Assessment of the Performance of Environmental Desulphurization on Arsenic Release from Tailings

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Abstract

Contaminated (acid or neutral) mine drainage management is one of the most important environmental challenges related to tailings management for most polymetallic mine operators around the world. The contamination happens when sulphide minerals within tailings oxidize under atmospheric conditions. Contrary to acid mine drainage, contaminated neutral drainage (CND) occurs when neutralizing minerals counterbalance the acidity produced by sulphide oxidation. CND Effluents are characterized by circum-neutral pH and metals release (like arsenic). Tailings desulphurization is an attractive alternative technique for the control of both types of contaminated mine drainage. This paper presents the application of desulphurization to prevent CND from a gold mine tailings (Lapa mine, Qc, Canada) which contains arsenic (2080 g/t), mainly as arsenopyrite. Desulphurization tests conducted by ore flotation produced two successive concentrates (the first being mostly composed of talc and the second contained mainly sulphides) and a desulphurized final residue. To predict their arsenic release potential, the present work evaluates the environmental behavior of the initial ore (reference), the talc concentrate and the desulphurized residue using laboratory kinetic tests (weathering cells) and arsenic batch sorption tests.

Key Words: contaminated neutral drainage, desulphurization, arsenic, prediction, weathering cell, sorption

Introduction

Contaminated neutral drainage (CND) is characterized by circum-neutral pH effluents with concentrations of metals such as arsenic, nickel or zinc above regulations (Mayes et al. 2009; Plante et al. 2010; Warrender et al 2009). Many mine wastes containing arsenopyrite produce arsenic contaminated mine drainage as the mineral is often contained in tailings being barren or gold free. Arsenopyrite can be oxidized through the following reaction resulting in arsenic leaching as arsenate in circum-neutral pHs and oxidizing conditions (Haffertand Craw, 2008):

Arsenopyrite oxidation: $$4\ FeAsS + 13\ O_2 + 6\ H_2O \rightarrow 4\ Fe^{2+} + 4\ H_3AsO_4 + 4\ SO_4^{2-} \quad (1)$$

Prevention of CND or acid mine drainage (AMD) would require removing one of the components of the sulphide oxidation reaction shown above. Some techniques such as tailings covers intend to prevent oxygen and/or water infiltration to tailings. Environmental desulphurization aims at removing sulphides and producing a desulphurized tailings that does not generate contaminated mine drainage. In most cases, the sulphide concentrate, asa small volume, has a greater number of storage options such aspaste backfill (Benzaazoua et al., 2004). Desulphurization has been used on several mine tailings to prevent AMD (Leppinen et al. 1997, Benzaazoua et al. 2000, Yalcin et al. 2004) but, to the author's knowledge, its application to prevent CND has not yet been attempted.

Lapa processing plant (Agnico-Eagle Mine) produces gold using a standard circuit which consists of grinding and gravimetric separation followed by milling andcyanidation processes. The Lapa tailings are deposited in the main pond within the LaRonde mine site where cyanide destruction occurs through natural UV exposition followed by a Degussa process (H_2O_2, Soluble silicate) to remove remaining cyanide phases. Desulphurization by flotation was undertaken upstream of the cyanidation in two

flotation stages that produced a talc concentrate and then a sulphide concentrate. As some arsenic was entrained in the talc concentrate, arsenic release potential determination of this product is essential to decide whether to combine the talc concentrate with the desulphurized tailings or with the sulphide concentrate if the arsenic release is too important.
The aim of this paper is to evaluate and assess the performance of environmental desulphurization on arsenic release from the Lapa mine arsenic bearing wastes.

Materials and Methods

Materials

In order to work on fresh slurries, desulphurization flotation tests were conducted at the Lapa concentrator using a laboratory Denver D-12 cell. Pulp sampling was done before the cyanidation process twice a day to preserve the pulp physico-chemical properties. Feed solid percentage and chemical composition was monitored during the testing period. Testing was carried on a desulphurized tailings with an arsenic grade of 0.02 wt% and sulphur grade of 0.17 wt%. The two flotation stages produced two concentrates: the first concentrate mostly consisted of phyllosilicates (mainly talc) floated without collector and the second concentrate was composed of about 15 wt% sulphide floated with a xanthate collector at a pH=4.5, which provided an arsenic recovery of 86 %. The feed, the desulphurized tailings, the talc concentrate and a talc concentrate–tailings blend in balanced massic proportion (16 wt% talc concentrate and 84 wt% tailings) were tested to evaluate their geochemical properties. The blend of talc concentrate and tailings was tested in the prospect of combining the talc concentrate to the desulphurized tailings as an option for Lapa tailings management.

Physical, chemical and mineralogical characterization methods

Chemical composition of the Lapa tailings sample was evaluated through a complete digestion in HNO3/Br2/HF/HCl; the solution was then analysed using an inductively coupled plasma and atomic emission spectroscopy (ICP-AES, Perkin Elmer). The liquid samples (weathering cell leachates) were also analysed for chemical content by ICP-AES. Specific gravity (Gs) was determined with a helium pycnometer (Micromeretics, Accupyc 1330). Particle size distribution was determined using a Malvern Mastersizer laser particle size analyser. The specific surface area (SSA) was analysed by a Micromeritics surface area analyser using the B.E.T. method (Brunauer et al., 1938). Mineralogical characterization was done using a Bruker A.X.S. D8 advance x-ray diffraction (XRD) instrument equipped with a copper anticathode. Spectra were interpreted using EVA software for identification and the quantitative Rietveld method with TOPAS software (Rietveld, 1993). Mineralogical investigation was also achieved using micro-scale optical microscopy (OM) examinations of the solid samples using a metallographic microscope (Nikon Optiphot2-Pol) and a Scanning electron microscope (SEM) (Hitachi S-3500 N) using backscattered electrons (BSE) mode at 20 kV coupled to an X-ray energy dispersive spectrometer (EDS) (Oxford Instruments). Spectra were acquired with INCA software. SEM-EDS analyses were performed on polished sections coated with carbon.

Environmental characterization methods

Static and kinetic tests

Acid-base accounting was performed using the modified Sobek method (Lawrence and Wang, 1997) to evaluate the acid-generating potential (AGP). The acidity potential (AP) is calculated from the sample sulphur percentage (under the sulphide form) assuming that all sulphide are available for oxidation, and neutralization potential (NP) is determined using an acid-base titration.

Geochemical behaviour of the solid samples was investigated through small scale leaching cells referred as weathering cells (Cruz et al., 2001; Villeneuve, 2004). Weathering cells are used to accelerate tailings alteration in order to evaluate their reactivity. Cyclic flushes were performed on 67 g. of material with 50 mL deionized water twice a week, on days 3 and 7. The leachates were analyzed for pH, Eh, conductivity and chemical content by ICP-AES (after 0.45µm filtration and acidification with HNO_3). The weathering

cells were exposed to ambient air throughout the test period and kept in a humid state by regular deionized water spraying as weathering cell modified by Bouzahzah et al. 2010 (Fig.1). Thirty-five cycles were performed in this study to reach a geochemical steady state.

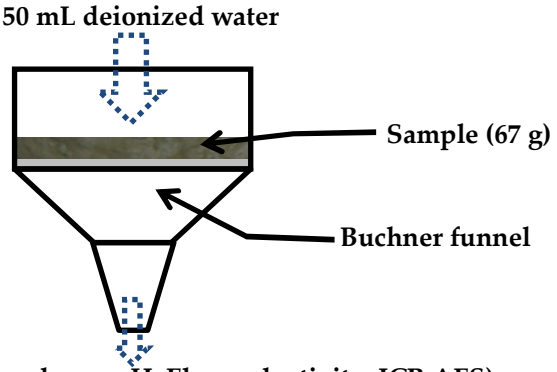

Figure 1: Weathering cells installation (adapted from Cruz et al. 2001).

Retention test

Arsenic retention tests were conducted on the feed, the desulphurized tailings and the talc concentrate. The arsenic retention test is a batch test that mixes a given tailing mass with different As(V) concentrations (1; 2; 5; 10; 20 mg/L) at backgroundpH. The feed and tailings samples were tested in triplicates for the 2 and 20 mg/l As concentrations.High repeatability was observed with standard deviation of the amount of arsenic retained by the solid phase below 10^{-3} mg/m^2. Blank tests with arsenic-free solution were conducted to evaluate the arsenic leached from each material. As(V) synthetic solution was prepared from sodium arseniteheptahydrate ($Na_2HAsO_4.7H_2O$) purchased from Sigma-Aldrich and a constant ionic strength background was established with 0.05 M of $NaNO_3$. All tests were carried out with 125 mL solution in an Erlenmeyer flask using a liquid/solid ratio of 25 mg/L. The Erlenmeyer flasks were shaken for 24 hours at room temperature (25.0 ± 1.0°C) and the content was then filtered using a 0.45 µm membrane filter. The liquid phase was analysed for pH, Eh and chemical content by ICP-AES (after acidification). Test parameters were chosen in accordance with the literature (Plante et al., 2010, Lim et al., 2009, Gu et al., 2010, Paikaray et al., 2011). The amount of arsenic retained by the solid phase was calculated by the following equation 2:

$$Qretention=(Ci-Ce^*)V/m \qquad (2)$$

Qretention is the retained amount of arsenic by the mineral surfaces (mg/kg). V is the solution volume (L) and m is the sample mass (g). Ci is the initial arsenic concentration (mg/L) and Ce* is the corrected equilibrium arsenic concentration calculated by the following equation 3:

$$Ce^*=Ce-Co \qquad (3)$$

Ce and Co are respectively the equilibrium arsenic concentration and the arsenic released by the material in the blank test (mg/L).

Interpretation of the retention phenomena can be made through the modeling of retention isotherms also referred as sorption isotherms. Langmuir and Freundlich modelswere used to evaluate if the sorption mechanism followed a homogeneous active site and a monolayer adsorption process (Langmuir) or heterogeneous active sites and a multilayer adsorption (Freundlich).

Results and Discussion

Physical, chemical and mineralogical characterization of samples

Table 1 presents the chemical and mineralogical characteristics of the products. The materials main constituents are quartz, plagioclase, dolomite and phyllosilicate. The phyllosilicates are mainly chlorite,

biotite, muscovite and talc. Talc proportions within the feed, talc concentrate and desulphurized tailings are 8, 34, and 1 %, respectively.

Table 1: Chemical and mineralogical characterization of the feed and flotation products.

	Feed	Talc concentrate	Desulphurized tailings	Blend talc concentrate-tailings*
Weight (%)	100	14.5	78.9	93.4
Major Elements				
As (wt%)	0.21	0.09	0.02	0.03
Fe (wt%)	5.25	5.53	4.54	4.70
S (wt%)	0.37	0.19	0.17	0.17
Mg (wt%)	4.58	10.9	3.48	4.67
Ca (wt%)	2.90	1.45	2.99	2.74
K (wt%)	1.72	0.87	1.88	1.72
Na (wt%)	1.51	0.75	1.56	1.43
Cu (g/t)	58	80	6	18
Sb (g/t)	240	220	110	130
Mineral quantification (XRD / Reitveld)				
Quartz (%)	26	9	32	28
Phyllosilicate (%)	34	70	25	32
Plagioclase (%)	27	16	32	29
Dolomite (%)	10	5	9	8
Actinolite (%)	2	/	1	1
Sulphide (%)	1	/	1	1
Mineral calculation (from chemical analysis**)				
Arsenopyrite (%)	0.46	0.20	0.04	0.07
Pyrrhotite (%)	0.63	0.33	0.37	0.36
Chalcopyrite (%)	0.02	0.02	0.00	0.01
Berthierite (%)	0.04	0.04	0.02	0.02

* Calculated from talc and tailings analyses
** As assigned to arsenopyrite, Cu assigned to chalcopyrite, Sb assigned to berthierite, remaining sulfur assigned to pyrrhotite

XRD analysis detected some pyrrhotite within the feed and the desulphurized tailings. However arsenopyrite was present in all samples as shown by OM analysis and SEM-EDS micro-analysis (Figure 2). Mineral characterization gave evidence that arsenopyrite was mainly present as liberated particles of about 5 to 10 µm in the talc concentrate, contrary to the desulphurized tailings which contained mainly locked arsenopyrite as illustrated by the figure 2. Berthierite($FeSb_2S_4$) was identified by SEM-EDS micro-analysis.

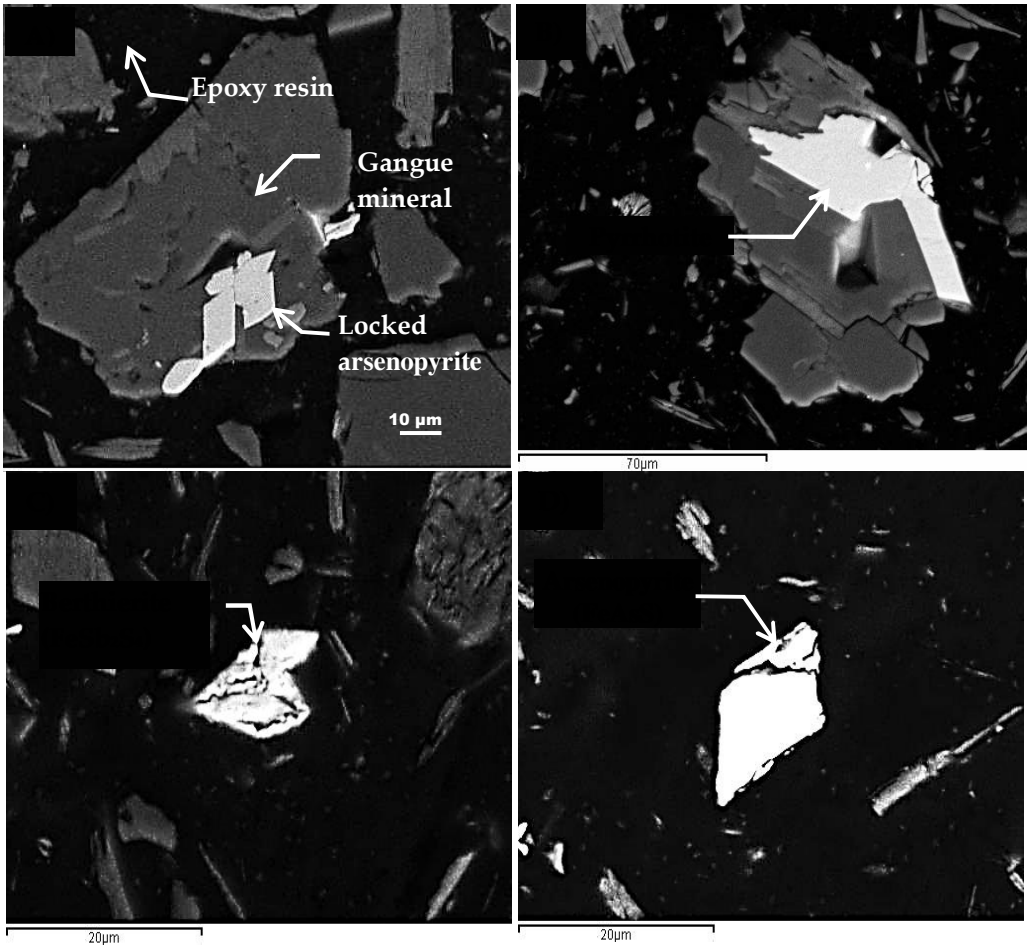

Figure 2: SEM images illustrating A) locked arsenopyrite and B) partly locked pyrrhotitewithin the desulphurized tailings, C) liberated fine berthierite andD) arsenopyrite within the talc concentrate.

Physical characterization and acid generating potential classification are summarized in Tables 2 and 3. The high specific surface area reflects the phyllosilicate content of the different material as the talc concentrate has the higher specific surface area. All materials have similar particle size distribution. The net neutralization potential (NNP=NP-AP) and the NP/AP ratio are tailings classification criterion used for acid generating potential interpretation (Aubertin et al., 2002; SRK, 1989, Adams et al., 1997). All samples have NNP above 20 kg $CaCO_3$/t and NP/AP ratio above 2 and are therefore classified as non-acid generating.

Table 2: Physical characteristics of the feed and the flotation products.

	Feed	Talc concentrate	Desulphurized tailings	Blend of talc concentrate and desulphurized tailings
Physical characteristics				
D_{10} (µm)	2.65	3.09	3.09	/
D_{50} (µm)	15.40	15.40	16.57	/
$Cu=D_{60}/D_{10}$	7.88	6.77	7.28	/
SSA (m^2/g)	2.4	3.5	2.4	/
Gs (g/cm^3)	2.8	2.9	2.8	/

Table 3: Acid generating potential of the feed and the flotation products.

	Feed	Talc concentrate	Desulphurized tailings	Blend of talc concentrate and desulphurized tailings
Environmental characteristics				
AP	11.6	5.9	5.3	5.3
NP	71.1	36.5	69.6	66.2
NNP	59.6	30.6	64.2	60.8
NP/AP	6.2	6.2	13.1	12.5

Kinetic tests

The weathering cell leachate chemistry is presented in Figure 3 showing pH, conductivity, arsenic and antimony release related to sulphide mineral oxidation. Figure 4 shows the evolution of the major ions Ca, Mg, Al and S release related to neutralizing minerals dissolution and acidifying mineral oxidation. Sulphur was assumed to be expressed as sulphate phases and iron is not presented here as its concentration was below the detection limit for all samples during the test (precipitation in situ of iron as ferrihydrite according to Visual Minteq simulations).The pH remained neutral at about 7.5-8 for all samples and the Eh, not presented in this paper, remained oxidizing at 400- 600 mV (SHE) allowing oxidation of the sulphides present in the samples. The conductivity quickly stabilized to approximately 150 µS/cm, typical of those encountered in neutral mine drainage (Plante 2011b).The feed and talc concentrate samples had similar leachates characteristics (conductivity, element release). Arsenic, antimony and sulphur release decreased steadily during the first 60 days to reach a dissolved concentration of 1 mg/L for arsenic and antimony and 2 mg/L for sulphur. Calcium and magnesium release also decreased for the first 25 days to reach a dissolved concentration of 15 and 3 mg/Lrespectively.

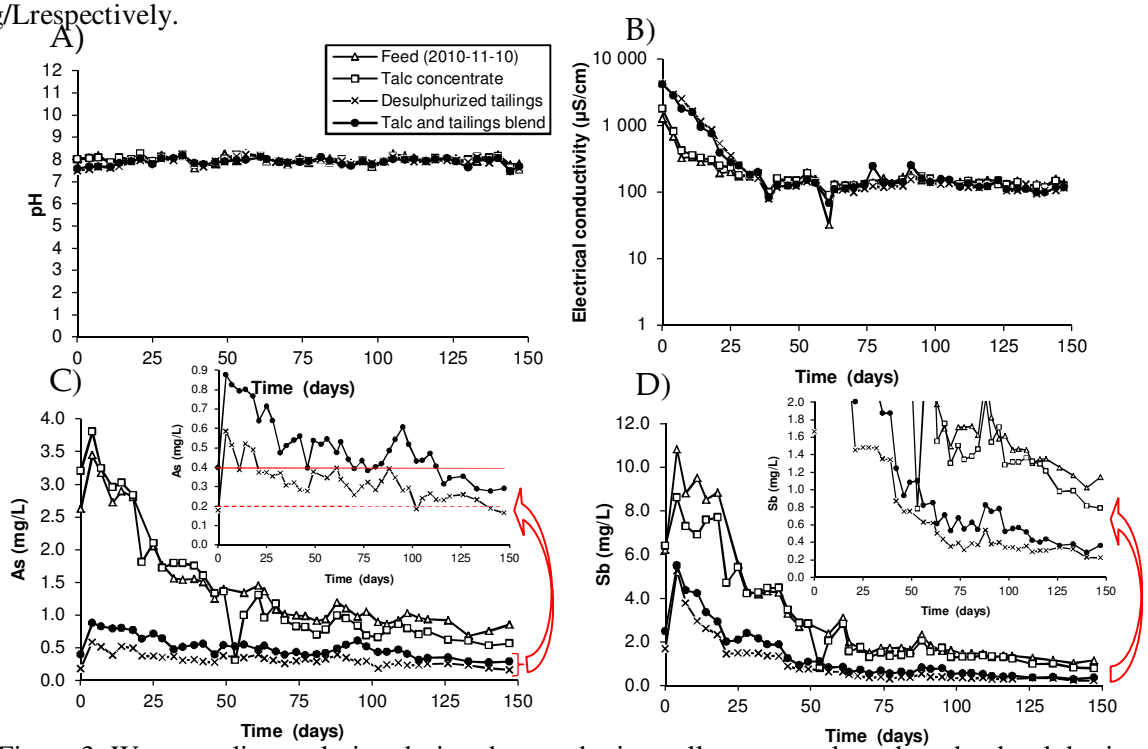

Figure 3: Water quality evolution during the weathering cells tests conducted on the desulphurization products. Notes: log scale for electrical conductivity; the line on the graph C and represent the punctual upper limit of As concentration (norm from Directive 019, 2005); the dotted line correspond to mean upper limit of As concentration. Arsenic concentration limits were used for Sb release concentrations as there are no official limits for this contaminant

On the other hand, the leachates from the desulphurized tailings and the desulphurized tailings and talc concentrate blend samples displayed similar trends. The sulphur (likely sulphates) release concentration decreased steadily during the first 60 days reaching a dissolved concentration of 0.6 mg/L. The arsenic release decreased progressively reaching 0.2 mg/L for the desulphurized tailings and 0.3 mg/L for the blend of tailings and talc concentrate. The blend of desulphurized tailings and talc concentrate was slightly above the limit set at 0.2 mg/L by the Directive 019 (2005) for maximum mean arsenic aqueous emission but the desulphurized stayed within this limits. Antimony release decreased steadily to stabilize around 0.2-0.5 mg/L after 60 days for the desulphurized tailings and the blend.

The calcium and magnesium release concentrations within the desulphurized tailings and the blend decreased during the first 25 days reaching a dissolved concentration of 15 and 3 mg/L like the feed and the talc concentrate respectively. During this period, the sulphate release was greater from the desulphurized tailings and the blend than from the feed and talc concentrate but after reaching the steady state, this trend was reversed.This trend inversion may be explained by the fact that the remaining sulphides contained in the desulphurized tailings are mainly pyrrhotite that may have been destabilized/activated by the acid conditioning during flotation leading to a first 25 days of higher sulphur release (no gypsum detected in materials).

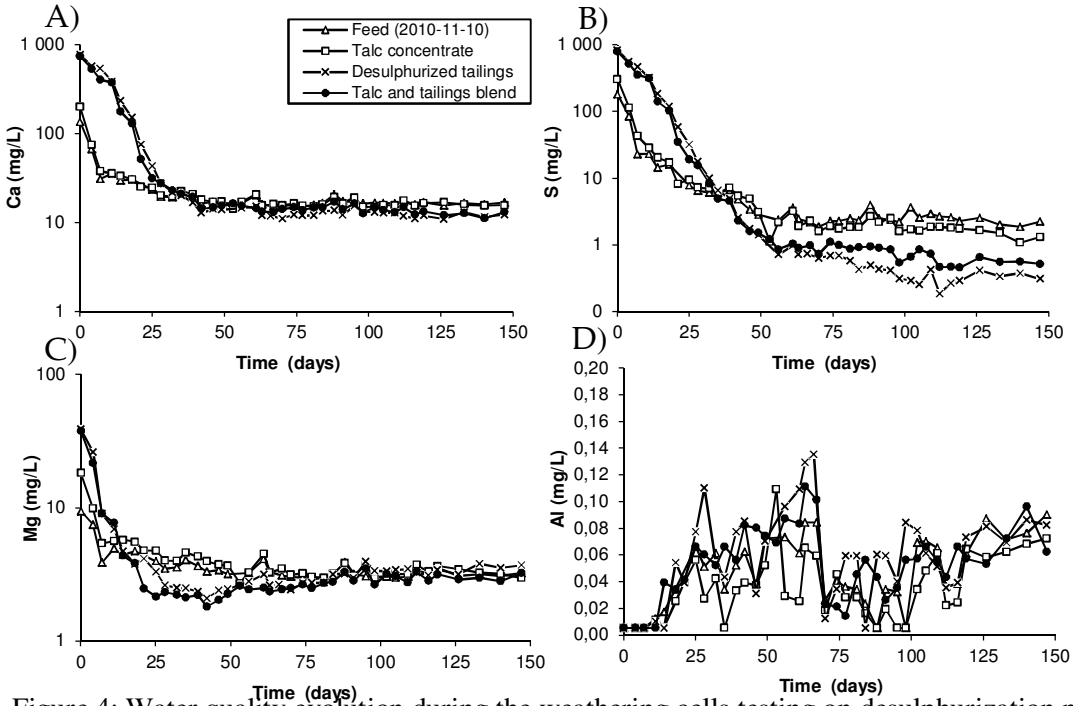

Figure 4: Water quality evolution during the weathering cells testing on desulphurization products
Note: log scale for Ca, S and Mg.

The concentrations of released elements were added and normalized to the sample mass and leachate volume to obtain cumulated value in mg/kg (not presented in the paper). The slope of the cumulated element versus time when a plateau was reached (after 25 days) represent the element release rate (mg/kg/days) which was calculated by linear regression on this portion (Plante et al. 2011a). Release rate data corresponding to the most pertinent elements are presented in Table 4. All linear regressions had high determination coefficient above 0.96. The feed and the talc concentrate released much more arsenic, antimony and sulphate than the desulphurized tailings and the blend which also released more neutralizing cations.

Table 4: Release rates of arsenic, sulphur and neutralizing cations with corresponding determination coefficient (R^2).

	Release rates (mg/kg/day)							
	As	R^2	S	R^2	Sb	R^2	Ca+Mg+Mn	R^2
Feed	0.2093	0.999	0.4638	0.997	0.3723	0.998	2.7728	0.999
Talc concentrate	0.1982	0.997	0.4477	0.993	0.3608	0.994	2.8032	0.999
Desulphurized tailings	0.0554	0.996	0.1445	0.967	0.0872	0.989	3.8655	0.995
Talc and tailings blend	0.0753	0.998	0.1680	0.984	0.1216	0.984	3.4613	0.995

The depletion of sulphur and antimony (related to the oxidation of sulphides), calcium and magnesium (related to the dissolution of neutralizing minerals) are presented in Figure 5. The depletion curves of sulphur and calcium show a two-stage geochemical behavior with a first progressive decrease and a plateau at about 25 days. This kind of depletion trend has been outlined in previous works (Benzaazoua et al. 2004; Cruz et al. 2001; Plante et al. 2011a). The first stage may be related to a rapid dissolution of pre-oxidized soluble phases and to fine particles quicker dissolution. The second stage may be related to passivation due to precipitation of secondary minerals around the sulphide particle. The depletion curve of arsenic and antimony followed a constant decrease that may be related to progressive oxidation of arsenopyrite and berthierite particles.

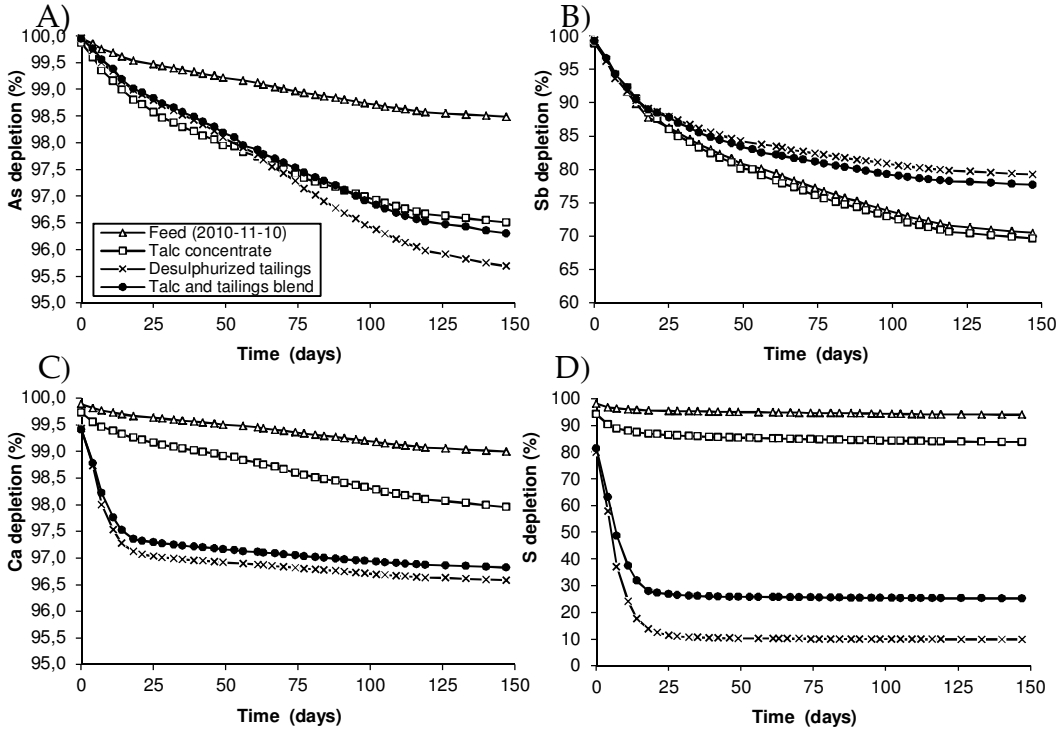

Figure 5: Arsenic (A), antimony (B), calcium (C) and sulphur (D) depletion curves of the four products studied by weathering cells.

The experimental conditions of Eh-pH within the weathering cells are represented in Figure 6 illustrating Eh-pH arsenic species distribution diagram.

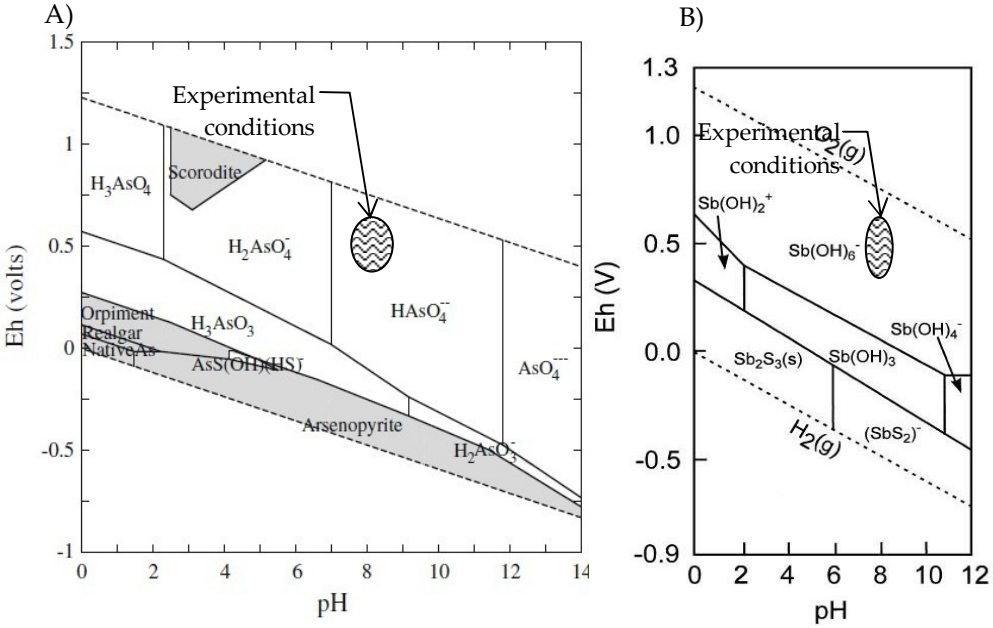

Figure 6: Eh-pH diagram of the system As-O-H-S modified after Lu et al. 2010 (A) and of the system Sb-S-H2O modified after Filella et al. 2002 (B).Circled area corresponds to experimental conditions.

In the experiment, the arsenate ion $HAsO_4^{2-}$ and the antimony ion $Sb(OH)_6^-$ were respectively the major arsenic and antimony phases present in solution. In order to evaluate the impact of arsenic retention phenomena on the arsenic release behavior during the weathering cell testing, the arsenic retention potential of the different materials was estimated through retention tests. As (V) was chosen as the major arsenic phase present in weathering cell leachates (Figure 6).

Retention test

The blank test allowed determination of initially retained arsenic quantity for each tested material. During the blank test, the feed and talc concentrate had the highest concentration of arsenic release with 1.36 and 1.40 mg/L respectively. The desulphurized tailings did not release significant amount of arsenic with only 0.12 mg/L dissolved arsenic in the blank test. Results of retention isotherm tests are presented in Figure 7. The three tested materials showed arsenic isotherms without a strict plateau (Limousin et al. 2007). The isotherms were well fitted with both Langmuir and Freundlich models with determination coefficient above 0.95 (fitting parameters not presented herein). Therefore it is difficult to ascertain whether the arsenic retention process results from a monolayer or multilayer process involving homogeneous or heterogeneous active sites.

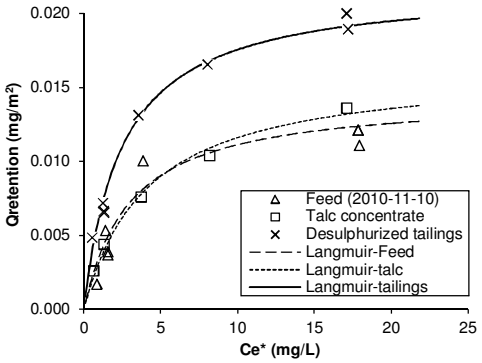

Figure 7: Arsenic retention isotherms (mg/m^2) of the feed, the talc concentrate and the desulphurized tailings showing data point and Langmuir fit arsenic retention.

Some differences could be observed between the three materials tested: the feed and talc concentrate had similar arsenic retention isotherm parameters in accordance with their similar specific surface area (Figure 7). The desulphurized tailings seemed to have slightly higher arsenic retention capacities than the two other materials in accordance with less arsenic release in the blank test.

The maximum sorption capacity estimated from the Langmuir model gave evidence that the three materials of this study had poor arsenic retention capacities of around 50 mg/kg probably due to the neutral pH of leachates as described in a Chakraborty et al. (2007) which evaluated the arsenic retention ability of phyllosilicates and determined that a maximum arsenic retention of 200 mg/kg is reached at acidic pH. Therefore at neutral to high pH, the arsenic retention phenomenon does not have a major impact on arsenic release.

Conclusion and Recommendations

The evaluation of the arsenic release potential on various flotation products resulting from an environmental desulphurization process (talc concentrate and desulphurized tailings) led to the following conclusions and recommendations:

- The weathering cells results suggest that the desulphurized tailings could be classified as a non-arsenic generating material regarding the Directive 019 arsenic limits. However, scale-up testing such as columns or field cell are recommended for final statement.
- Desulphurization lowered antimony release in leachates bringing its concentrations close to arsenic concentration limits.
- The feed and the talc concentrate have equivalent arsenic release potential. The latter is above the limits of arsenic concentration in effluents set by Directive 019 regulation for the mining industry in Quebec (mean upper limit of 0.2 mg/L).
- The talc concentrate has half the arsenic content of the feed but still has high arsenic generating potential. This high arsenic generation potential is probably related to the dissolution of highly reactive fine particles of arsenopyrite entrained in the talc concentrate during its flotation.
- The leachates from the blend of talc and tailings were slightly above the Directive 019 limits for arsenic generation. Therefore, the talc concentrate should be stored with the sulphide concentrateas an arsenic generating material.
- The authors suggest evaluating the kinetic of arsenic entrainment during the talc flotation so that it may be possible to separate the talc concentrate into more or less arsenic rich fraction and to minimize the volume of arsenic generating material. Processing the talc concentrate through physical separation to remove fine particles of arsenopyrite could also be considered.
- The arsenic retention capacity of the three tested material is very low due to the neutral pH of leachates. Arsenic retention does not have a major impact on the arsenic release phenomenon observed in the weathering cells at neutral pH.
- The sorption capacities of the different minerals constituting the Lapa material were not investigated separately as pure minerals. Further investigations would be required to evaluate the sorption capacity of each constituting mineral on the overall arsenic retention process. The role of phyllosilicates such as chlorite and biotiteas well as siderite in arsenic retention have been evaluated in the literature (Lin and puls 2000;Chakraborty et al. 2007;Guo et al. 2011).

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Acronyms

AMD: Acid Mine Drainage
AP: Acid Potential
B.E.T. method: Brunauer, Emmet Teller method
CND: Contaminated Neutral Drainage
ICP-AES: Inductively Coupled Plasma and Atomic Emission Spectroscopy
NP: Neutralization Potential
NNP: Net Neutralization Potential
OM: Optic Microscopy
SEM: Scanning Electron Microscopy
Gs: Specific gravity
SSA: Specific Surface Area
XRD: X-Ray Diffraction