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Kinetic tests of mine waste neutralization by coal fly ash, Jerada coal district, Morocco

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ABSTRACT

Acid mine drainage (AMD) originating from mine waste (SM) is a major environmental problem of many metal mines in the world, both for operational and abandoned mines. The abandoned anthracite mine of JERADA, which is the subject of this study, is located in northeast Morocco. In the past, this mine has produced about 20 million tonnes of solid mine waste. The aim of this work is to study the feasibility of chemical neutralization of AMD using the alkaline industry residuetermed Coal Fly Ash (CFA) as a cover form. The Coal Fly Ash available on site was characterized by chemical and mineralogical analyses, which showed that it contains about 9.43wt % of calcite, known for its acid neutralization capability. The neutralization capacity of the CFA was then assessed by kinetic (column) tests and leachate analyses, which demonstrated the alkaline character of CFA, and confirmed a significant reduction of generated acidity that nearly reached a neutral pH, but also showed low potentials of oxidation-reduction, and a low content of SO_4, Fe, and other soluble elements.

1. Introduction

The biggest environmental concern in the mining field is associated with Acid Mine Drainage (AMD), caused by the alteration of sulfide minerals present in mine waste and tailings, which upon oxidation generate sulfuric acid. Thisincrease in acidity liberates various toxic heavy metals initially contained in the mining waste [1], affecting both operating and abandoned mines [2]. The rate of acid production depends on several factors such as the mineralogy of the ore and waste, the involvement of bacterial neutralization, the speciation of the sulfur-bearing compounds generating the acidity, the conditions of water infiltration, and the availability of oxygen [3]. Many solutions are employed to minimize and remediate the problems associated with AMD. The most efficient environmental methods to alleviate the AMD problem involve internal neutralization, the recovery of aqueous and/or organics compounds, and processes of biological degradation [4]. Various factors can influence how successful these methods of environmental remediation may be. These include the chemical characteristics of AMD, the quantity of leachate requiring treatment, the local climate conditions, the topography of the area in question, the characteristics of the materials used, and the expected lifetime of the mining project.

The encasement of mine tailing seems to reduce AMD in the spring either by eliminating the supply of oxygen and/or the infiltration of water into the mine tailings. However, the most promising technical solutions are quite expensive. These technologies aim to reuse the industrial tailings as a cover material, using the fact they have an alkaline pH and, therefore the power of neutralizing acidity by eliminating the diffusion of oxygen in the underlying acidogenic field. Thus, the addition of neutralizing elements is the most commonly used method [5]. The main advantages of this process are: (1) the recovery and reuse of an industrial tailings for rehabilitation of the area with low cost procurement of encasement material; (2) the reuse of fly ash to prevent and remediate the AMD [6-8].

(3) The treatment of groundwater at abandoned mines, which was contaminated by AMD by injection of alkaline materials [9]; (4) and the mixing of fly ash with red mud [10]. In Morocco, power plants produce ca. 467,000 tonnes of fly ash per year. In the eastern part of the country, the Jerada 3.55 SM thermal power plant has been in operation since 1970, producing about 147,000 tonnes of fly ash per year. This material is discharged simultaneously with clinkers by hydraulic systems to an ash basin located 2 kilometers away from the power plant [11]. In consists principally of the products of combustion and incineration of solid wastes [12]. The physical, chemical, and mineralogical characterization of these ashes is important prior to their reuse. The potential application of fly ash in waste

water treatment is justified by its chemical and physical components, as alkaline fly ash constitutes a good neutralizing agent [13]. Coal Fly Ash (CFA) is characterized by fine spherical particles of silt size, ranging from 0.002 to 0.075 mm [12] and its composition is related to incombustible materials present in the coal such as silicon, aluminum, iron, calcium, and magnesium. It has the ability to react with calcium hydroxide to form hydrated calcium silicates, which have pozzolanic and hydraulic properties. Two main groups of fly ash can be distinguished: siliceous and calcic (which are respectively high in SiO_2 and CaO). The potential applications of CFA in the control and neutralization of the AMD lixiviates [14], in the stabilization and attenuation of metallic element mobilization [15-16] and in a control of the behavior of the storage areas during a prolonged time [12] constitutes an excellent way to reduce the impact on the environment.

In this respect, our study had as principal objective the verification in laboratory of the technical and environmental feasibility of chemical neutralization of AMD derived from mine waste (SM) at Jerada by applying the alkaline CFA. The purpose of the study was:

- The chemical and mineralogical characterization of mine tailings and fly ash;
- Kinetic tests monitoring physico-chemical parameters of SM and CFA +SM; and
- The assessment of the power neutralization of the CFA and the feasibility of the technology assessed in this study.

The anthracite mine of Jerada is located in the northeast of Morocco (Figure 1), in a region characterized by a semi-arid climate, hot in summers and cold in winters, with an average monthly temperature of 33°C and 3°C respectively, and a mean annual rainfall not exceeding 200 mm [17].The coal deposits are hosted by Westphalian series rocks and consist of eight separate coal layers. A detailed geological description of this region is given by several authors such as [18-19]. The random mine waste is uncontrolled and located into the city.

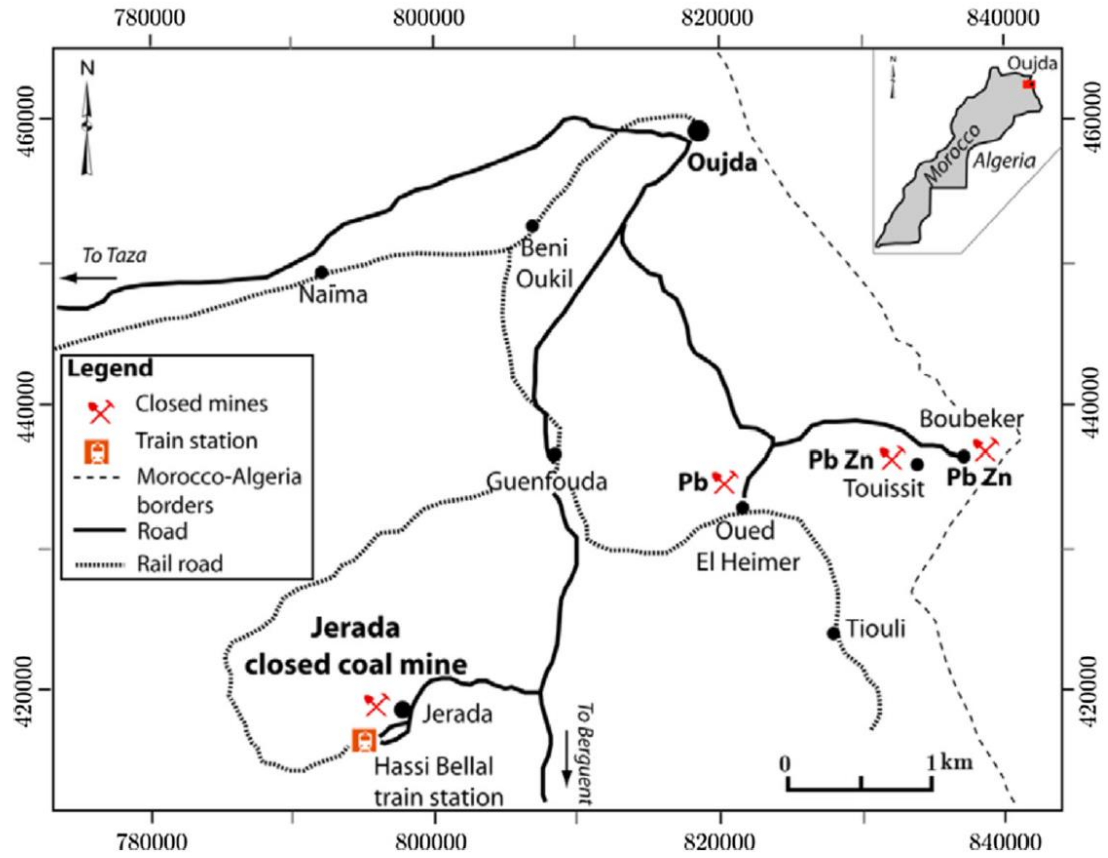

Figure1: Location of the study area - Jerada anthracite mine [17]

2. Materials and methods

2.1 Characterization of solids

The mine waste (SM) used in this study comes from a waste rock dump located in the mine of Jerada (Figure 1). It is characterized by a high state of oxidation and coarse grain size. The samples were stored without drying at low temperature for the subsequent kinetic tests. For each analysis, a quartering was performed according to the AFNOR standard[20], in order to obtain a representative sample of the overall sample. The CFA are produced at the Jerada thermal power plant, by collection of fine gray particles during the dusting of gas coming from the combustion of pulverized coal.

XRD analyses were performed to evaluate the initial mineralogy of the mining wastes. The total sulfur (S_{total}) and carbon (C_{total}) contents were determined by combustion in an induction furnace (ELTRA CS-2000) with O_2 at 1370°C, H_2O 1% trapping of bubblers, and dosing by ionic chromatography exchange. The Acid Potential (AP) and the Neutralization Potential (NP) were determined according to the modified protocol of the Sobek test as described by [21]. The dosing of neutralization minerals was carried out by acid-base titration. The

determination of Net Neutralization Potential (NNP) was obtained by calculating the difference between NP and AP values. These values were used to constrain the potential of the material for acidity generation [22]. The NP/AP ratio was also used as a way to determine the acid generation potential of the samples [23].

2.2 Kinetic tests (column tests)

Leachate column tests of lixiviates allow to assess the effect of alkaline fly ash on the acid drainage waters and evaluate the performance of this method to control the AMD. The columns are made of PVC (Polyvinyl Chloride), have 14 centimeters in diameter and a length of 1 meter, and are open at the top to the atmosphere. The bottom of the columns is made of a perforated Plexiglas plate and a geotextile filter to hold the fine particles. The tests lasted 20 months and were conducted in conditions simulating the climate in the field. Subsequently, the columns were subjected to wetting/drainage cycles by the passage of 2 liters of distilled water in the top of columns, and leachate fluid samples were collected at the bottom of the columns in polyethylene bottles.

The fluid samples were then filtered through cellulose filters (0.45 µm) using a vacuum filtration pump. The determination of Fe, Ca^{2+}, and $Mg^{2+}$was performed by atomic emission spectroscopy (ICP-AES) after acidification with 1% (v/v) of nitric acid (HNO_3). Temperature (Tp), pH, electric conductivity (EC), and oxidation-reduction (Eh) were measured by LUTRONTM (WTW530) and LUTRON TM (WTW 197) respectively. The SO_4^{2-}content was determined by spectrophotometry, according to the AFNOR, T90-009 standard.

Two columns were used according to the following experimental set (Figure 2):

- Column 1: witness column containing mine waste (MS) of acidic character,
- Column 2: 40-centimeters-thick cover layer of CFA was placed on top of the mine waste.

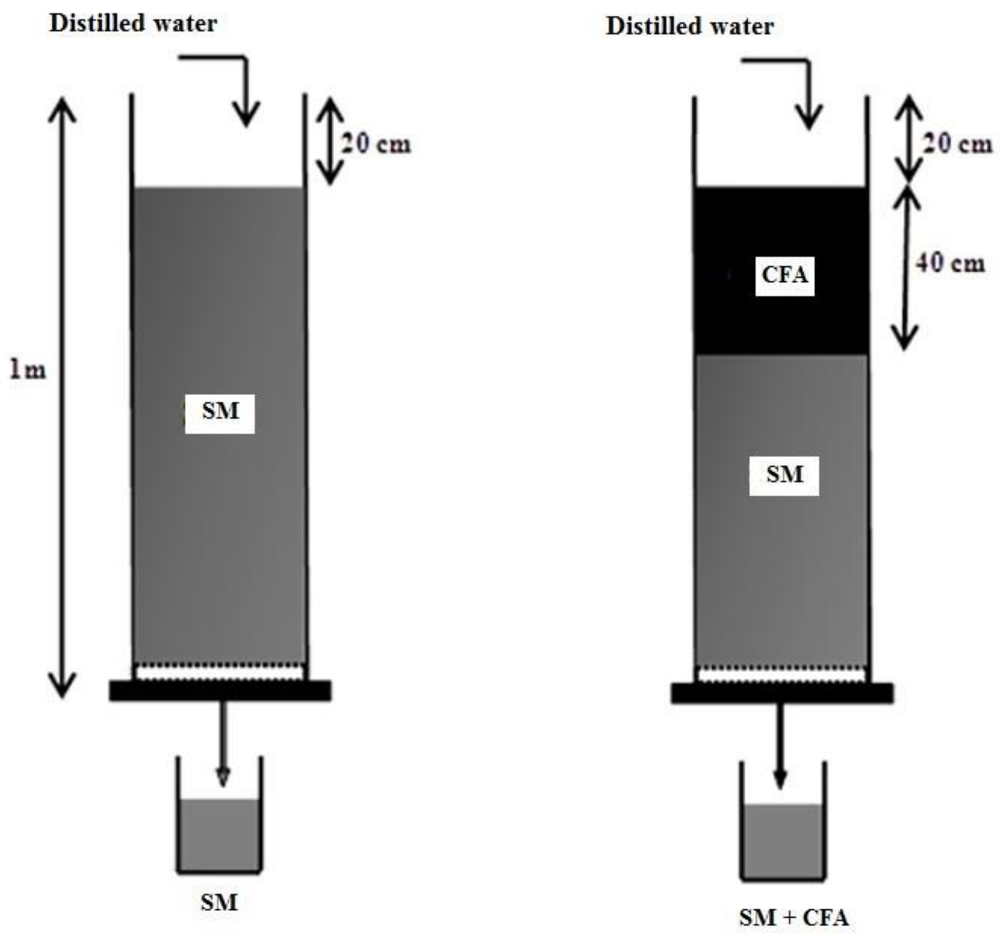

Figure 2: Experimental setup of kinetic tests. The SM is mine waste and the CFA is coal fly ash.

3. Results and discussion

3.1 Chemical and mineralogical composition of solids

Mine waste (SM) and coal fly ashes (CFA) are low in Ca and Mg, but CFA has up to of 3.1%Ca and 1.37% Mg respectively (Table 1). They are related to carbonate minerals and can also enter in the composition of the silicates.

Table 1: Chemical composition of minewaste (SM) and Coal Fly Ash (CFA) from the Jerada mine district, as anlyzed by ICP-AES.

%	**SM**	**CFA**
Ca	0,81	3,10
Mg	0,44	1,37
Fe	4,86	0,52
Al	10,0	10,5
Ni	0,004	0,027
Pb	0,050	0,024
Zn	0,005	0,018

The Fe content, which is higher in the SM, comes from the presence of iron sulphides (pyrite FeS_2). The Al content is important and much more significant in the SM and CFA with 10%and 10.5% respectively. The latter value reflects the presence of silicates, more particularly aluminosilicates in the samples. In contrast, the Ni, Pb, and Zn contents are negligible.

Table 2 shows the mineralogical phases identified by XRD of SM and FCA samples. The SM is mainly composed of aluminosilicates and secondary minerals. The results also show the large proportion of quartz, followed by muscovite and chlorite. Gypsum, anhydrite, and goethite are probably of secondary origin and likely indicate an early alteration of the mine waste. However, the detection limit of the XRD method (1%) a possible presence of pyrite was suspected in the mine waste, and was subsequently confirmed by optical microscope (Figure 3). The mineralogy of CFA consists of quartz, aluminosilicates (muscovite), chlorite, pyrophyllite, calcite, and gypsum.

Table 2: Approximate mineralogical composition of Mine Sterile (SM) and Coal Fly Ash from the Jerada mine district, obtained using XRD analysis

Wt %	**Chemical formula**	**SM**	**CFA**
Quartz	SiO_2	30.16	25.13
Muscovite	$KAl_2(Si_3Al)\ O_{10}\ (OH,F)_2$	49.93	34.75
Chlorite	$(Mg,Fe)_3(Si,Al)_4O_{10}(OH)_2$	14.15	16.11
Gypsum	$CaSO_4.2H_2O$	2.73	1.09
Pyrophyllite	$Al_2Si_4O_{10}(OH)_2$	13.89	13.49
Goethite	FeO OH	2.82	-
Titanite	Ca Ti SiO_5	2.91	-
Anhydrite	$CaSO_4$	0.89	-
Calcite	$CaCO_3$	-	9.43

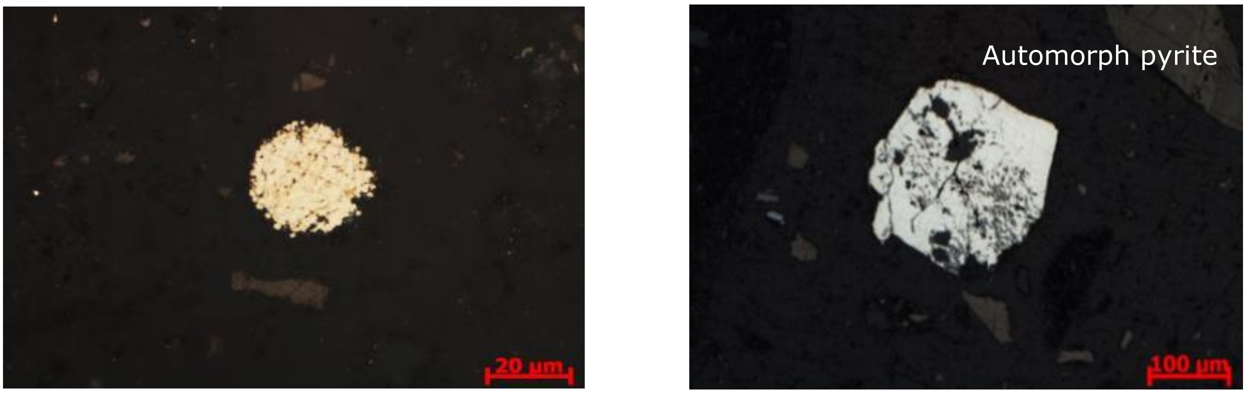

Figure 3: Examples of microscopic pyrite grains present in the samples

3.2 Static test: Acidity generation potential

Table 3 shows the results of static tests using the limits of [21]. The results ofSMare located in the incertitude interval of the test (-20 to 20 kg$CaCO_3$/t) with an NNP value of -14.1 kg$CaCO_3$/t. The NNP is used to highlight the propensity of the sample for acidity generation. The FCA does not generate anyacidity with 64.9 kg$CaCO_3$/t of NNP. According to [22], an NP/AP ratio lowers than 1 for SM and CFAsuggests they are potential generators of acidity. According to this ratio, which for the SM is equal to 0.03, the mine waste is a potential generator of acidity (power neutralization is insufficient to neutralize all the acid produced by

the sulfides), and the high value of CFA (6.59) demonstratesits strong ability to neutralize acidity.

Table 3: Sulphur (%) and Carbon (%) content in Mine waste (SM) and Coal Fly Ash from the Jerada mine district, obtained using XRD (AP: Acid Potential, NP: Neutralization Potential, and NNP: Net Neutralization Potential in kg $CaCO_3$/t)

Samples	**S_{total}**	**C_{total}**	**$S_{sulfate}$**	**$S_{sulfure}$**	**AP**	**$NP_{modified}$**	**NNP**	**NP/AP**
SM	0,98	34,44	0,51	0,47	14,6	0,5	-14,1	0,03
CFA	0,46	12,10	0,10	0,36	11,6	76,5	64,9	6,59

3.3 Kinetic tests: lixiviates obtained from the columns with SM and SM+CFA

3.3.1 pH, Eh, and EC

The leachates of the columns solely filled with mine waste (SM) yielded pH values of the order of 5 and Eh values between 142 and 25 mV (Figure 4). This indicates favorable oxidation conditions and significant acidity generation. In contrast, the tests for columns filled withSM+ CFA indicate increasing pH values of the order of 7, and decreasing Eh values of the order of 12 mV.

During the first few weeks, the variation of pH was not significant nor consistent, as the systems had not yet stabilized. From the third rinse (90 days) onwards, the leachates exhibited pH values of 7 to 7.5 (neutral to slightly basic), and a significant decrease of the Eh from 67.2 to 12.3 mV. The increasing acid-neutralization was related to the presence of the carbonates contained in the CFA (calcite).

The EC decrease during the two tests (SM and SM + CFA) is shown in Figure 4 and indicates a decrease in the concentration of soluble ions. This could be due to the stabilization of the sterile or the depletion of the ions. In fact, the stabilization of the sterile is due to an increase in the pH values, above a pH of 6, with a solubility reduction effect of the ionic compounds.

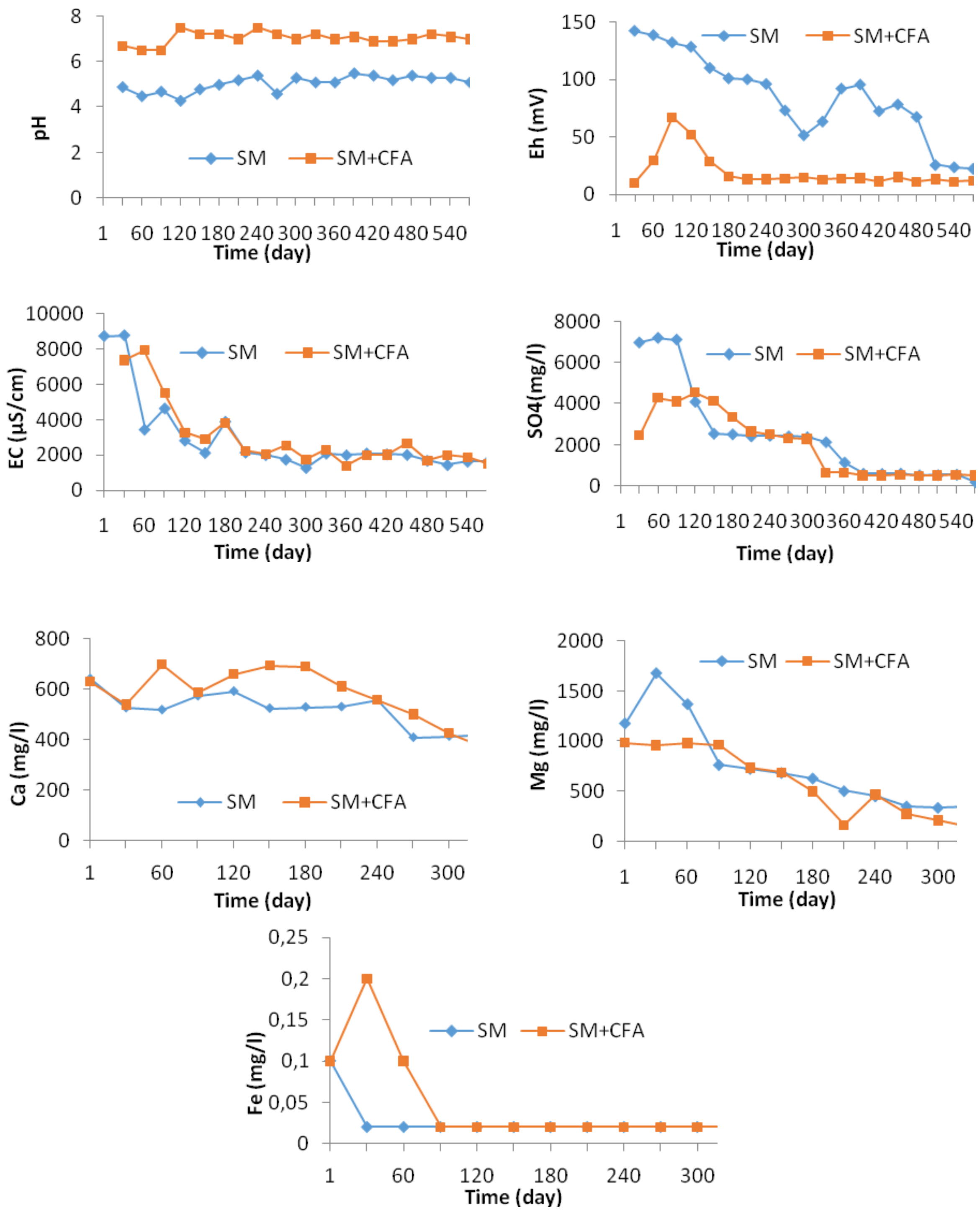

Figure 4: Kinetic tests of pH, Eh, EC, SO_4, Mg, Ca, and Fe concentration versus time in the leachates coming from the mine sterile (SM) and mining waste with coal fly ash (CFA).

3.3.2 SO_4^{2-} and Fe concentrations

Taking into account the equilibrium conditions imposed by the physico-chemical parameters in the environment. The concentrations of SO_4 observed in the leachates are the result of a complex series of reactions, beginning with the oxidation of sulfide minerals, followed by

neutralization, and subsequent precipitation. The decrease in the concentration of SO_4 during the tests (Figure 4) reflects a decrease in the oxidation rates. For the SM and SM+CFA, the respective SO_4concentration values started from 6900 up to 520 mg/l and from 4300 up to 520 mg/l respectively, with an inhibition effect due to alkaline additives (CFA). The acid available for dissolving calcite ($CaCO_3$, which a spring of Ca^{2+} to precipitation of gypsum ($CaSO_4.2H_2O$), and consequently the limitation of concentration of SO_4. The low concentration of SO_4 observed at the end of the tests indicates a deceleration or even the ceasing of sulfide oxidation reactions. The iron concentrations were very low throughout the experiment, and remained below the detection limits. The precipitation of these minerals would be favored by the passivation of the surface of the iron sulfides or by the high pH of the interstitial water.

3.3.3 Ca and Mg concentrations

The main products of the sulfide oxidation process are Fe and SO_4, and the other elements released reflect the interaction between the solids (minerals) and liquid (acidic water) phases. In the case of mine waste (SM), the concentration of Ca^{2+}decreased with the progress of the test from 600 to 421 mg/l (Figure 4). The quantities of Ca^{2+} released were most probably due to the dissolution of gypsum ($CaSO_4.2H_2O$), anhydrite ($CaSO_4$), and titanite ($CaTiSiO_5$). For the neutralization test (SM + CFA), the concentration of Ca^{2+} decreased from 697 to 370 mg/land was due to the dissolution of calcite ($CaCO_3$) in the oxidation reactions of sulfides and the ensuing acidification.

The concentration of Mg^{2+} followed the same pattern for the two tests, decreasing from 1680 to 342 mg/l for the SM and from 983 to 147 mg/l for theSM+CFA, respectively. In the latter case, we can observe very low concentrations as opposed to the first test. The higher pH and alkalinity would promote adsorption of precipitation of Mg. On the other hand, this concentration indicates the dissolution of the mineral content in the mine waste consisting essentially of aluminosilicates associated with sulfides.

3.4 Alkalinity and acidity

Figure 5 shows the evolution of acidity and alkalinity in the two tests. For the first test (SM) the acidity fluctuated over the time and ranged between 430 and 720 mg $CaCO_3$/Kg, and the alkalinity increase after the 6th experiment of up to 280 mg $CaCO_3$/Kg could be related to the presence of chlorite and muscovite known for their neutralization power. In the second test (SM+CFA), the results were unlikein the first test (low acidity and high alkalinity), with a decrease in the acidity from 680 to 460 mg$CaCO_3$/Kg after the 4th experiment and a very

small variation over time. On the other hand, the alkalinity for the same test shows a variation in time between 480 to 1200 mg $CaCO_3$/Kg.

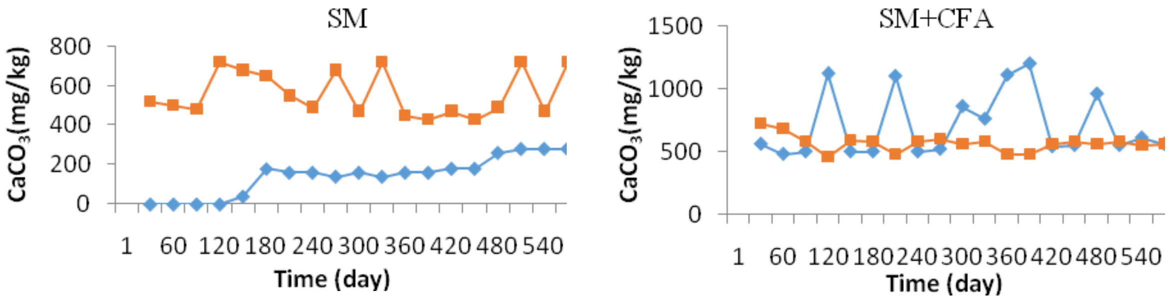

Figure 5: Kinetic tests of acidity (brown line) and alkalinity (blue line) in the leachates of mine sterile (SM) and mine sterile with coal fly ash (CFA).

3.5 Efficiency of alkaline residues for AMD control

The kinetic tests (leaching tests) demonstrated the effectiveness of the addition of alkaline residues ($CaCO_3$) as a cover to oxide mine sterile. This procedure achieved a neutral pH, and the decrease in the solubility of the ions was due to the increase in pH, which decreased the concentration of different elements in the leachate. The calcite ($CaCO_3$) tended to hydrolyze in the presence of water, thereby releasing hydroxides that neutralized the acidity of the aqueous medium. The dissolution of carbonates (Calcite) led to the reduction in acidity by consumption of H^+ ions. In the presence of protons, the calcite kept dissolving and neutralizing the acidity according to following equation, when pH was inferior to 5.8 (equation 1):

(1) $CaCO_3 + 2\ H^+ \rightarrow H_2CO^{3-} + Ca^{2+}$ (pH < 5.8)

When the pH value exceeded 6.3, the reaction was (equation 2):

(2) $CaCO_3 + H^+ \rightarrow HCO^{3-} + Ca^{2+}$ (pH > 6.3)

We could observe in this medium the chemical interaction between sulfuric acid and calcite following equation (3), resulting in the formation of gypsum ($CaSO_4H_2O$). So, the sulfuric acid could also contribute to the formation of melanterite ($FeSO_4.7H_2O$), as well as other sulfates [24].

(3) $Ca(OH)_2 + 2\ H_2SO_4 \rightarrow CaSO_4.2H_2O$

The dissolution of carbonates (release of Ca, Mg, Fe, and Mn) and hydroxides was generally fast compared to the transport rate. The cation release participating in the formation of secondary minerals such as hydroxides, subsequently contributed to neutralization of a very low pH. In fact, the neutralization of the acidity and the precipitation reactions reduced the

oxidation rate through the formation of inhibitory mineral layers. In contrast, the oxidation reactions took predominant place in unsaturated conditions, but the dissolution of carbonates and the production of alkalinity occurred in saturation and unsaturation conditions. Finally, the complete process of oxidation, hydrolysis, and neutralization of pyrite [2]can be described by the following equations (4 and 5):

(4) $FeS_2(s)+4CaCO_3(s)+3.75O_2(g)+3.5H_2O \rightarrow Fe(OH)_3(s)+2SO_4^{2-}+4Ca^{2+}+ 4HCO^{3-}$

For pH > 6.3

(5) $FeS_2(s)+2CaCO_3(s)+3.75O_2(g)+1.5H_2O \rightarrow Fe(OH)_3(s)+2SO_4^{2-}+2Ca^{2+}+2CO_2(g)$

For pH < 5

4. Conclusion

The use of industrial materials as a cover to reduce acid mine drainage (AMD) is very practical for both environmental, technical, and economic reasons. Various materials can be used as a cover depending on their availability and the physico-chemical conditions of the target area. The use of alkaline residues as cover on the oxide mine residues gives a good result both in the short and long term. In fact, these residues serve as a barrier for oxygen by neutralizing the acidogenic medium.

The results of this study assessed the geochemical conditions which make the evaluation of efficiency methods. This is an effective alternative and economic way to control the AMD. Thus, the results of the kinetic tests indicate that the CFA has an alkaline power capacity in the management of mine waste by reduction of the production of the AMD. The monitoring of the physico-chemical parameters resulting from the kinetic tests did not reveal any sign of oxidation. Neutral pH values, sulfates, and metal concentration contents in the leachate confirmed that the CFA reduces greatly the production of AMD and that of the soluble elements. In summary, the results suggest that a CFA composed mainly of calcite ($CaCO_3$) has promising physico-chemical properties for AMD mitigation, and its effectiveness should now be tested on a larger scale.

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