the three considered metals.

I. Introduction

Mining is one of the most important sources of heavy metals in the environment. In Northern Tunisia, there are ~50 metallic mines (Pb, Zn, Fe, etc.), >98% of which are now closed and may be long-term sources of environmental pollution [1]. While physico-chemical extraction techniques generally imply degradation of soil structure and high costs, in situ stabilization of heavy metals in soils, by the addition of various amendments, can improve soil physico-chemical properties (increase of soil pH, reduce heavy metals mobility, bioavailability and toxicity, supply beneficial nutrients, etc.) [2,3], are less expensive and therefore are more suitable for remediation of extensive areas of low-value land [4].

Numerous amendments have been proposed and tested for in situ stabilization of heavy metals in soils such as lime [5], phosphate [6], and organic matter [7], as well as various industrial products, such as zeolites [8] and red-mud [9,10]. Their use also achieves reduction of waste disposal through revalorization of industrial wastes into industrial co-products. Experiments carried out by Pérez de Mora et al. [11] showed that the leaching of Cd, Cu

and Zn in a soil affected by the Aznalcóllar (Spain) mine accident was reduced using sugar beet lime, biosolid compost and municipal waste compost as amendments. Caliani and Barba-Brioso [12] assessed the potential value of a waste sludge generated during the processing of marble stone for remediation of acid mine soils. They showed that in the abandoned mine site of Tharsis (Spain) this amendment, joint with lime, significantly reduced the water-soluble concentrations of Cu, Zn, Pb and Cd one month after the amendment application, remaining extremely low throughout the one-year monitoring period.

In previous studies, we have evaluated the use of several amendments (phosphate rock, activated carbon, CaCO₃ and exhausted olive cake ash) to reduce the leachability and the phytoavailability of Cd, Zn, and Cu in a slightly metal polluted soil [13]. Our experiments show that the most effective treatment to reduce both the leachability and the phytoavailability of three metals is CaCO₃. Pursuing this further, the objective of this study is to ascertain the feasibility of these amendments as immobilizing agents of toxic elements present in a highly contaminated mine soil. Based on these results, it will be possible to further elucidate the

benefits and/or potential risks, derived from the application of different types of amendments in the remediation of contaminated mine soils.

II. Materials and method

II.1. Experimental set-up

Heavy metal-contaminated soil has been sampled in Jebel Ressay Pb/Zn mine area in Ben Arous Province, Tunisia (36°36'40.5"N 10°18'58.8"E) . 1). In this mine, the mineralogy consists of abundant primary Pb, Zn and Fe sulfides (galena, sphalerite and pyrite), and abundant secondary minerals that occur as carbonates (cerussite, hydrozincite), silicates (hemimorphite) and iron oxides (hematite) hosted in limestones. Many other accessory minerals such as smithsonite, Pb-strontianite, barite, celestite, willemite, voltaite and zincite have been detected in the deposit [14-16]. Soil samples were collected from the top 10–20 cm at the mine sites. The samples comprised superficial tip material which was exposed to the atmosphere for several years. The soil sample (150 kg) was then air-dried for two weeks, crushed and sieved to a particle size of <2 mm and then stored at 4°C prior to use.

Soil texture was determined following the methodology of [17]. Soil pH was determined using 1:5 soil/H₂O [18]. The organic matter content was determined by wet combustion with sodium dichromate and sulfuric acid without heat application [19]. Cation exchange capacity was determined using the ammonium-saturation and distillation method [20]. The chemical compositions of contaminated soil and amendments were measured in laboratory of Water Energy and Environment by inductively coupled plasma atomic emission spectroscopy (ICP-AES) after calcination at 450°C followed by either (i) Li-metaborate/Li-tetraborate fusion for major elements [21] or (ii) acid digestion (HNO₃, HClO₄ and HF) for trace metals. All the analyses were carried out in triplicate. The total contents of Pb, Cd and Zn in the polluted soil were than the maximum admissible levels for agricultural soils [22].

Diffraction analyses by X-ray (XRD) were performed on powdered bulk mine soil and phosphate rock samples after grinding of representative sub-samples using a Philips X'Pert-PRO diffractometer equipped with a diffracted beam monochromator and an X'Celerator multichannel detector. The analyses were performed under the following conditions: Cu K α radiation, 40 kV, 30 mA, step scanning at 0.01°/2 θ /s in the range 1–30°. Mineral identification was made by reference to the International Center for Diffraction Data (ICDD) and the International Crystal Structure Database (ICSD) patterns.

Four amendments were tested: calcium carbonate (CaCO₃), phosphate rock, activated carbon and exhausted olive cake ash. Calcium carbonate was of pro analysi grade (Merck). Phosphate rock comes

from the Metlaoui ores located in the south of Tunisia. Activated carbon was produced in our laboratory from date stones residues [23]. The exhausted olive cake ash from the exhausted olive cake combustion was obtained from SIOS ZITEX (Sfax, Tunisia).

The amendments were added to the mine soil at 5% (w/w), and the combination was mixed thoroughly to obtain homogeneity, followed by equilibration for 60 d at 20 °C and at 70% of the water holding capacity (WHC). The soil without amendments was used as reference. The application rates of the different amendments used in this study were selected based on rates frequently used by other researches [24].

II.2. Heavy metal leachability

To evaluate the effects of amendments on heavy metal leachability, the experimental setup was designed in order to collect leachates and to guarantee a sufficient volume of soil for root growth. It was composed of two parts: a soil compartment and a leachate tank. The soil compartment consisted of a polyethylene pot (30 × 20 cm) with a depth of 20 cm. The bottom of the soil compartment was perforated to allow free drainage. At the bottom of each pot, a Whatman filter was installed followed by fiberglass layer in order to prevent coarse material from draining out of the pot and ensure free-drainage conditions. The leachate tank was placed below the plastic pot, and consisted of a polyethylene box (30 × 20 cm) with a depth of 20 cm. Total volume of leachate was collected and quantified at 2, 4, 9 and 12 weeks after sowing. pH-H₂O was determined immediately after the collection. Leachates were then filtered through a 0.45 μ m membrane filter. Concentrations of Cd, Pb and Zn were measured by ICP-AES spectrometry (Jarrell Ash).

II.3. Phytoavailability

To examine the effect of the addition of amendments on the phytoavailability of contaminants, ryegrass (*Lolium perenne* L.) was grown in plastic pots filled with contaminated soil, prepared as described above. Ryegrass seeds were initially sown in peat-based horticultural compost. Two week after germination, the seedlings were then transplanted into the mine soil (5 seedlings/pot, 5 pots per treatment). The trials were conducted under controlled greenhouse conditions (temperature: 15-25°C; relative humidity: 60-70%) and a daily watering schedule.

At the end of the experiment, shoots were harvested, dried at 80°C for 48 h. Quantification of heavy metal concentrations within the shoots was performed by ICP-AES after a tri-acid (HClO₄, HNO₃ and HF) dissolution.

II.4. Statistical analysis

Statistical testing was performed using a one way analysis of variance (ANOVA). For significant differences ($p < 0.05$), a post hoc Tukey honestly significant difference test was used to further elucidate differences among means ($p < 0.05$). Pearson's correlation coefficients (r) were calculated to determine the relationships between the heavy metal concentrations in leachates and their pH. Two levels of significance were considered: $p < 0.05$ and $p < 0.01$. All statistical analyses were carried out using SPSS16.0 for Windows.

III. Results and discussion

III.1. Characterization of contaminated soil and amendments

Table 1, shows some characteristics of the soil arising from the jebel Ressay mining site and the amendments used. The soil used was a sandy clay loam. It was characterized by a slightly alkaline pH. The organic matter content was 0.9% and the CEC was 12.6 cmol/ kg. The presence of vegetation in soils of the mining sector may explain the highest organic matter contents. High contents of Pb, Cd and Zn were found in the mine soil due to the contamination caused by the local Pb/Zn smelter. Concentrations of three metals were significantly higher than limit values in soils which are 2 mg/kg soil for Cd, 300 mg/kg soil for Zn and 100 mg/kg soil for Pb [22].

XRD analysis showed that the main mineral phases of the untreated soil include calcite, dolomite, clay mineral, cerusite (PbCO_3), Hemimorphite ($\text{Zn}_4\text{Si}_2\text{O}_7\cdot 2\text{H}_2\text{O}$) and Ankerite ($\text{Ca}(\text{Fe}, \text{Mg}, \text{Mn})(\text{CO}_3)_2$). The phosphate rock consists mainly of carbonate fluoroapatite with lesser amounts of quartz and calcite.

III.2. Leaching analysis

After two weeks, the application of all amendment significantly increased leachates pH compared to the control (Fig. 1). At the end of the experiment, this pH increase was still statistically significant ($p < 0.05$) with the exception of activated carbon and phosphate rock treatments.

Compared to non-amended soil, the application of all amendments significantly ($p < 0.05$) decreased the amounts of Pb, Cd and Zn in leachates (Fig. 2a, b, c). In fact, the Cd concentration was decreased by 93.42%, 93.56%, 94.46% and 94.36% in the phosphate rock, activated carbon, CaCO_3 and exhausted olive cake ash treatments, respectively. The Zn concentration was lowered by 94.78%, 85.45%, 98.32% and 87.55%, in the phosphate rock, activated carbon, CaCO_3 and exhausted olive

cake ash treatments, respectively. Finally, in the presence of phosphate rock, activated carbon, CaCO_3 and exhausted olive cake ash, the Pb concentration was lowered by 85.55%, 65.55%, 88.88% and 35.55%, respectively.

Table 2 shows that a very high positive Pearson's correlation coefficients was observed between Cd-Zn, Cd-Pb et Zn-Pb ($r = 0.98$, $p < 0.01$, $r = 0.98$, $p < 0.01$ and $r = 0.98$, $p < 0.01$, respectively). Furthermore, a significant negative correlation (for $p < 0.01$) was observed between heavy-metal concentrations (Cd, Zn and Pb) and pH in leachates during the experiment. These results can be explained by the fact that acidity is one of the most important factors controlling solubility and adsorption-desorption of metal in soils [25-27]. Consequently, the alkaline amendments decreased metal mobility/bioavailability mainly because they raised soil pH. The application of phosphate rock amendment to the polluted mine soil significantly reduced Cd, Zn and Pb concentrations in leachates. The retention of heavy metal cations by phosphate minerals occurs by several mechanisms. Several studies reported that Pb fixation by phosphate amendments is mainly due to the dissolution/precipitation mechanism through the formation of chloropyromorphite or hydroxypyromorphite [28,29]. However, the contribution of this mechanism in Pb stabilization by phosphate minerals is still open to debate [30,31]. In particular, Mavropoulos et al. [32] found that sorption mechanisms contributed to the immobilization of about 30% of the total Pb. Recent studies also reported the contribution of surface mechanisms in the overall fixation of Pb [33,34]. As for Cd, Da Rocha et al. [35] suggested that Cd immobilization can be associated to the ion exchange and complexation mechanisms. Similar results were obtained also in subsequent studies suggesting that Cd fixation results in the formation of Cd-containing phosphates [36-40]. Several studies experimentally determined similar immobilization mechanisms for Zn [30,41,42]. In particular, the fixation mechanism involves the formation of Zn-phosphate on the surface of the amendment grains, followed by the diffusion of Zn ions into the phosphate structure. Finally, being carbonaceous materials, activated carbon is able to limit metal leaching through their repartition from the contaminated matrix to the activated carbon particle. These pollutants are then very strongly bound and considered unavailable to further leaching which can explain the strong metal immobilization observed with this treatment [43].

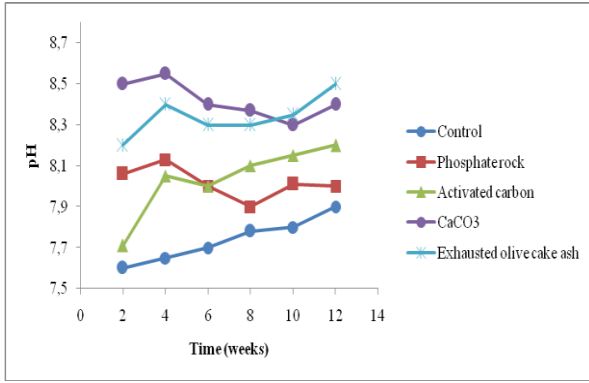


Figure 1. Evolution of leachates pH as a function of time. Each point represents the average of five replicates.

III.3. Heavy metal phytoavailability

Compared to the control soil, all treatment significantly ($p < 0.05$) decreased the concentrations of all heavy metals in shoots of plants. The effectiveness of the amendments in Pb, Cd and Zn immobilization follows the sequence: $\text{CaCO}_3 >$ activated carbon $>$ phosphate rock $>$ exhausted olive cake ash (Fig. 3 a, b, c). Pb, Cd and Zn concentration in shoots was lowered by 33,81%, 84,22% and 79,03% respectively for phosphate rock, 81,36%, 93,70% and 94,50% respectively for CaCO_3 , 80,52%, 82,38% and 70,16% respectively for activated carbon and 49.4%, 33.81% and 32.65% for exhausted olive cake ash. The results obtained in this work indicate that only CaCO_3 addition effectively contributed to a strong reduction of Cd, Zn and Pb phytoavailability and leachability which shows that the pH could be one of the most important parameters responsible for the control of these two pathways [44]. Phosphate rock was the third efficient treatment in decreasing metal concentrations in plants. This effect may be attributed to both the capacity of P to form sparingly soluble metal phosphates in soils and the enhanced P availability that in the tested soil may represent a limiting factor to plant growth [45].

The effect of contact time on the Ni(II) ions adsorption was determined and the corresponding variation of removal rate is shown in Figure 3. The obtained results indicate that the removal of Ni(II) ions increased with time up to 90 min and thereafter increased slowly. The removal rate of Ni(II) ions was 35.4 % at 90 min and 35.46 % at 120 min by chicken eggshells, respectively. According to the results, the equilibrium reached at 120 min was taken as the optimal contact time for the subsequent experiments.

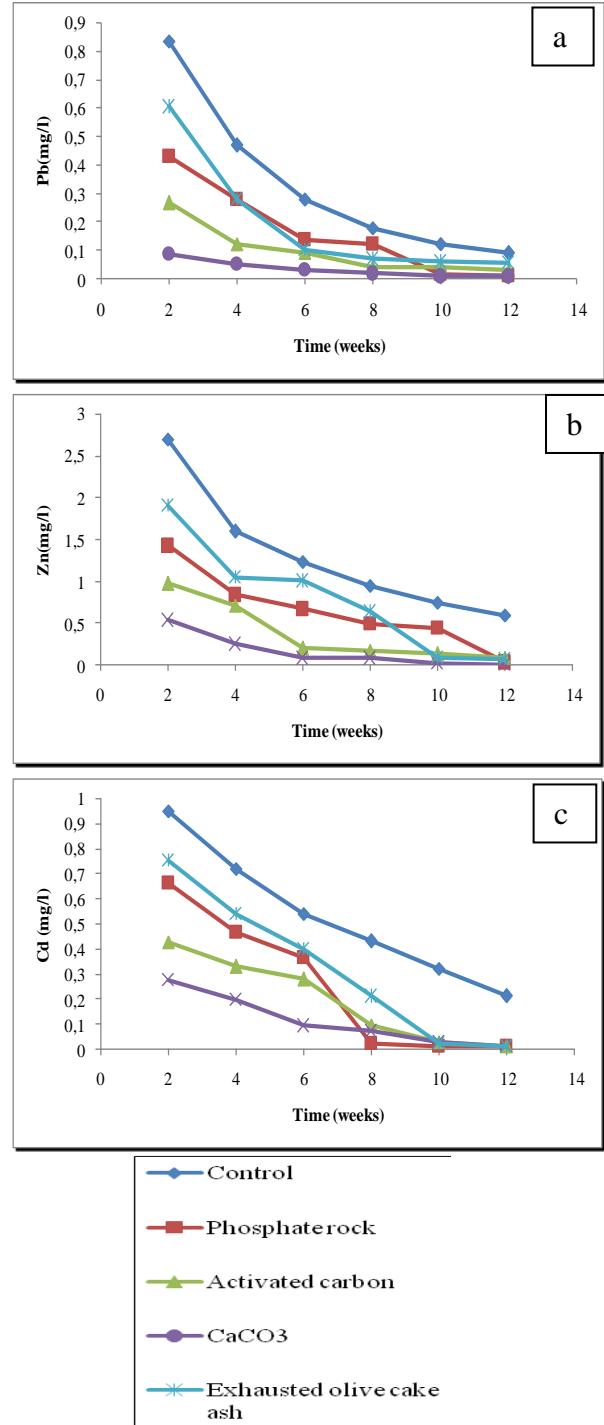


Figure 2. Evolution of (a) Pb, (b) Zn and (c) Cd concentrations in leachates as a function of time. Each point represents the average of five replicates.

Table 1. Characteristics of the mine soil and amendments used in the experiment, mean \pm SD ($n = 3$).

Parameter	Mine soil	Phosphate rock	Exhausted olive cake ash	Activated carbon
pH	7.6 ± (0.02)	7.34 ± (0.04)	10.4 ± (0.05)	6.78 ± (0.03)
Clay	25 ± (0.03)	-	-	-
Silt	34 ± (0.5)	-	-	-
Sand	41 ± (1.9)	-	-	-
CEC	12.6 ± (0.3)	-	-	-
Organic matter, %	0.9± (0.2)			
Fe ₂ O ₃ (%)	-	0.33 ± (0.03)	2.51 ± (0.01)	-
Al ₂ O ₃ (%)	-	0.58 ± (0.08)	9.04 ± (0.04)	-
P ₂ O ₅ (%)	-	29.01 ± (0.15)	16.17 ± (0.85)	1.4 ± (0.01)
K ₂ O (%)	-	-	3.40 ± (0.01)	-
MgO (%)	-	0.63 ± (0.03)	2.62 ± (0.01)	-
CaO (%)	-	49.7 ± (0.25)	22.84 ± (0.12)	-
Cd	Total	53 ± (0.02)	38 ± (0.19)	ND
Pb	content	970 ± (0.02)	12.64 ± (0.06)	ND
Zn	(mg/kg)	9641 ± (0.24)	328 ± (1.64)	ND

Standard deviation in parentheses

ND: not detected

« - » not determined

Table 2. Pearson's correlation coefficients r between heavy metal and pH in leachates ($n=5$).

	Cd	Zn	Pb	pH
Cd	1	0.98**	0.98**	-0.97
Zn	0.98**	1	0.98**	-0.95
Pb	0.98**	0.98**	1	-0.93

** signification at $p < 0.01$

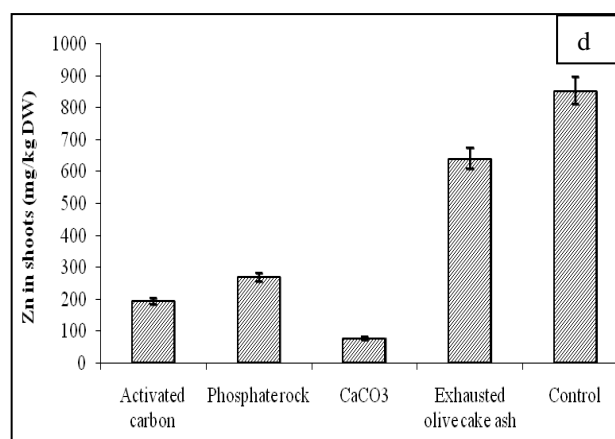
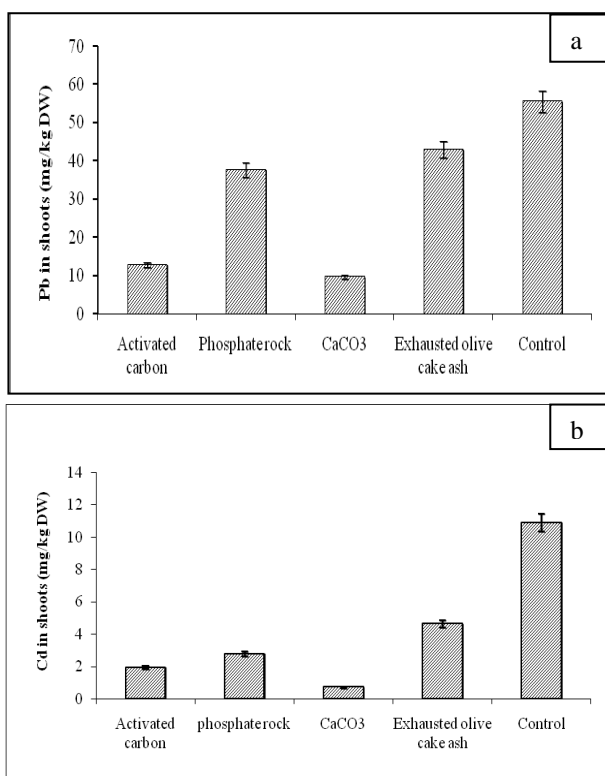


Figure 3. Average concentration of (a) Pb, (b) Cd, and (c) Zn. Values are average ($n=5$) \pm standard deviation. Columns with the same letter do not differ significantly at the 5% level.

IV. Conclusion

The high degree of contamination of the mine soil studied in this work provoked an elevated toxicity that impaired its basic ecological functions. Most of the amendments tested in this study acted as chemical stabilizers of heavy metals and reduced both leaching and phytoavailability of Cd, Zn and Pb. Indeed, Cd, Zn and Pb do not respond similarly to the same amendments. This highlights the difficulty in finding amendments that are equally effective for every target contaminant in multi-element contaminated soils. Overall, the addition of CaCO₃ turned out to be the best compromise to reduce both leaching and phytoavailability of the three considered metals.

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