Kinetic Tests to Evaluate the Relative Oxidation Rates of Various Sulfides and Sulfosalts

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ABSTRACT

Mine wastes produced from sulfide-bearing ores exploitation and processing are often characterized by acid mine drainage (AMD) generation, which leads to high dissolved metal concentrations in addition to acidity. When the acidic drainage is neutralized (generally through neutralization by carbonates), the phenomenon is called contaminated neutral drainage (CND). The quality of these drainages can be predicted by static tests, which can quickly assess the acid-generating potential (AP) and the neutralization potential (NP) by chemical and/or mineralogical calculations. With the Paktunc static test, the mineralogical composition of the sample is used to calculate both AP and NP. For AP calculation, equal oxidation rates are assumed for all sulfides. Since the sulfide minerals oxidize at different rates, the objective of the present paper is to compare the oxidation rates of various sulfides and sulfosalts often encountered in mine wastes in order to take them into account in AP prediction. Thus, seventeen pure sulfide minerals and sulfosalts (pyrite, pyrrhotite, bornite, chalcocite, chalcopyrite, covellite, galena, sphalerite, arsenopyrite, stibnite, gersdorffite and fahlore) were characterized physically, chemically and mineralogically, and submitted to kinetic tests (modified weathering cells) in order to compare their oxidation rates, acidity generation, and metal leaching potentials (As, Cu, Fe, Ni, Sb or Zn). The oxidation rates vary from 2 to 15 times between the sulfide minerals studied. Moreover, the metal concentrations in leachates from the sulfosalts are 30 to 40 times higher than those of the sulfide minerals.

This study fills a knowledge gap in the oxidation rates of common sulfides and sulfosalts, which enable a more precise prediction of AMD and CND generation, mainly through the integration of a kinetic parameter in the modified Paktunc AP calculation.

INTRODUCTION

Solid mine wastes resulting from extraction and ore treatment are often stored at the surface in waste rock piles and tailings impoundments. Mine wastes from sulfide-bearing ores contain different sulfide minerals which, upon exposure to atmospheric conditions, may generate acidic effluents in the absence of neutralizing minerals. The phenomenon, well-known as Acid Mine Drainage (AMD), is a complex process of chemical, physical and biological reactions. The prediction of water quality is of significant importance for the mining industry, due to increasingly restrictive regulations and higher social awareness.

AMD is very well documented in the literature (Brodie, Broughton & Robertson, 1991; Alpers & Nordstrom, 1999; Aubertin, Bussière & Bernier, 2002; Blowes et al., 2014). A reliable prediction of the quality of mine drainage at the earliest stage of the mining projects is beneficial. Different water quality prediction tools such as static and kinetic testing were developed in the past 30 years (Sobek et al., 1978; Adam et al., 1997; Lawrence & Scheske, 1997; Paktunc, 1999; Blowes et al., 2014). Prediction static procedures often examine the balance between the acid generating potential (AP) and neutralization potential (NP) of samples and are called acid–base accounting (ABA) tests (Ferguson & Morin, 1991). There are distinct classes of static tests: chemical methods and mineralogical methods. Chemical methods require testing in a chemistry lab, while mineralogical methods are mainly based on the mineralogical composition of the mine wastes. The AP and NP are calculated as the sum of the individual contributions of each acidifying and neutralizing mineral, based on their proportions in the sample and their relative reactivity (only for NP). The NP procedure has been largely investigated in the literature (e.g. Lapakko, 1994; Lawrence & Wang, 1997; Li, 1997; Paktunc, 1999). On the other hand, the AP is still often simply calculated on the basis of the bulk sulfide concentration in the sample, considering all sulfides are present as pyrite and based on the stoichiometry of pyrite oxidation (EQUATION 1). Thus, this simple AP calculation is biased because the possible different acid generation (on a molar basis) of other sulfides is not taken into account. To overcome this issue, (Paktunc, 1999) proposed an AP calculation based on the theoretical oxidation rates of the different sulfides (EQUATION 2).

$$AP = 31.25 \times \%Ssulfide \qquad (kg\ CaCO_3/t) \qquad (1)$$

$$AP = \sum_{s=1}^{m} \left(\frac{n_s \times 98 \times X_s \times 10}{w_s} \right) \qquad (kg\ H_2SO_4/t) \qquad (2)$$

- AP: acidification potential;
- ns: number of moles of H2SO4 formed by the oxidation of one mole of sulfide mineral s;
- 98: molecular weight of H2SO4 (g.mol–1);
- 10: conversion factor for recasting in kg.t–1;
- Xs: amount of sulfide mineral s in wt%;
- ws: molecular weight of sulfide mineral s (g.mol–1);
- m: number of sulfide minerals in the sample.

This equation improves the AP calculation but does not take into consideration the relative acidity production rates of the different sulfides; the latter was evaluated by several other authors (Nicholson, 1994; Rimstidt, Chermak & Gagen, 1994; Jambor & Blowes, 1998; Plumlee, Logsdon & Filipek, 1999). Moreover, based on the Paktunc equation, Bouzahzah, Benzaazoua & Bussière (2013) proposed another modified equation taking account the relative rates of acidity production of each sulfide by using a relative reactivity factor. This factor is determined by the average of the total

acidity produced by each sulfide calculated relatively to the acidity production of pyrite. However, only five common sulfides have been studied (arsenopyrite, chalcopyrite, galena, pyrite and sphalerite). Thus, based on their work, seventeen sulfide minerals were submitted to kinetic testing. The main objective of this study is to compare the acidity production rate of various sulfide minerals and sulfosalts often encountered in mine wastes, including pyrite (FeS_2), As and Ni-bearing pyrite, and pyrrhotite ($Fe_{(1-x)}S$, $0<x<0,17$) (hereinafter referred to as iron-sulfides); bornite (Cu_5FeS_4), chalcocite (Cu_2S), chalcopyrite ($CuFeS_2$), covellite (CuS), galena (PbS), sphalerite (($Zn,Fe)S$), and sphalerite-Fe (hereinafter referred to as base-metals sulfides); arsenopyrite ($FeAsS$), fahlore (tetrahedrite-tennantite solid solution) ($(Cu,Fe,Ag,Zn)_{12}(Sb,As)_4S_{13}$), gersdorffite ($NiAsS$), and stibnite ($Sb_2S_3$) (hereinafter referred to as As/Sb-bearing sulfides). To do this, a series of standard sample were prepared by mixing pure sulfide minerals with quartz (considered as inert). The reactivity of each compound within standard samples is evaluated by laboratory kinetic tests in modified weathering cells (small-scale humidity cell tests; see Cruz et al., 2001 and Benzaazoua et al., 2004 for details).

METHODOLOGY

Materials

A total of 17 sulfides and sulfosalts were selected based on their presences in typical polymetallic sulfides-rich deposits. These sulfides were acquired as pure mineral samples from a specialized minerals supplier. Five of these seventeen sulfides (arsenopyrite, chalcopyrite, galena, pyrite and sphalerite) were previously studied at the RIME (Bouzahzah, Benzaazoua & Bussière, 2013; Bouzahzah et al., 2008). The other samples were hand sorted to remove macroscopic impurities under a binocular lens before grinding to reach typical tailings grain size distribution (Bussière, 2007) according to their own grindability. Representative samples of the pure mineral powders were submitted to a full characterization and then used into the kinetic test.

Polished sections of the powders were examined and showed that some materials contain minor sulfide and/or gangue contaminants. Pure quartz was mixed with the pure sulfides and sulfosalts (95 wt% quartz and 5 wt% pure minerals) for kinetic testing.

Solid material characterization

The physical, chemical and mineralogical properties were determined for all studied materials (pure and mixtures). The grain size distributions were measured with a Microtrac S3500 laser grain size analyzer. The D_{10}, D_{50} and D_{90} values taken from the grain size distributions are presented below. The grain size distributions of the studied pure minerals are typical of tailings with D_{10} values ranging approximately from 1 to 20 μm and 80 to 95 % passing 130 μm. The total sulfur content S_t, including S from sulfide and sulfate minerals, was analyzed by an ELTRA induction furnace coupled with an infrared analyzer. The bulk chemical composition was analyzed by ICP-AES (Perkin Elmer Optima 3100-RL) following a multi-acid digestion ($HNO_3/Br_2/HF/HCl$). TABLE 1 summarizes the physical and chemical properties of the pure minerals.

Table 1 Physical and chemical properties of the pure studied minerals as solid samples

Mineral	D$_{10}$ (µm)	D$_{50}$ (µm)	D$_{90}$ (µm)	St (%)	Fe (%)	Cu (%)	Zn (%)	Pb (%)	As (ppm)	Ni (ppm)	Sb (ppm)	Other traces (<2000ppm)
Iron-sulfides												
Pyrite 1	8.7	49.0	129.2	50.1	46.46	0.04	0.01					
Pyrite 2	5.8	29.8	75.9	45.62	45.29	0.14	2.48	0.65	1110	458	<4	Bi, Cd, Co, Mn
Pyrite 3	6.2	35.9	98.3	34.89	32.75	1.72	0.1	0.15	3280	498	<4	Bi, Cd, Co, Mn
Pyrrhotite 1	4.6	21.1	74.3	36.63	64.29	0.24	0.11	0.04	1050	71	<4	Bi, Co, Mn
Pyrrhotite 2	11.7	54.5	128.8	37.67	58.2	0.22	0.03	0	917	47410	981	Bi, Co, Mn
Base-metals sulfides												
Bornite	19.9	75.9	163.7	24.13	11.44	61.56	0.18	0.02	148	9	699	Bi, Co, Mn
Chalcocite	17.1	66.1	151.3	20.34	0.04	78.57	0.23	0.02	<30	10	90	Co, Mn
Chalcopyrite 1	6.2	33.3	94.8	32.5	34.49	21.1	1.55	0.01	752	69	<4	Bi, Co, Mn, Se
Chalcopyrite 2	10.5	44	161.0	32.77	29.74	30.2	0.02	0.02				
Covellite	14.4	72.2	161.7	32.39	0.02	64.18	0.21	0.01	<30	12	6	Se
Galena	6.1	41.3	115.1	12.64	<0.001	0	<0.0055	87.36	<30	6	527	
Sphalerite	4.6	43.2	160	32.19	6.9	0.01	63.1	0.02				Cd, Mn
Sphalerite-Fe	10.4	57.9	142.1	32.94	11.6	0.1	60.43	0.01	177	16	<4	Co, Mn (0.5%)
As/Sb-bearing sulfides												
Arsenopyrite	3.6	18	102.9	18.83	32.35	0.01	0	0.07	434000	94	399	Bi, Mn
Gersdorffite	4.2	18.2	49.2	11.56	0.8		0.12		385000	235500	14900	Mn, Co (3.5%), Se
Fahlore	4.5	21.9	68.7	24.81	4.71	40.63	2.03	0.01	105000	240	62000	Bi, Co, Mn
Stibnite	4.5	30.5	84.6	28.74	<0.001	0.02	<0.0055	<5	<30	<5	712000	Co, Se

Polished sections were observed by optical microscopy to identify the gangue minerals or/and other minor sulfides. The composition of gersdorffite and fahlore was first evaluated using a JEOL J7600F Field Emission Scanning Electron Microscope (FE-SEM) coupled with an Oxford EDS (Energy Dispersive X-ray Spectrometry) spectrometer. Then, the more precise composition of these two samples and trace elements in pyrite 2 and 3, pyrrhotite 1 and 2, and in sphalerite-Fe (choosed along the assay of the sample and/or the oxidation behavior) was determined by analyzing 10 particles from each section using a Castaing Cameca SX100 Electron Probe MicroAnalyser (EPMA) coupled with a WDS (Wavelength-dispersive X-ray Spectometry) spectrometer for the microanalysis. All quantitative EPMA were done in wavelength-dispersive mode with an

accelerating voltage of 20 kV and a constant beam current of 20 nA. The counting time on each peak was 10 s with the exception of Mn (40 s), Ag (40 s) and trace elements (Cd, Co:40 s). The results are shown in TABLE 2. Gersdorffite is a solid solution (Fanlo et al., 2004; Ahmed, Arai & Ikenne, 2009) which contains more As and less S and Ni than the gersdorffite approved by the IMA, and which also contains Co. Fahlore is a solid solution between the tetrahedrite and tennantite poles.

Table 2 Microanalysis by EPMA of the pure studied minerals as solid samples

Element (w%)	S	Fe	Cu	Zn	As	Ag	Sb	Ni	Cd	Mn	Co
DDL (ppm)	672	1067	1046	1222	664	959	1805	546	440	461	329
Fahlore	27.73	3.58	42.68	3.31	14.97	0.12	8.23				
Gersdorffite	11.8	0.63			56.65			27.24			5.21
Pyrite 2 (As)	53.27	47.12			0.1						
Pyrite 3 (Ni)	52.47	46.24						0.24			0.13
Pyrrhotite 1 (SEM)	40.38	60.83									
Pyrrhotite 2	39.19	57.44						1.18			
Sphalerite-Fe											
50% of type 1	33.57	7.29		58.98					0.08		
50% of type 2	33.65	8.71		56.54					0.16	1.18	

Modified kinetic testing

The samples were submitted to kinetic testing in modified weathering cells. These weathering cells (Cruz et al., 2001) are small-scale humidity cells which render similar results for the rates of reactions (Cruz et al., 2001). These tests could be considered as reproducible (Demers et al., 2011). Moreover, weathering cells can be used as an alternative to humidity cells when working on small sample quantities (< 100 g). This method is also known to be more aggressive than humidity cells (Villeneuve, 2004). The procedure consists of a 100 mm Büchner funnel with a 0.45 µm glass-fiber filter holding a 67 g sample. The sample is leached twice a week (3 days between flushes) with 50 mL deionized water. The cells were placed in a controlled-weather box to maintain the samples under optimal saturation conditions and avoid extreme drying (Bouzahzah, Benzaazoua & Bussière, 2013). Leachates from the weathering cells were analyzed for pH, conductivity, acidity, sulfur and elemental concentrations. The element concentrations in the leachates are analyzed by ICP-AES on an aliquot acidified to 2% HNO_3 for preservation. The weathering cells were run for 55 to 70 cycles (stopped upon reaching steady-state).

RESULTS AND DISCUSSION

pH

FIGURE 1 shows the comparison of the pH leachate of all studied minerals. The pyrrhotite 1 provided neutral pH at the beginning of the test which is related to the neutralization by calcite (6.8%). After its complete dissolution, the pH became acidic and then reached the pH value of the pyrrhotite 2. Almost all of the iron sulfide minerals showed more acidic pH at the beginning, certainly due to the dissolution of secondary products at the surface of the samples. The sphalerite

and sphalerite-Fe display very similar pH around 6. The covellite pH fluctuated between 5 and 6. The arsenopyrite generated the most acidic leachates of all samples. The gersdorffite produces acidic leachates at the test start and increases to more neutral pH along the test.
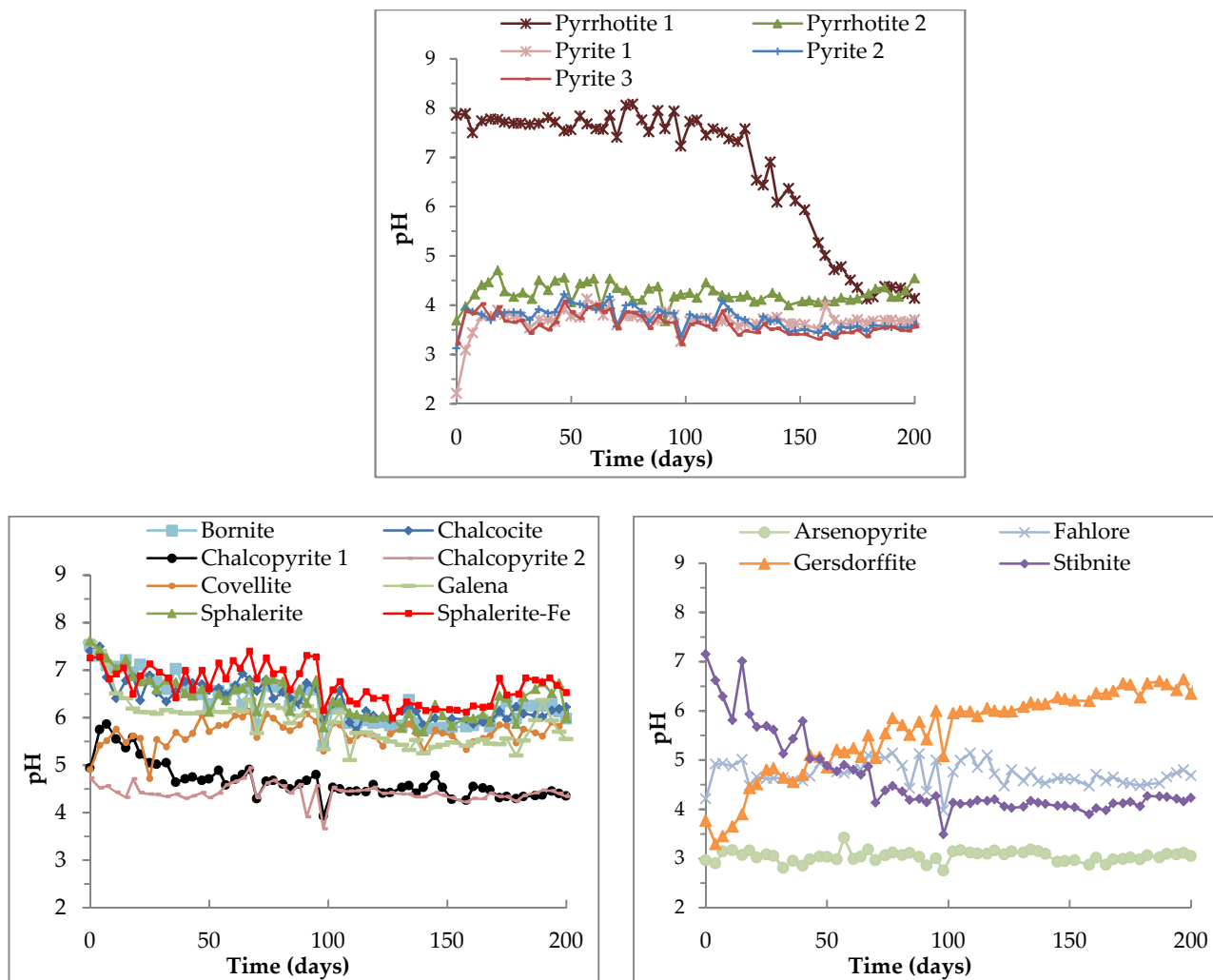

Figure 1 pH results of the leachates of the weathering cells for all minerals studied

Reactivity rates

As no other S-bearing oxidation products are suspected to form under the acidic and highly oxidative conditions of the kinetic tests, it is assumed that the entire S was present as sulfate in solution (pH > 2 and Eh > 0.4 V during all experiments) and ended up in the leachates. Therefore, this study uses the rate of sulfate production as the direct indicator of the acid generation rate and of the sulfide oxidation rate. The cumulative dissolved element loads are normalized regarding the initial sample mass for expressing the rates. The Specific Surface Area (SSA) of the studied minerals will be determined in a further study to take into account the influence of particle sizes in the oxidation rates.
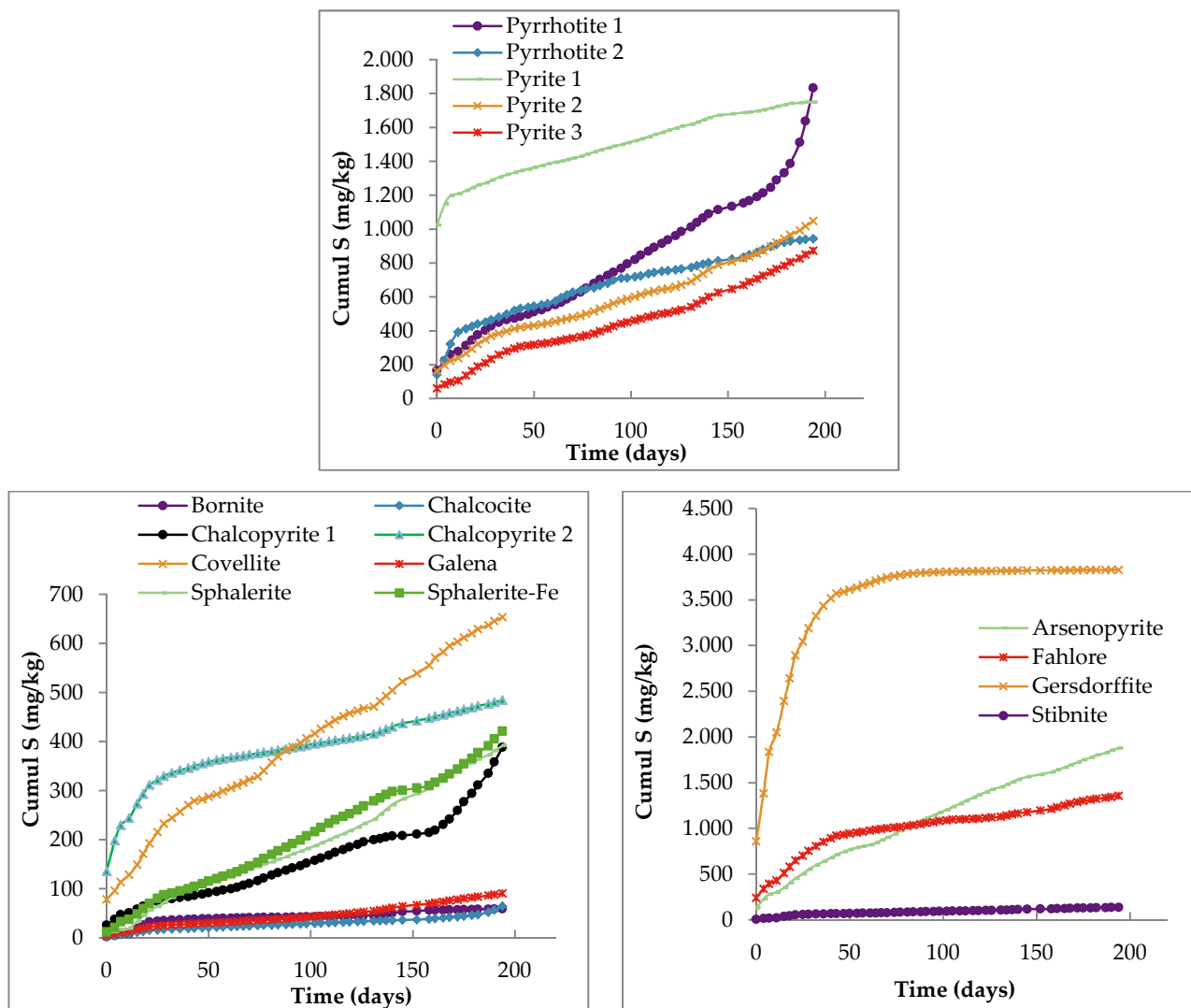
Figure 2 Cumulative normalized charges of S for the studied sulfides. Black arrows show points of inflexion for three specific minerals curves

FIGURE 2 shows the S release rate of the studied sulfides. The inflexion point of pyrrhotite 1 is due to the complete depletion of the carbonates content in the sample. The inflexion points of the sphalerites and chalcopyrites need further investigations and are not currently explained. We can note that the Mn content of the sphalerite-Fe may influence its rate of oxidation. This will be verified in a further study. TABLE 3 shows the oxidation rates of the studied sulfides, obtained by calculating the slope of the linear regression within the stabilized portion of these cumulative normalized loadings over time (mg of S/kg/day). These rates allowed the classification of iron-sulfides based on their reactivity as follows: pyrrhotite 1 > pyrite 2 > pyrite 3 > pyrrhotite 2 > pyrite 1. We have to mentionnthat the pyrrhotite 2 sample is not pure and contains 10 % of pentlandite. Moreover, this pyrrhotite contains trace Ni, certainly in its crystal network (about 1%, see TABLE 2), which may influence its reactivity (Dold, 2010). For this mineral, the Ni concentration in the leachates is higher (from 2 to 16 mg/L). The high concentration of Ni in pyrrhotite's leachates is

probably both due to the presence of pentlandite and trace Ni in the pyrrhotite grains. At the end of the test, the outline of almost all pyrrhotite grains is altered. For the base-metals sulfides, the classification is as follow: sphalerite-Fe > covellite > sphalerite > chalcopyrite 1 > chalcopyrite 2 > galena > chalcocite > bornite. Chalcopyrite 1 is more reactive than chalcopyrite 2 because it contains 26.5% of pyrrhotite. The sphalerite-Fe is more reactive than the sphalerite because of the presence of trace Mn (see TABLE 2) and its higher Fe-content. The As/Sb-bearing sulfides can be classified as: gersdorffite >>> arsenopyrite > fahlore > stibnite. We have to keep in mind that the gersdorffite is very reactive at the beginning of the test. Actually, the stabilized portion for gersdorffite either shows a depletion of the mineral of 70% (calculated by depletion of S) or could be due to a passivation of the reactive surfaces. Another duplicate test is currently ongoing.

Table 3 Experimental oxidation rates for the studied sulfides

Oxidation rates (mg of S/kg/d)					
Iron-sulfides		*Base-metals sulfides*		*As/Sb-bearing sulfides*	
Pyrrhotite 1	8.2	**Bornite**	0.23	**Arsenopyrite**	6.9
Pyrrhotite 2	2.6	**Chalcocite**	0.28	**Gersdorffite**	93.6
Pyrite 1	2.4	**Chalcopyrite 1**	2.0	**Fahlore**	3.2
Pyrite 2	4.8	**Chalcopyrite 2**	1.0	**Stibnite**	0.49
Pyrite 3	4.6	**Covellite**	2.7		
		Galena	0.53		
		Sphalerite	2.3		
		Sphalerite-Fe	3.2		

CONCLUSIONS AND FURTHER WORK

In order to complete the lack of knowledge about the oxidation rates of certain sulfide and sulfosalt minerals often encountered in mine wastes, the oxidation rates of seventeen of them were assessed by a modified weathering cell test. Based on the obtained results, these minerals can be classified in terms of oxidation based on their S release rates from the highest to the lowest: gersdorffite > pyrrhotite > arsenopyrite > pyrite > fahlore > covellite > sphalerite-Fe ≥ chalcopyrite > sphalerite > galena > stibnite > chalcocite > bornite. The results highlight the importance of trace element composition in the stability of individual sulfides (Dold, 2010). Pyrrhotite 2, which contains around 1.2% Ni, is less reactive than the pure pyrrhotite 1. Gersdorffite shows a different behavior which can be interpreted as a high initial oxidation rate and a high initial release of As. On the other hand, this study demonstrates that minerals could be very problematic for the environment, even if present at low concentrations in a sample. In fact, gersdorffite (As), arsenopyrite (As), stibnite (Sb) and fahlore (As, Sb) are very reactive minerals and release metal and metalloids. Further work will consist in the evaluation of their influence in real mine waste samples. Moreover, other metals or metalloids, like Cd, Co, Mo, Mn, Ni or Zn have been found in the leachates at concentrations over the existing regulation. More work will be done to improve the prediction of CND.

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