Phosphate Amendments for the Inhibition of Acid Mine Drainage Generated from Sidi Kamber Tailings Facility

Amira Merchichi, Malek Ould Hamou, Mostafa Benzaazoua, Farid Aghilasse Mansour, Nabil Babahoum, and Hakim Rabia

Abstract—The aim of this paper was to evaluate the efficiency of phosphates product and by-products from the Djebel Onk mine for the control of acidic mine drainage generated by exposure of Sidi Kamber mine tailings to climatic conditions. Kinetic tests in humidity cells were carried out according to the modified ASTM standard, was used to investigate the geochemical behaviour of the Sidi Kamber tailings mixed with phosphate materials (phosphatic limestone wastes, raw low grade phosphate ore and phosphate mine tailings) after being charcaterized. All amendment compositions led to an increase of the pH of the leachate from approximately 3 to circum-neutral values. Humidity cells experiments indicated that the materials were adequate to immobilize Pb, Zn, Cu and Fe by about 99% and prevent their release from the tailings. The tests have also shown that the metals concentrations within the leachate are lower than the limits allowed by Algerian legislation for industrial liquid effluents. Among the tested material amendments, PLW was more effective than other products in immobilizing metals. The result are most probably related to their Ca-carbonate content and/or adsorption capacity.

Keywords—Sidi Kamber mine, Acid Mine Drainage (AMD), Phosphate material, Mining tailings, humidity cell leaching test, Neutralization.

NOMENCLATURE

SK Sidi Kamber.
SKF Flotation Sidi Kamber tailings.
SKG Gravimetry Sidi Kamber tailings.
PLW Phosphatic Limestone Wastes.
RLGP Raw Low Grade Phosphate ore.
PMT Phosphate Mine Tailings.
AMD Acid Mine Drainage.

I. INTRODUCTION

The mining industry is one of the most important and fundamental industries for the Algerian economy. It generates substantial inflows of foreign currency and contributes to the national development. However, it generates large amounts of liquid and solid wastes, which affect the environment, in particular water, soil, landscape, fauna and flora.

The oxidation of sulphide minerals within solid mine wastes can occur under natural conditions, according to the succession of climatic seasons. During acid-neutralization reaction, acidic aqueous effluent enriched with high metal concentrations and sulphates can be generated [1] [2] [3] [4]. In the presence of oxygen and water, the sulphide can be oxidized according to the following reaction (Eq.(1)):

\[ \text{FeS}_2 + 7/2\text{O}_2 + \text{H}_2\text{O} \rightarrow \text{Fe}^{3+} + 2\text{SO}_4^{2-} + 2\text{H}^+ \] (1)

This reaction depends on the oxygen concentration, the water saturation index of the porous media and the temperature. Also, the sulphide oxidation can be induced by aqueous ferric iron ions into sulphates and the conversion of Fe\(^{3+}\) ions into Fe\(^{2+}\) ions [6].

Moreover, the ferric iron hydrolys can increase the acidity of the effluent and metal leaching. Any change of physicochemical conditions (during climate seasons) can lead to dissolution, and precipitation/co-precipitation, which can affect the overall drainage water quality.

The presence of some bacteria, such as Thiobacillus, are known to be strong catalyzers at acidic pHs of oxidation reactions, which consequently increase their kinetics [5]. These bacteria oxidize the surface of the mineral and favour the production of sulphates and the conversion of Fe\(^{3+}\) ions into Fe\(^{2+}\) ions [6].

The secondary minerals formed can again release metals into the natural environment at varying concentrations [7] [8]. In order to reduce the effect of contaminants on the environment, the contaminated effluent must be treated [6] [9].

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The design of contaminant effluent treatment plant and the choice of treatment method are based on several parameters such as the chemical characteristics of AMD, climatic conditions, type of residues, hydraulic retention time, etc. [10]. Acid production can be reduced to very low levels by eliminating one of the three components of sulphide oxidation reaction (i.e., mining waste, water, oxygen; [9] [11]). Recent improvements in AMD waters treatment techniques have allowed lowering the level of contamination to acceptable environmental limits regarding many elements [12] [13].

Recently, several AMD treatment techniques have been developed to neutralize the acidity of AMD and reduce hazardous and toxic effluents. AMD treatment methods are divided into two main categories: a) Active methods: which involve the continuous addition of an alkaline compound to AMD to neutralize acidic effluents produced at mine sites and to precipitate metal contaminants [14] [15] [16] [17]; b) Passive methods: which use economical and environmentally friendly processes to achieve the same effects with a long-term, effective treatment, and offer the possibility of re-using materials at low cost [18] [19].

The alkaline amendment is one of the AMD control methods that involves mixing the tailings with alkaline materials to limit the oxidation of sulphides and neutralize the acidity generated [20] [21]. The purpose of this study is to evaluate the effectiveness of an amendment consisting of phosphatic materials (phosphatic limestone wastes, Raw low grade phosphate ore and phosphate mine tailings) for the control of the DMA phenomenon. The effectiveness of these materials for the remediation of contaminated water and soil has been widely studied [22] [23] [24] [25]. The main originality of this research compared to previous studies on AMD treatment using phosphate amendments relies on the nature of the tailings (high concentration of heavy metals) and the variety of P products that have been used. Indeed, the used materials, i.e.,These later include phosphatic limestone waste, raw low-grade phosphate ore and phosphate mine tailings that are locally available at low cost and efficient for AMD control and treatment.

II. MATERIAL AND METHODS

A. Sidi Kamber mine site description and sampling

The abandoned Sidi Kamber mine is located south of the Collo massif and about 36 km west of the town of Skikda (Fig. 1). This mining site fall within the Oued Es-Souk sub-watershed (12.49 km²), which supplies the Guenitra dam [26] [27]. Sidi Kamber region is characterized by a Mediterranean climate (wet and cool winter and a dry and arid summer) with average annual precipitation of around 742.35mm, average annual temperatures of 19 °C and average monthly humidity of around 68, 8% [28]. The mined ore deposit consisted of eight quartz veins mainly hosted in gneiss and granulite formations belonging to the metamorphic Precambrian Kabylid bedrock [30] [37]. The mineralization is composed of sulphide minerals (pyrite, pyrrhotite, marcasite, sphalerite and galena) and gangue minerals such as magnetite, hematite, barite and aluminosilicates [27] [30] [38]. The Sidi Kamber fine and coarse tailings stockpiled in dyke/piles are highly acid generating, with NNP s varying from -31.13 and -152.38 kg/ CaCO₃/t [30]. This mine is a major source of contamination of surface water and groundwater [27] [29] [30] [31]. Oxidized minerals dissolved over several years generate acidic effluents that disturb the environment. Chemical analyses of weathering products in mine wastes (tailings and waste rock) and soil contaminated by AMD, showed serious contaminations with some metals (Zn, Fe, Pb, Mn, Hg, and Cd) [27].

Sidi Kamber samples were collected using an auger and were then air-dried for 2 weeks. These tailings were crushed and sieved through a 1mm.The sieved samples were stored for analysis.

B. Phosphate amendments

The phosphate deposits are concentrated in the Djebel Onk mining district situated in the northeastern part of Algeria (Tebsessa department). The studied sedimentary succession is about 500 m thickness and aged of Upper Cretaceous (Maastrichtian) to Middle Eocene (Lutetian). The Thanetian phosphate layer is about 30 to 35 m thick [39] [40] [41]. Three types of materials from the Djebel Onk phosphate deposits, raw low-grade phosphate ore (RLGP), phosphatic limestone wastes (PLW), and phosphate mine tailings (PMT) were used to amend the Sidi Kamber tailings. The samples were dried, crushed, sieved and stored in plastic bags before testing.

C. Physical, chemical and mineralogical characterization

The samples were sieved using a series of sieves with standardized openings (ISO R-20 series sieves) and by a laser analyser (Malvern Mastersizer S) to measure the particle size distribution of particles from 0.06 to 80 μm.

For solid samples, metals concentrations were analyzed by fluorescence using Niton XL3t 900SHE instrument. This device can measure concentrations ranging from ppm to high levels but less accurate than ICP-AES spectroscopy.

The mineralogy of the samples was determined using an optical microscope and a Bruker AXS Advance D8 diffractometer (in 0 / 2θ mode for diffraction angles between 5 ° to 70 °). Eva and TOPAS software are used for the identification and quantification of mineral phases.

D. Kinetic tests in weathering cells (method developed by Crua et al., 2001, modified by Villeneuve, 2004)

The geochemical behavior of samples was evaluated in weathering cell (WC) kinetic test [43]. The WC simulates an accelerated and natural oxidation of mine discharges [44]. These tests have been recommended due to the small quantity of sample, the rapidity and stability of the geochemical behaviour during the leaching cycles of wetting/drying [45] [46]. Two WCs were set up, for SKG and SKF tailings. Approximately 67 g of tailings were placed in a 10 cm diameter Buchner funnel, equipped with a 0.45 μm filter (Fig. 2). A 250 mL vacuum flask was installed under every Büchner to collect the filtrate after flushing. Twice a week, the samples were allowed to contact 50 mL deionized water for 3h. Between two flushes, WCs were exposed to ambient air. Leachates from each WC were analyzed for pH, Eh, electrical conductivity and metal (Fe, Ca, Mn, Ni and Zn) [42] [43] [47].

E. Alkaline amendment tests

Alkaline amendment is the technique which prevent and control the formation of acid mine drainage [32]. It consists of an alkaline addition (limestone, dolomite, phosphate) directly in contact with the tailings, in order to limit the oxidation of sulfides and maintain the pH of the pore water in the tailings close to neutrality [20] [33].
Three alkaline materials (carbonate phosphate waste rock, crude phosphates and tailings) were used to control the formation of DMA and improve water quality. To this end, kinetic tests in humidity cells were carried out according to the modified ASTM standard (American Society for Testing and Materials D5744-07). The experimental cells (10.2 cm high and 20.3 cm in diameter), five in number, contained about 1 kg of mine tailings SK (SKG + SKF), mixed with phosphate materials. Leaching involves circulating approximately 500 ml of deionized water through the cell once a week for six months (Table I).

The cycle begins with a rinse with deionized water which remains in contact with the sample for a period of 4 hours, followed by 3 days of dry air circulation and 3 days of humidified air circulation between 90% and 100%. The airflow is maintained at 1 to 10 l/min (Fig 3).

### F. Analysis of leachate quality

A Starter ST310 electrode was used for the pH measurements, coupled with a Jenway 3510 pH meter (precision ± pH 0.01). Buffer solutions (pH4, pH 7 and pH10) were used for the calibration of the pH meter. The redox potential is measured using an electrode and an Eutech Instruments Ion2700 device. The obtained values were corrected to the standard hydrogen electrode (SHE; 200 mV). The Conductivity was measured using an OHAUS ST 20 C-A conductivity meter. The device was calibrated with a standard solution (200 µS/cm).

The concentrations of chemical elements were measured by atomic absorption spectrometry of the Contraa 800 (Analytik Jena) type.

The samples were first filtered at 0.45 µm, then acidified with 2% nitric acid (HNO₃) and refrigerated until analysis.

### III. RESULTS AND DISCUSSION

#### A. Physical, chemical and mineralogical characterization

### Sidi Kamber tailings

The results of the sieve analysis were presented in Table II. The calculated values of the coefficients of uniformity and curvature (Table II), were estimated at 2.5 (2≤Cu≤5) and 0.98 (Cc=1) for the flotation tailings, the material is poorly graduated with a tight particle size. More than 80% of the particles had a diameter between 63µm and 160µm. The gravimetric separation tailings curve is continuous; the material is well graded (1<Cc<3) and the particle size was semi-spread (5<Cc≤20).

The mineralogical composition of Sidi Kamber tailings determined by XRD was summarized in Table II.

The samples SKF and SKG were generally composed of the same mineralogical phases but with different proportions: quartz (20 – 61.9%), muscovite (32.4 – 8.2%), barite (4.7 – 5.4%), pyrite (3.05 – 4.3%), and galena (1.34 – 0.96%). The secondary minerals identified in the SKF sample include jarosite (14.2%) and gypsum (2.2%). Barium hydroxides (12.2%) and gypsum (7%) were also present in SKG tailings.

Chemical compositions of Sidi Kamber samples were presented in Table II. The analyses showed similar concentrations of SiO₂ (21.65-23.54%), MgO (0.63-0.79), Al₂O₃ (7.22-7.98%), K₂O (2.38-3.45%), CaO (0.67-0.876%) and a higher variation in Fe₂O₃ (3.12-6.96%) (Table 2). Total sulphur had very high levels ranging from 3.18% to 5.36%, this is explained by the presence of sulphide minerals.

Significant concentrations of Zn (1052-1423 mg/kg) and lead (847-954) were measured, corresponding to the sphalerite and galena minerals that are abundant in SKF and SKG tailings.

### Phosphate amendments

Phosphate materials (PLW, RLGP and PMT) showed more than 50% of the elements by weight > 80 µm, so there is the presence of gravel, sand and silt while tailings are mostly fine. The mineralogical characterization of the raw phosphates was determined by X-ray diffraction spectrometry, which mainly identified gangue minerals. Phosphate waste rock was mainly composed of carbonate minerals (dolomite and calcite), quartz, and ilmenite, whereas the minerals present in the raw phosphates include dolomite, calcite and fluorapatite (Table II). The tailings had similar mineralogical compositions as raw phosphates [31].

The chemical composition of phosphatic limestone wastes is characterised by very high (32.5% CaO and 2.2% MgO) contents. Phosphate ore (RLGP) and phosphate mine tailings (PMT) had high levels of CaO (45.2%), P₂O₅ (28.8%) and SiO₂ (4.0%). Cobalt, Zinc, Barium and lead are also present at low concentrations [31].

#### B. Kinetic tests in mini weathering cells

Kinetic tests in weathering cells were carried out over a period of 175 days to assess the environmental risks of metal contamination (Fe, Cd, Zn and Pb). Figure 4 shows the evolution of pH, Eh, conductivity and metal concentrations in the leachates obtained from SKF, and SKG samples. The geochemical behaviour of the two samples is similar. During the first cycles of the experiments, the pH of the leachates was about 3.43 and 3.02 for the SKF and SKG tailings respectively. Then, the pH increased to stabilize at 4.25 after 20 weeks for SKG sample. At the end of the test, the pH decreased to strongly acidic values (pH less than 3.9) for SKF. These values indicate strong bacterial activity that oxidises iron ([Acidithiobacillus Ferrooxidans], *Leptospirillum Ferrooxidans*) [48] [49]. It should be noted that a pH below 6.5 does not meet the Algerian environmental criterion for the final discharge of industrial liquid effluents.

The redox potential values ranged from 450 mV to 540.3 mV for SKF and from 420 mV to 540 mV for SKG, indicating that the environment is oxidizing. The conductivity showed a decreasing trend for the two samples (1365 µS/cm to 260 µS/cm for SKF and 1100 to 255 µS/cm for the SKG).
Fig. 1: Geographical sites location of the Djebel Onk phosphate mine and Sidi-Kamber polymetallic mine

Fig. 2 Schematic diagram of the weathering cell test
In fact, Mayer [50] explained this decrease in Eh values by a pre-oxidation of the sulphide grains which can lead to a change in their reactivity.

**Figure 4** shows high initial concentrations that may be related to the dissolution of ultrafine particles [51] [52] [53] and to the dissolution of highly reactive mineral surfaces [51]. Weakly adsorbed elements were then quickly released into solution. Lead concentrations in leachates ranged from 0.48 to 0.78 mg/L for SKF and from 0.39 to 1.1 mg/L for SKG. The assembly of minerals (galena PbS, anglesite PbSO$_4$ and cerussite PbCO$_3$) is the most cause of the release of lead. The measured Zn concentrations showed high values during the first over cycles (SKF, 6.5 mg/L), (SKG, 2.8 mg/L). This result can be explained by the oxidation and high reactivity of the ZnS sphalerite. At the end of the tests, Zn concentrations were about 0.38 mg/L for SKF and SKG. Cadmium concentrations in the leachates decreased from 0.7 to 0.43 mg/L for both Sidi Kamber samples. **Figure 4** showed that low Fe concentrations were leached from residues (0.36 mg/L for SKF and 0.32 mg/L for SKG). The iron had probably precipitated into the cells.

The initial leachates had low pH (from 3.02 to 4.9) and high metal concentrations such as Cd (from 0.7 to 0.8 mg/L), Zn (from 3 to 7 mg/L) and Pb (from 0.6 to 1.2 mg/L). The measured concentrations do not meet the Algerian environmental standards: 1.5 mg/L for Cu, 5 mg/L for Fe, 3 mg/L for Zn, and 0.2 mg/L for Pb and Cd (Executive Decree No. 06–141). It should be noted that in terms of AMD prediction, weathering cell tests do not realistically reflect natural conditions compared to humidity cells or real field, since AMD takes a long period of time to achieve equilibrium and it depends on several specific factors (grain size distribution, mineralogical and chemical compositions, and climatology) [1] [54].

### C. Alkaline amendment

**Figure 5** showed the water quality of the leachates (variations in pH, conductivity, redox potential and metal concentrations) at different time during the humidity cell tests for the 5 phosphate amendments tested.

The pH value in the first leaching cycle for the five humidity cells exceeded 7.5. In cells C1 (SK+PLW); C3 (SK+PMT) and C4 (SK+PLW+PMT), the pH increased considerably to reach maximum values of 8.25, 8.27 and 7.91 respectively for cell C1, C4, and C3. Cells C2 and C5 contained respectively (SK+RLGP) and (SK+PLW+RLGP) had a neutral pH (7.25 to 7.56) at the last leaching cycle (**Fig. 5a**). This increase in pH is due to the contact between the tailings and each of the materials tested and was a result of the high buffering effect of the amendments consisting mainly of carbonate and apatite minerals.

Redox potential values during the humidity cells tests are shown in **Fig. 5b**. As with the pH variation, the graphs showing the variation of the redox potential as a function of time had the same allure. The Redox potential of leachates containing each of the materials decreased significantly, reaching similar values of 329 mV and 326 mV respectively for C3 and C4 cells and lower values of 317 mV for C1 cell contained PLW. For leachate from cells C2 and C3, the redox potential varied from 360 to 395 mV throughout the test period. The results indicated a chemical reaction between SK tailings and phosphate alkaline materials, explained by an acid neutralization.

The conductivity values measured in the cells are shown in **Fig. 5c**. The conductivity is proportional to the concentration of ions in solution. Ions resulting from carbonate dissolution and SO$_4^{2-}$ ions contribute to conductivity variations. Conductivity decreased during leaching cycles in 5 cells from 6500 to 3300 µS/cm.

In order to assess the effectiveness of phosphate treatment, metal concentrations were measured and used as a criterion for performance [34] [35]. The evolution of metal concentrations in solution during the amendment tests is shown in **Figure 5**. The variation in the concentrations of iron in leachate showed that it was easily eliminated by the phosphate materials with final concentrations of 0.005 mg/L for cell C1, 0.012 mg/L for C2, 0.009 mg/L for C3, 0.0085 mg/L for C4 and 0.01 mg/L for C5 (**Fig. 5d**).
Table. II Physical, chemical and mineralogical characterization of Sidi Kamber mine tailing

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Fig.4 Water quality evolution during kinetic weathering cell test
Fig. 5: Water quality evolution of the amended tailings in the humidity cells tests.
Low iron values were explained by precipitation of iron oxyhydroxides and/or iron phosphate. These results are consistent with those of Hakkou et al [33] using phosphate waste rock to control AMD from Kettara tailings (Morocco). The addition of 15 wt% alkaline phosphate wastes to these tailings produced leachates with lower acidities (7<pH<7.5) and metal concentrations than unamended controls. **Figure. 5e** shows changes in Zn concentrations in leachate for 25 flushing cycles. Zine showed a gradual decrease in concentration over time. The final concentrations of this metal in cells remained between 0.081 mg/L (C1) and 0.33 mg/L (C5). Zinc probably precipitated into all the cells. Zn immobilization is due to the ion exchange and complexation mechanisms; it results in the formation of Zn-containing phosphates [54]. The variation curves for lead concentration in all leachates indicated a descending trend from an initial concentration of (0.80 to 0.012 mg/L for C1), (0.71 to 0.013 mg/L for C2), (0.80 to 0.01 mg/L for C3), (0.82 to 0.01 mg/L for C4) and (0.85 to 0.015 mg/L for C5) (Fig. 5f). Xu et al [55] reported that lead can be immobilized in contaminated soils by phosphate rock (PR) amendment, but its efficiency is generally limited by low solubility of PR. Indeed, phosphate solubilizing bacteria (PSB) (*Pantoea ananatis* and *Bacillus thuringiensis*) can promote Pb immobilization through PR by producing citric, glucose, and a-Ketoglutaric acids. The variation of copper in leachates indicated that it had been easily eliminated by all alkaline amendments with final concentrations not exceeding 0.01 mg/L. These findings are in agreement with previous studies reported by Mignardi et al [56] where natural phosphate rock (PR) were used to heavy metal polluted soils from sulfide mine areas in Sardinia and Tuscany (Italy). The application of phosphate amendments to the polluted mine waste soils reduced water-soluble concentrations of metals by about 99%. Mechanisms involved in the immobilization of metals by phosphates are: 1) ion exchange, 2) surface complexation, 3) precipitation of new metal phosphates, and 4) substitution of Ca in phosphate rocks by metals during recrystallization [22] [31] [36].

The results showed that phosphate compounds (PLW, RLGP and PMT) are effective amendments to immobilize metals (Pb, Zn, Cu, Fe,) in Sidi Kamber tailings. The Phosphate stabilization of metal-contaminated soils and wastes can be achieved in 3 phases: (1) dissolution of soluble, metal-bearing phases that need to be stabilized, (2) dissolution of a phosphate source, and (3) precipitation of insoluble metal phosphates. Previous studies suggested that acid buffering and metal retention in phosphate-amended materials can be the results of phosphate precipitation, Fe hydroxide formation, or carbonate dissolution and resultant acid neutralization [35] [57].

**IV. CONCLUSION**

The main objective of this study were to reduce the risk of environment contamination caused by AMD and metal leaching potential emanating from Sidi Kamber mine tailings. Weathering cell tests showed that higher concentrations of trace elements such as Cd, Cu, Pb, Zn and Fe were leached from these tailings. Humidity cell kinetic testing evaluated the effect of alkaline changes based on phosphate products and by-products from the Djebel Onk mine (phosphate limestone wastes, low phosphate crude ore, phosphate mine tailings) on the neutralization of the AMD. The results of the electrochemical and geochemical analysis of the leachates showed that all materials (used alone or in a mixture) are capable of immobilizing the metal elements (Pb, Zn, Cu, Fe) through the combination of highering the pHs and others processes such as adsorption/or co-precipitation. The insitu tests are recommended to evaluate the performance of this technique and its feasibility.

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