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Phosphate Carbonated Wastes Used as Drains for Acidic Mine Drainage Passive Treatment

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Abstract

This study focused on the removal of heavy metals from a synthetic acid mine water by using continuous column experiments and Phosphate carbonated Wastes as alkaline drains. The passive treatment system targeted aims in neutralizing the acid mine drainage (AMD) containing high concentrations of dissolved iron and other metals. In Morocco, the phosphate mine industry produces huge quantities of overburden waste rocks (named herein PLW) which contain significant quantities of carbonates (calcite (46 wt %) and dolomite (16 wt %)). The column experiments were set-up in laboratory and the testing were run under anoxic and oxic conditions by using a hydraulic retention time was 15 hours. The inflow to the treatment system ranged 5.5 mL/min, with acidic pHs of around 3, concentrations of dissolved Fe, Mn, Al, Ca, Zn and Cu were 600, 20,166, 350, 15 and 23 mg/L respectively, containing also some traces of Co, Cr and Ni. The test results showed that pH became neutral and a significant decrease in terms of metal concentrations; in particular for Fe (600 to 120 mg/L), Al (160 to 1.7 mg/L) and Cu (23 to 0.002 mg/L).

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1. Introduction

The Kettara pyrrhotite mine site (Morocco) contains more than 3 million tons of mine wastes that were deposited on the surface without any concern for their environmental consequences. Fine tailings were stockpiled in the main pond, coarser wastes in a dyke which confine the fine tailings, and the additional wastes (coarse) are deposited as small spoils all around the site area which total surface is approximately 16 ha. The site have generated intense acid mine drainage (AMD) for more than 30 years. The local climate is as semi-arid with a mean annual rainfall of approximately 250 mm. Rainfall can occur over short periods and at high intensity. The annual potential evaporation typically exceeds 2,500 mm which induce important secondary precipitations. Static tests indicated that Kettara fine and coarse tailings are highly acid-generating, with NNPs ranging from -22 to -453 kg/CaCO₃/t [1]. The acid mine drainage is formed when sulfide minerals such as pyrite are exposed to oxygen and water. The surface drainage water at Kettara mine site had low pH (2.9 to 4.2) and elevated concentrations of sulphate (from 47 to 5000 mg/L) and iron (from 1 to 1200 mg/L) [2]. Concentrations of Cu and Zn reached 58 and 45 mg/L, respectively [3]. AMD is toxic towards aquatic organisms, damages ecosystems of receiving surrounding rivers and lakes, [4-5]. Pyrite and pyrrhotite are the most common sulfide mineral AMD sources, and occur in most metal polymetallic ores and coal deposits. Therefore AMD is very common and one of

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the worst environmental problems across the globe [5-7].

Various strategies to treat acidic mine drainage effluent could be appropriate depending on the volume of the mine discharge the concentrations of the various concerned metal species, and the available resources for construction and maintenance if a passive treatment system is privileged [8-10]. In the context of sustainable development, active treatment using chemical treatment systems to neutralize acidity [11] is not considered as viable option for long-term remediation of AMD-generating in closed or abandoned mined as it is the case of Kettara mine site [12-14]. Moreover, AMD passive treatment methods require less energy and use natural or even alkaline waste materials. Two main objectives are generally considered in the design of passive treatment system [15]: (i) generating sufficient alkalinity to neutralize the acidity, and (ii) decreasing the metals loading through oxidation/hydrolysis, and precipitation mechanisms. The dissolution of calcite (CaCO_3) and dolomite ($\text{CaMg}(\text{CO}_3)_2$), which are the principal active minerals encountered in limestones, leads to the increase of pH, alkalinity, and concentrations of calcium (Ca) and magnesium (Mg) in the AMD treated [10]. The dissolution of one mole of CaCO_3 consumes one to two moles of acidity and releases alkalinity (as HCO_3^-) into the aqueous solution [16]. The treatment of AMD with limestone can increase the pH up to 6.0-7.5 [17-18], allowing the metals to be removed from solution via precipitation, co-precipitation, and/or sorption processes. Many passive treatment systems incorporate crushed limestone in a drain channel that is flooded continuously with AMD in order to neutralize the acidity, thereby generating alkalinity and ensure metal up-takes. The flow within the drain must be optimized depending on AMD loading and the drain size. "Anoxic limestone drains" (ALDs) consists of crushed limestone of uniform size that is placed in a buried bed to intercept net-acidic AMD before exposure to atmospheric O_2 [18-19]. They are designed to exclude O_2 within the trench, thereby inhibiting $\text{Fe}(\text{OH})_3$ precipitation onto the limestone particles and increasing partial pressure of CO_2 . Continuous inundation and retention of CO_2 can enhance limestone dissolution and alkalinity production, because the rate and extent of limestone dissolution tend to increase with increased partial pressure of CO_2 (Pco_2) and/or decreased pH [20-23].

The other passive treatment systems are "oxic limestone drains" (OLDs), open channels (OLCs), successive alkalinity-producing system (SAPS), and limestone pond, microbial reactor systems, reactive walls, and bio-sorption systems [14], aerobic and anaerobic wetlands, biological and abiotic permeable, and reactive barriers [5, 24].

Morocco is the leading phosphate exporting country in the world and the third largest in terms of phosphate production after USA and Russia [25]. At the "Exploitations Minières du Gantour Company" 'Recette VI' site, 30 km North of the abandoned Kettara mine (Fig. 1), large quantities of overburden phosphatic carbonated wastes (PLW) are removed to reach the phosphate ore layers. Trucks transport the ore to a dry screening plant for phosphate separation/concentration and manage the oversize (>40 mm) low-grade material as wastes on site. Thus, the "Recette VI" phosphate mine produces two waste types: the overburden waste rock (PLW used in this study) and the wastes produced during the pre-concentration process. Large amounts of these wastes are produced and stockpiled in different waste rock dumps. The PLW are chemically stable. Indeed, results of waste phosphate rock leaching tests with deionized water in a liquid/solid ratio of 2.5 showed that a leachate had a neutral pH (6.5), low conductivity (0.3 mS/cm), and very low iron concentrations [26].

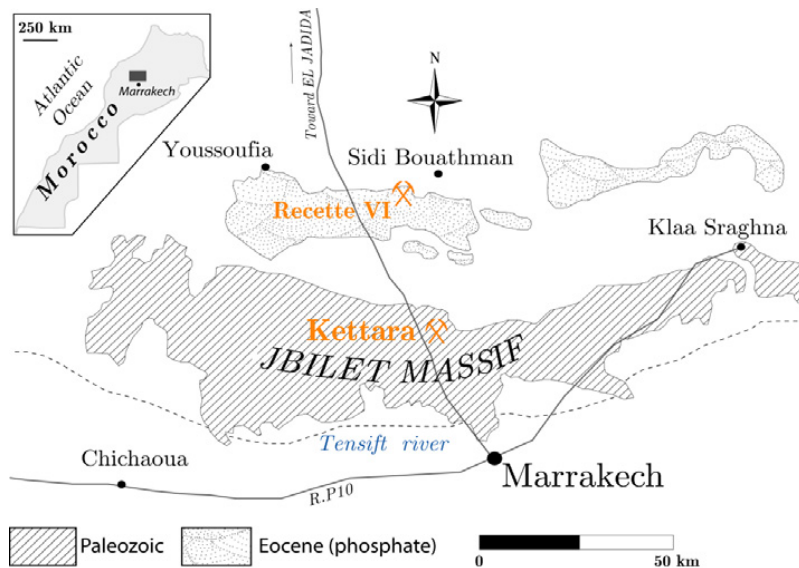


Figure 1. The geographic location of the Kettara and surrounding phosphate mines

Kettara mine site is currently one of several abandoned mine sites in Morocco producing AMD, that contaminates soils, surface and surface and ground waters [26-28]. More information on the water contamination characterization at the Kettara site can be found in Hakkou et al. [3-4]

The rehabilitation scenario being investigated at the Kettara mine (Figure 2) involves the use of fine PLW as both alkaline drain material and a 'store and release' (SR) cover material [29-30]. The rehabilitation process would require the collection of all the

Kettara coarse tailings and placing them over the fine tailings pond. Then, a capillary barrier made of fine PLW would be placed over the Kettara mine wastes, this barrier is expected to limit the infiltration of water to the underneath reactive mine wastes, hence controlling the production of AMD. The treatment of AMD with PLW drains can be an alternative mitigation solution and used in the case of possible AMD effluent exfiltration.

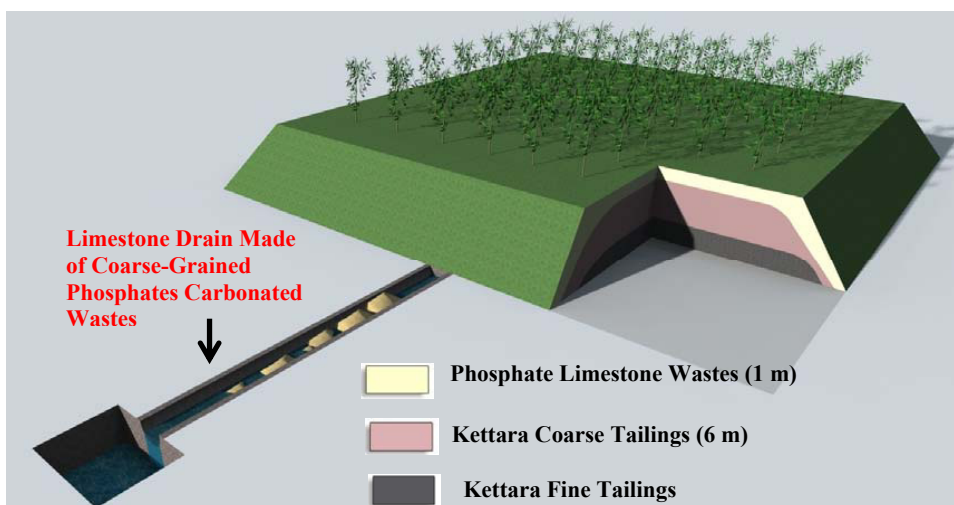


Figure 2. Scenario of abandoned Kettara mine reclamation

The present study focused on the neutralizing capacity of the PLW-drain based system for AMD treatment and also investigated whether residual phosphate in the PLW might affect the neutralization process. Building channel treatment systems at the closed Kettara mine with PLW material would be an inexpensive way to alleviate the exiting negative environmental effects of AMD. The study aims in evaluating, at laboratory scale, the capacity of PLW to be used to treat synthetic AMD similar to leachates from humidity cell tests conducted with Kettara mine tailings [2-3]. The synthetic AMD solution had a low pH (3) and contained 539 mg/L Fe, 3.4 g/L SO_4^- , 220 mg/L Ca, 160 mg/L Al, 20 mg/L Mn, 15 mg/L Zn, 23 mg/L Cu, and traces of Co, Cr, and Ni. The AMD treatment tests were conducted in both anoxic and oxic conditions in column tests with hydraulic retention times set at 15 hours.

2. Materials and Methods

2.1 Analytical Methods

The effluent obtained from the columns tests were filtered through a 0.45 μm nylon filter and were split into two subsamples. The first was acidified with HNO_3 (to avoid metal precipitation) for metals analysis and was refrigerated at 4°C. The second (un-acidified) was used for the analyses of sulfates, total alkalinity, and total acidity as well as pH and oxidoreduction potential and conductivity. The concentrations of metals were determined using inductively coupled plasma optical emission spectroscopy, ICP-OES (Perkin-Elmer Optima DV 7000 ICP-OES). Sulfates were analyzed using the barium chloride precipitation.

Alkalinity and acidity (expressed in mg CaCO_3/L) were measured by acid-base titration, to pH endpoints of 4.5 and 8.3, respectively. Solution pH was measured by a combination of pH electrode with temperature compensation (Instruments Hanna model 209) after calibration with standard buffer solutions of pH 4.01 and 7.01 (± 0.01). Redox potential was measured using a Pt/Ag/AgCl combination electrode (thermo Orion SureFlow[®]) which was calibrated with standard redox buffer solution of 220 mV. The measurements were corrected to obtain Eh expressed in mV/SHE (Standard Hydrogen Electrode). Conductivity was determined with a HANNA HI 8773 conductimeter.

A solution of synthetic AMD was prepared by adding various proportions of hydrous metals sulfate with 100 L of tap water (Table 1). The physico-chemical properties of the solution were: a pH of 3 ± 0.2 , a conductivity of 4.2 ± 0.3 mS/cm, and a total acidity of 3670 mg CaCO_3/L .

Table 1. Composition of the synthetic AMD added to the tap water used for the column tests

Component	Concentration mg/L	Source
Ca ²⁺	350 ± 20	CaSO ₄ .2H ₂ O
Al ³⁺	160 ± 10	Al ₂ (SO ₄) ₃ .14H ₂ O
Fe ²⁺	600 ± 70	FeSO ₄ .7H ₂ O
Co ²⁺	4.68 ± 2	CoSO ₄ .H ₂ O
Cu ²⁺	23 ± 4	CuSO ₄ .5H ₂ O
Zn ²⁺	15 ± 3	ZnSO ₄ .7H ₂ O
Mn ²⁺	20 ± 2	MnSO ₄ .H ₂ O
Mg ²⁺	197 ± 70	MgSO ₄ .7H ₂ O
SO ₄ ²⁻	4200 ± 300	-

2.2 Column Experiments

A dynamic treatment system was simulated in a column under anoxic (Fig. 3.a) and oxic conditions in open percolating systems (Fig. 3.b). Twenty kg of PLW were used in the anoxic column tests, while 44 kg of PLW were necessary in the oxic column tests. The particle grain size fraction of PLW used in these tests was between 0.8 mm and 3 cm [2]. The column was fed with synthetic AMD at its bottom to control solution flow and to have a good impregnation and targeted residence time. The up-flow was set at 5.5 mL/min to have a hydraulic residence time in the column of approximately 15 h. The flow was controlled using a peristaltic pump. At the exit of the anoxic treatment column, the effluent was aerated within a glass ball before being sent to a clarifier tank. Parameters such as pH, EC, alkalinity, metals, and sulfate concentrations were determined in samples collected at the exit of the column and after aeration.

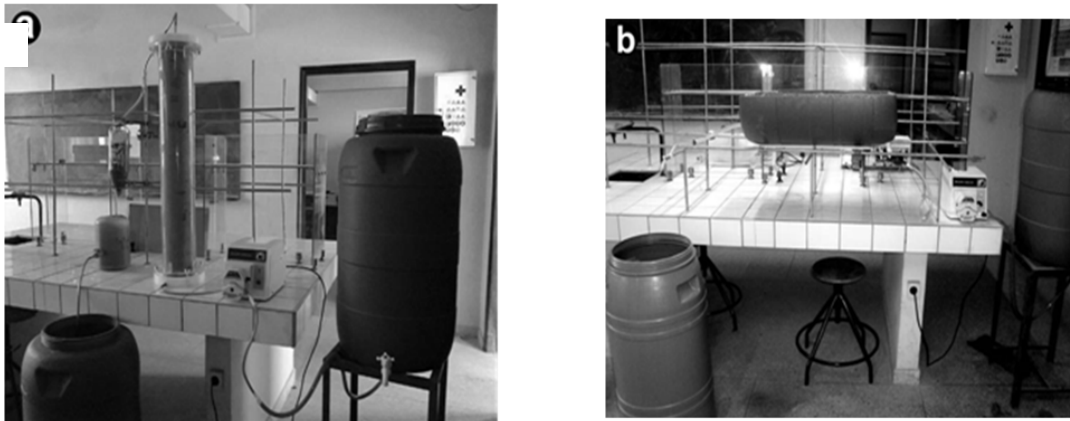


Figure 3. (a) Picture representation of the anoxic treatment set-up; (b) Picture representation of oxic treatment set-up.

3. Results and Discussion

The evolution of water quality after both treatment types is showed in Fig. 4. The results obtained were often similar in anoxic and oxic conditions. The pH (Fig. 4.a) of the treated effluent was between 8 and 7 in the first three days; thereafter, it reached a plateau around 6.5 over time. This pH is higher than the one obtained by Genty et al. (2012) [14] using limestone during AMD treatment in columns under anoxic conditions, this may be explained by the mineralogy difference of the two materials (PLW and limestone). The alkalinity of the treated solution for both columns increased by up to 700 mg CaCO₃/L in the anoxic condition treatment and up to 300 mg CaCO₃/L for oxic treatment.

The acidity (Fig. 4.c) of the effluent sharply dropped from 3000 to 2 mg CaCO₃/L during the first two days, after which it increased and then remained constant (at approximately 400 mg CaCO₃/L) for both types of treatment. The acidity of the solution was neutralized more efficiently under anoxic conditions than in the open system while the alkalinity increase in the anoxic system

was more significant than the one observed in the open system. This difference could be explained by the CO₂ pressure during anoxic conditions, which affects alkalinity production [8]. Retention of carbon dioxide (CO₂) can enhance limestone dissolution and alkalinity production because the rate and extent of limestone dissolution tends to increase with increased partial pressure of CO₂ and or decreased pH [20-23].

During the first day, the decreasing of the conductivity from 4000 to 1500 $\mu\text{S}/\text{cm}$ (figure 4.e) could be attributed to precipitation and co-precipitation of ions and adsorption of some major elements by alkaline phosphates. The redox potential continued to decrease during treatment until the fifth day (650 to 250 mV/SHE), and then stabilized with time at about 250 mV/SHE.

The concentrations of Ca and Mg (Fig. 4.f and Fig. 4.g) in the effluent decreased during the first day and then increased to a maximum value of about 900 mg/L for Ca and 350 mg/L for Mg. The high Ca concentration in the effluent indicates that calcite was responsible for the neutralization. The slight increase of Mg shows the low reactivity of dolomite. P concentrations were very low (<1 mg/L) in the effluents, indicating that either the fluorapatite was not dissolved during the tests or that P precipitated as phosphate complexes (iron phosphate precipitates readily) within the column solids.

The Cu and Al were totally eliminated (Fig. 4.h and Fig. 4.i). However, the Fe and Mn (Fig. 4.k and Fig. 4.j), which were totally eliminated during the first two days subsequently increased in the treated effluent, reaching 300 mg/L and 15 mg/L, respectively. Low Mn removal could be explained by several causes [31]: precipitation is inhibited if the Fe/Mn ratio is too high (which is the case in our study), precipitates were dissolved when ferrous concentrations are too high, and most other metals preferentially complex with sulphides before Mn. The reduction of the efficiency of the passive treatment could be attributed to precipitation and co-precipitation of Fe hydroxides and oxhydroxide onto the surface and passivate the PLW. The ochreous color was observed on the surface of PLW.

The sulfate concentrations (Fig. 4.l) in the effluent decreased during the first day by approximately 500 mg/L, but after five days, it reached a value of 2500 mg/L for the oxic treatment and 3500 mg/L for the anoxic treatment.

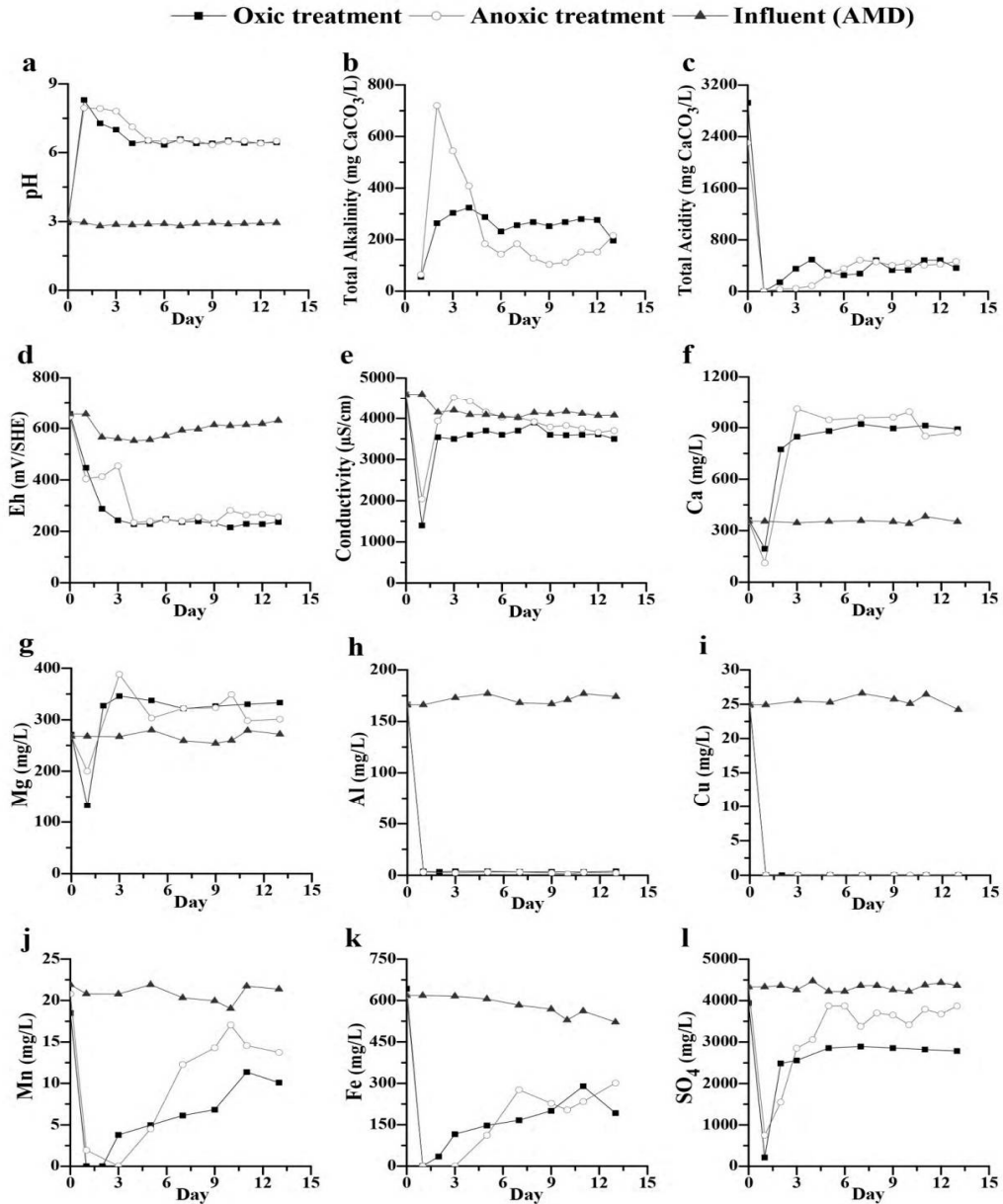


Figure 4: Water quality evolution for the influent (AMD) and columns effluent during treatment

4. Conclusions

In this work, the efficiency of PLW as an alternative alkaline material for the passive treatment of AMD was assessed in the laboratory. Field AMD waters were first characterized. Then, a series of experiments were conducted for the passive treatment of a pH 3 synthetic AMD solution containing 600 mg/L Fe, 4 g/L SO₄, 350 mg/L Ca, 160 mg/L Al, 20 mg/L Mn, 15 mg/L Zn, 23 mg/L Cu, and traces of Co, Cr, and Ni.

The tests were performed under anoxic and oxic conditions in columns tests with a hydraulic retention time of 15 h. These tests allowed us to evaluate the neutralization kinetics with particle size of the LPW (0.8 to 3 cm). The results indicated that carbonates (calcite and dolomite) from the PLW increased the alkalinity and the pH of the AMD, causing metals to precipitate. The pH increased from 3 to values between 6.5 and 8. After 3 days in columns tests under both anoxic and oxic conditions, the increase in pH and alkalinity was accompanied by a significant decrease in metal concentrations: for Fe (going from 618 to 300 mg/L), Al

(going from 177 to 2.5 mg/L) and Cu (going from 26 to 0.002 mg/L).

The results also showed that the neutralization capacity under oxic conditions is higher than under anoxic conditions. During columns tests, the Ca concentrations in the effluent increased rapidly, indicating that calcite was responsible for the neutralization. Dolomite dissolution seems to be negligible and fluorapatite was not dissolved under the testing conditions.

The anoxic treatment of synthetic AMD by PLW was more efficient than oxic treatment; more acidity was neutralized in the anoxic system and the rate of metal removal was greater than was observed in the open system. However, oxic conditions are more appropriate to a semi-arid climate, since it is difficult to exclude oxygen by submerging LPW beds under water when annual precipitation is so low.

The use of PLW to generate alkalinity in passive treatment systems design appears promising but more research and a technico-economic study is mandatory before full-scale implementation. Based on these test results, a pilot-scale open limestone PLW channel will be constructed and evaluated at the Kettara mine site under field conditions. However, it should be noted that such a channel cannot be expected to completely treat such a highly contaminated low-pH AMD; to remove all of the contaminants, the channel would have to be combined with other mitigation and rehabilitation actions. Sulfate-reducing bacterial bioreactors or natural solar evaporation ponds are some of alternative ways to be investigated.

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