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TECHNICAL ARTICLE

ASTM Normalized Humidity Cell Kinetic Test: Protocol Improvements for Optimal Sulfide Tailings Reactivity

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Abstract The humidity cell (HC) kinetic method is widely used to assess acid-generating potential and is the only method normalized by the American Society for Testing and Materials (ASTM). HC test results can be influenced by complete drying of the sample or by a long water saturation step during weekly cycles, which significantly reduces sulfide oxidation rates, leading to erroneous interpretations. This paper investigates a protocol modification of the HC, which involves keeping the sample permanently at an optimal degree of saturation, between 40 and 60 %, corresponding to maximal sulfide reactivity, as demonstrated in the literature. For this purpose, two mine tailings with different acid-generating potentials were submitted to both modified and standard ASTM HC protocols. The influence of the thickness and the porosity of the sample into the HC were also investigated. The results obtained clearly show that, for a given sample, the oxidation rate under the modified ASTM protocol was higher than the standard ASTM protocol, which underestimated tailings reactivity due to excessive drying of the sample. On the other hand, when the porosity of the sample decreased, and its thickness increased, the difference between the standard and the modified HC protocol results decreased. These differences were more significant when the sample was more acid-generating.

Keywords Acid mine drainage · Prediction · Oxidation rates · Sulfate production

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Introduction

Mining generates large amounts of tailings that often contain sulfide minerals. When exposed to water and oxygen, some tailings weather, producing acidity and dissolved metals. These acidic, metal-loaded effluents, called acid mine drainage (AMD), represent a major environmental problem facing the mining industry worldwide (e.g. Kleinmann et al. 1981). Static tests (acid–base accounting (ABA) or net acid generation) are frequently used as to assess acid-generating potential (AGP) and classify materials as acid generating, non-acid generating, or uncertain. When the AGP of a given material is uncertain, kinetic tests becomes necessary to: provide a direct estimate of relative acid generation and neutralization rates under fully oxygenated conditions, and better understand the geochemical behavior of tailings regarding metal solubilization and leaching.

The most commonly used kinetic tests for AMD characterization are laboratory column, humidity cells, and field-based pad tests (Bowell et al. 2006; Frostad et al. 2002; Lapakko and White 2000; Price and Kwong 1997; Price 2009; Plante et al. 2012; Sapsford et al. 2009). Column and humidity cells kinetic tests are based on the alteration of mine wastes under controlled conditions simulating an intense weathering environment to evaluate their long-term environmental behavior. Humidity cells are a well-established form of kinetic test to supplement static ABA testing; they are widely used to estimate weathering rates in order to predict the depletion of acid generation (AP) and neutralization potential (NP), and the lag time to the onset of AMD (Benzaazoua et al. 2001, 2004; Frostad et al. 2002; Villeneuve 2004; Villeneuve et al. 2003, 2009).

Previous laboratory work has shown contradictory results on AGP prediction for the same tailings, depending

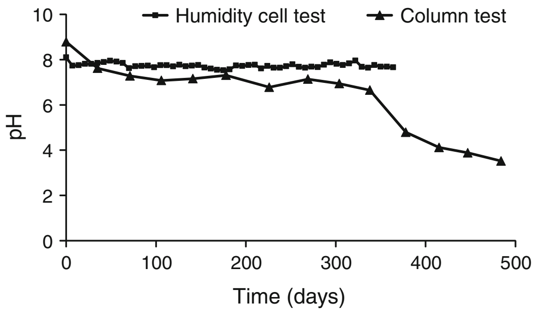


Fig. 1 Evolution of leachates pH for the same sample submitted to column and humidity cell kinetic test (after Benzaazoua et al. 2008; Demers et al. 2008)

on the type of kinetic test used. Benzaazoua et al. (2008) noticed some reactivity inhibition when tailings samples were submitted to the normalized American Society for Testing and Materials (ASTM) humidity cell. They reported that the leachate pH remained neutral over 364 days, corresponding to 52 cycles (Fig. 1), while Demers et al. 2008 noticed that the same sample submitted to a column kinetic test became acidic after 380 days, which corresponds to only 10 cycles. Sapsford et al. (2009) tested crushed waste rock (100 % passing 5 mm) in an aerated ASTM humidity cell kinetic test in duplicate. They showed that the calculated sulfate release rate (mg/kg/cycle) from the two cells were very different, and found that the aeration system of one cell had malfunctioned, leading to a much drier condition than in the other cell. This had the effect of reducing the quantity of water available to sustain pyrite oxidation. Bowell et al. (2006) observed the same phenomenon when analyzing weathering of volcanogenic massive sulfide material (crushed to 100 % passing 5 mm) on two replicated humidity cells (HCs). The difference in terms of sulfate release rates between the two HC tests were attributed to excessive drying in one cell, which was thought to be due to an increase in air-flow through it. These results suggest that complete drying of the materials in the cells during weekly cycles can inhibit pyrite oxidation rates by removing interstitial water. Frostad et al. (2002) evaluated five laboratory kinetic test protocols (standard humidity cells, non-aerated cells, tall cells, shaken cells, and NP depletion columns) by comparing sulfate release and NP depletion rates. Based on the results, the authors reported that the standard humidity cell set-up creates an unnatural oxidizing environment, leading to extreme wetting and drying cycles, and therefore produced inconsistent results. Bowell et al. (2006) also provided evidence that kinetic test results can be influenced by internal factors (particle size, mineralogy, solution/mineral interaction within the cell induced by biotic and abiotic interactions) in the sample being tested and by external factors related to testing conditions such as aeration degree, sample weight, flushing frequency, and the duration of the test. The internal processes and the extent of aeration appear to have significant impact on the mineral species leaching rates.

Based on sulfide reactivity measurements made on diverse tailings, reactive tailings consume more O_2 than less reactive ones, and, for any given tailings, the reactivity rate coefficient (K_r) is low when saturation (S_r) is ≤ 20 % and ≥ 80 % (Demers et al. 2009; Gosselin et al. 2007; Hamdi 2011). The K_r reaches a maximum at S_r values between 20 and 80 %, allowing greater sulfide oxidation, especially around $S_r = 50$ % (Gosselin et al. 2007; Hamdi 2011). Martin et al. (2006) showed that K_r is influenced by S_r and by in situ porosity (n). Less S_r or greater n can cause oxygen flux (or O_2 consumption) by the reactive tailings and AMD production to increase. In summary, a high degree of saturation ($S_r > 85$ %) of a given tailings reduces oxygen availability and sulfide oxidation (Hornberger and Brady 1998; Mbonimpa et al. 2003; Ouangrawa et al. 2009) while a low degree of saturation ($S_r < 20$ %) reduces the water availability required for optimal oxidation reactions (Godbout et al. 2010).

Based on these findings, the present study focused specifically on the humidity cell testing procedure, the only one normalized by ASTM and the most widely used method for AMD prediction (Benzaazoua et al. 2001, 2004; Morin and Hutt 1998; Plante et al. 2011; Price 2009; Sapsford et al. 2009; Villeneuve et al. 2003, 2009). We investigated some modifications of the ASTM humidity cell procedure for mill tailings, which are often characterized by a fine particle size distribution (<200 μm). The main objective of this paper is to suggest a modification to the standard ASTM protocol (ASTM D5744-07, option A), consisting of maintaining the sample at a degree of saturation (S_r) between 40 and 60 % during the 7 day duration of the HC cycles. The influence of the thickness and the porosity of the sample into the HC were also investigated. Hence, the paper will quantitatively show the influence of saturation, initial porosity, and sample thickness on the oxidation rate and the HC results.

Materials and Methods

Two mine tailings, named herein A and B, from the Abitibi region in Québec province (Canada) were used in this study. Tailings A, which is recognized as being highly acid generating, comes from a Cu–Zn mine, while tailings B comes from a Cu–Zn-Au–Ag mine. The two mine tailings were sampled in the non-oxidized zone in each corresponding impoundment (1 m deep). After sampling, both tailings were placed immediately in containers and flooded with deionized water to prevent sulfide oxidation during transport and lab storage.

Material Characterization

The specific gravity (G_s) of the tailings samples was determined with a Micromeritics helium pycnometer. The particle size distribution was determined using a Malvern Mastersizer laser particle size analyzer. The chemical analysis was performed using acid digestion ($HNO_3/Br_2/HF/HCl$) followed by ICP-AES analysis for over 20 elements. Silica is partially evaporated during the digestion procedure and therefore is not reported in this study. Sulfide sulfur was determined by subtracting the sulfate sulfur (determined by a 40 % HCl extraction; method adapted from Sobek et al. 1978) from the total sulfur. Analysis of total sulfur and total inorganic carbon was also performed by induction furnace (ELTRA 2000-CS).

The tailings were mineralogically characterized with X-ray diffraction (XRD) using a Bruker AXS D8 advance X-ray diffractometer equipped with a copper anticathode, scanning over a diffraction angle (2θ) range from 5° to 60°. Scan settings were 0.005° 2θ step size and 1 s counting time per step. DiffracPlus EVA software (v.9.0 rel. 2003) was used to identify mineral species and the TOPAS software (v 2.1) implementing Rietveld refinement was used to quantify the abundance of all identified mineral species (Young 1993). The absolute precision of this quantification method is of the order of ±0.5–1 % (Bouzahzah et al. 2008; Mermillod-Blondin 2005; Raudsepp and Pani 2003).

The mineralogy was more deeply investigated by optical microscopy. Polished sections prepared with bulk samples (impregnated in epoxy resin) were observed by reflected light microscopy. Scanning electron microscope (SEM) observations using backscattered electrons were achieved on a Hitachi S-3500N microscope equipped with an X-ray energy dispersive spectrometer (EDS; silicon drift spectrometer X-max 20 mm^2) with INCA platform (450 Energy). The operating conditions were 20 keV, $\approx$100 μA, and 15 mm working distance.

For AGP assessment using ABA, a Sobek et al. (1978) test modified by Lawrence and Wang (1997) was used, where NP and AP are expressed in kg of $CaCO_3$ equivalent per tonne of sample. The net neutralization potential (NNP) was used to predict the AGP by subtracting the AP from the NP (Ferguson and Morin 1991; Miller et al. 1991; Weber et al. 2004). If the NNP value is higher than 20 kg $CaCO_3$/t, the sample is considered not acid-generating, if NNP is lower than −20 kg $CaCO_3$/t, the sample is considered acid-generating, and finally, if the NNP is between −20 and 20 kg $CaCO_3$/t, the sample is uncertain with respect to AGP.

Kinetic Tests

The kinetic apparatus and procedures used in this study are based on the HC device and protocols described in ASTM D5744-07 (option A, 2007). The size and shape of the HC vary whether the sample consists of coarse waste rocks or fine tailings. For the mine tailings in this work, the HC tests are performed in a Plexiglas chamber that provides air input and output. The cells have an inside diameter (ID) of 20.3 cm and a height of 10.2 cm. Approximately 1 kg (dry weight) of sample is placed in the humidity cell on a perforated plate covered with two geotextile layers to prevent loss of fine particles during the test. The geotextile layer is chemically inert and porous enough for easy flow of leachate while retaining the sample.

The standard ASTM kinetic test consists of a weekly dry–wet cycle. The first 3 days of the cycle correspond to the dry period, during which dry air is blown over the sample to ensure sufficient oxygen availability so that reaction rates are not limited. ASTM recommends a wide range of airflows between 1 and 10 L/min. The next 3 day period is the "wet" step of the testing cycle, when air is first pumped through a humidifier unit supplying water-saturated air and then injected into the cell. On the seventh day, the sample is flushed by adding a fixed volume of deionized water (0.5–1 L) to the top of the cell. The purpose of the weekly leaching is to wash out all weathering reaction products that have accumulated during the week (Usher et al. 2003). The standard ASTM methodology suggests a minimum test period of 20 weeks, but they typically run for at least 40 weeks, or until the rates of sulfate generation and metal leaching are stable for at least 5 weeks (Sapsford et al. 2009; Usher et al. 2003).

For both tested tailings A and B, the kinetic tests were performed according to the HC set-up schematized in Fig. 2 to investigate the influence of the weekly dry–wet cycle, sample porosity, and sample thickness on reactivity. All test set-up and operatory condition details are summarized in Table 1. For the three studied parameters and for each tailings sample, two humidity cells were set up in parallel: the first cell was handled using the standard ASTM protocol described above, while the second cell was submitted to a modified protocol. The protocol modification consisted of keeping the sample at S_r values between 40 and 60 %, which is recognized as the optimal range of saturation to maximize sample reactivity (Bouzahzah et al. 2010, 2012; Demers et al. 2009; Gosselin et al. 2007; Hamdi 2011; Martin et al. 2006; Mbonimpa et al. 2003).

After the first flush of the cells submitted to the modified protocol, S_r was maintained between 40 and 60 % by manually adding deionized water during the dry cycle to ensure

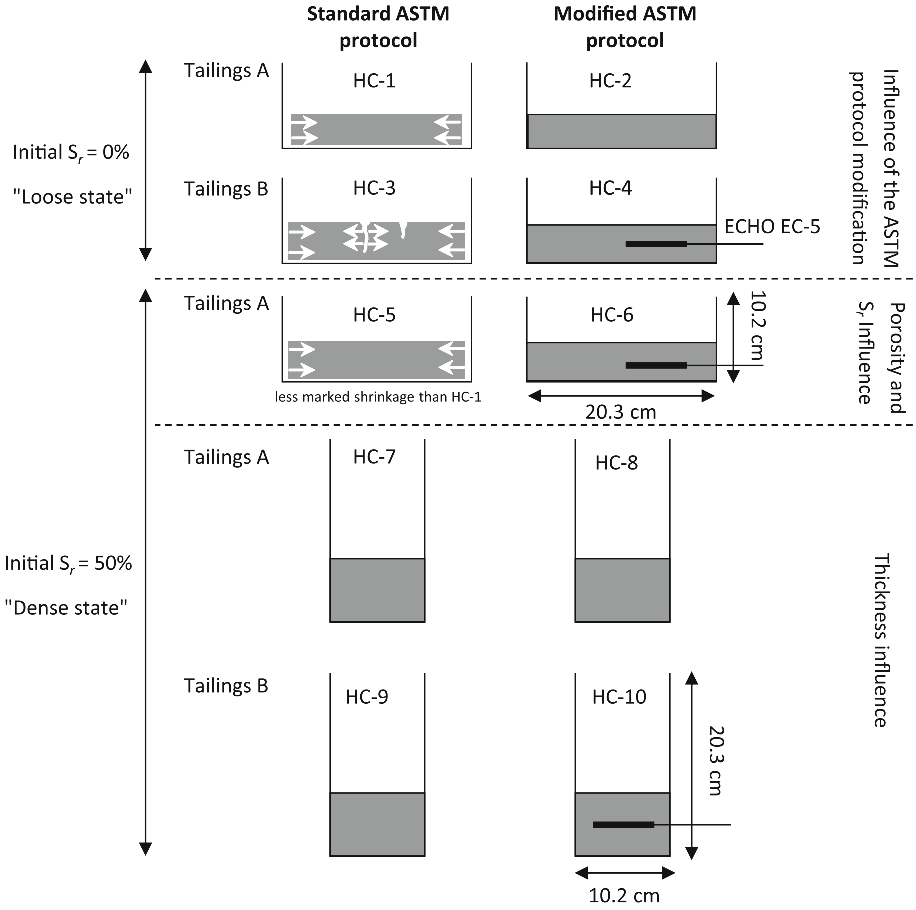


Fig. 2 Schematic representation and summary of humidity cell test set-ups

Table 1 Summary of kinetic test set-ups and operatory conditions adopted for the HC tests

Description	HC-1	HC-2	HC-3	HC-4	HC-5	HC-6	HC-7	HC-8	HC-9	HC-10
Tailings	A	A	B	B	A	A	A	A	B	B
Diameter (cm)	20.3	20.3	20.3	20.3	20.3	20.3	10.2	10.2	10.2	10.2
Height (cm)	10.2	10.2	10.2	10.2	10.2	10.2	20.3	20.3	20.3	20.3
Weight (kg)	1	1	1.39[a]	1.39[a]	1.13[a]	1.13[a]	1	1	1	1
Air flow	1–1.5 L/min									
Rinsing water	500 mL									
Dry air	3 days									
Moist air	3 days									
ASTM protocol	Std	Mod	Std	Mod	Std	Mod	Std	Mod	Std	Mod
Deionized water addition	No	Yes	No	Yes	No	Yes	No	Yes	No	Yes
Initial Sr (%)	0	0	0	0	50	50	50	50	50	50
Porosity *n* (–)[b]	0.72	0.7	0.54	0.54	0.47	0.48	0.12	0.13	0.12	0.17
Sample height (cm)[b]	3.50	3.30	3.36	3.35	2.13	2.15	4.47	4.51	4.98	5.23
Soil moisture probe	No	No	No	Yes	No	Yes	No	Yes	No	No

std standard ASTM protocol, *Mod* modified ASTM protocol

[a] The sample weight was >1 kg to ensure a sufficient thickness to cover the moisture probe

[b] Measured at the end of the test

uniform sample wetting. The saturation was monitored by (1) a soil moisture probe (ECHO EC5) in HC-4, 6, 10, and (2) by weighing the humidity cells (with sample) over the test period. The degree of saturation was deduced by calculating the water loss and water gain, and using geotechnical parameters of the material placed in the humidity cell (sample thickness, diameter, specific gravity, porosity, and the initial cell weight, with sample). Cell weight was measured 3 times a week: at the end of the sample wash, at the end of the dry air period, and at the end of the moist air period.

Dry and humid air fluxes (1–1.5 L/min) were kept constant during the test with a flow meter. The humidifier's water temperature (25–30 °C to give 99 % air moisture: ASTM D5744-07-2007) was also regulated. During the seventh day of each cycle, 500 mL of deionized water was added to each cell for a period of 3–4 h before letting the water drain. Leachates collected from the HCs were weighted and analyzed for several chemical parameters. Conductivity, pH, and Eh were measured using an Accumet excel XL60 dual channel pH/ion/conductivity/DO meter. The pH was determined with an AccupHast electrode, which had been calibrated against commercial buffer solutions (pH 4, 7, and 10) before each measurement. The leachate's redox potential (Eh) was measured using an ORP probe. Electrical conductivity of the samples was measured using an Accumet electrode. Alkalinity and acidity were determined using standard titration techniques at an endpoint pH of 4.5 with H_2SO_4 for alkalinity and 8.3 with NaOH for acidity (APHA 1995). Cell leachates were filtered through a 0.45 µm filter, acidified with 2 % HNO_3, and then analyzed using ICP-AES.

Results and Discussion

The following sections compare the results of HC tests run with the standard and modified ASTM protocols with respect to key parameters like sample saturation, pH, Eh, conductivity, acidity, and the most relevant chemical elements for the purpose of this study. Weekly leachate analyses (mg/L) were used with leachate volume (L) and sample initial mass (dry weight in kg) to calculate leaching rates (mg/kg/week) related to chemical elements of interest. The leaching rates related to the standard and modified ASTM protocols were compared by plotting the cumulative leached elements over time.

Tailings Characterization

Tailings A: Characterization results are summarized in Tables 2 and 3. The grain size parameters D_{10}, D_{50}, D_{80}, and D_{90} are typical of hard rock mine tailings (Aubertin et al. 2002; Bussière 2007) with a D_{90} value of 105 µm. The XRD results (Table 3) show that pyrite was the only sulfide mineral detected (20 wt%). Gangue minerals were mainly composed of quartz (44.3 wt%) and muscovite (22.6 wt%), with minor proportions of albite and chlorite at 6.7 and 3.8 wt%, respectively. Gypsum (2.5 wt%) was also present, possibly as a secondary mineral. Sphalerite, chalcopyrite, magnetite, and rutile were identified as trace minerals by reflected light and electronic microscopy. No mineral with neutralization potential is present in the sample (NP = 0), which is considered acid generating (NNP = −401 kg $CaCO_3$/t), according to static test classification criteria (Miller et al. 1991).

Table 2 Physical and chemical characteristics of the two studied tailings

	A	B
Laser PSD (µm)		
D10	5.1	3.9
D50	24	22
D80	56	145
D90	105	237
Specific gravity (Gs) (g/cm^3)	3.1	2.8
ICP-AES (wt%)		
Al	5.17	3.53
Ca	0.74	0.43
Cu	0.02	0.04
Fe	15.5	6.1
Mg	0.73	1.97
Mn	0.02	0.07
Pb	0.09	0.02
Ti	0.05	0.04
Zn	0.51	0.19
S_{total}	13.5	2.49
$S_{sulfate}$	0.66	0.0
S_{sulfur}	13.28	2.49
Induction furnace (wt%)		
S_{total}	13.99	2.55
C_{total}	0.06	0.31
ABA test (kg $CaCO_3$/t)		
NP	0	19
AP	415	77.8
NNP	−415	−59

Tailings B: The grain size distribution with D_{10}, D_{50}, D_{80}, and D_{90} values is presented in Table 2. The D_{90} value is 237 µm, showing that this sample was coarser than tailings A. The specific gravity (Gs), chemical composition, and ABA results are also summarized in Table 2. The XRD quantification results (Table 3) show that quartz (73.7 wt%), chlorite (11 wt%), and muscovite (9.4 wt%) are the major mineral phases dominating the gangue assemblage in tailings B. Pyrite is the dominant sulfide mineral (3.5 wt%) and there are trace amounts of sphalerite, chalcopyrite, rutile, and magnetite as proven by optical and electronic microscopy on a polished section. Carbonate minerals are dolomite (2 wt%) with a small amount of calcite (0.4 wt%), conferring to the sample a weak neutralization potential (NP = 19 kg $CaCO_3$/t) compared to the acidification potential of 78 kg $CaCO_3$/t (Table 2). Based on the NNP classification criteria proposed by Miller et al. (1991), this sample is considered acid generating (NNP = −59 kg $CaCO_3$/t).

Table 3 Semi-quantitative X-ray diffraction bulk mineralogy of the studied samples (wt%)

	Quartz	Muscovite	Albite	Chlorite	Calcite	Dolomite	Pyrite	Gypsum
Tailings A	44.3	22.6	6.7	3.8			20.1	2.5
Tailings B	73.7	9.4		11	0.4	2.0	3.5	

Kinetic Test Results

The following sections present quantitatively the influence of the ASTM humidity cell protocol modification, porosity and sample thickness on tailings oxidation rates by keeping S*r* within an optimal range during the weekly drying-wetting cycles.

Influence of the ASTM Humidity Cell Protocol Modification (Weekly Dry–Wet Cycles) on Sample Reactivity

Tailings A: Figure 3a shows the S_r variation within tailings A placed in the two humidity cells; the first runs under the standard (HC-1) and the second under the modified protocol (HC-2). The S_r was maintained between 40 and 60–70 % in the modified protocol, while it decreased progressively in the standard protocol over the 24 weeks of testing, until it reached 0 % by the end of the test. In fact, the sample started to dry out from week #12 and continued to lose water gradually until it was desaturated at week #17 (as determined by cell weight), even with the water addition for rinsing at each cycle. The drying was also visually apparent: the dry material (standard protocol) was light grey compared to the dark grey of the moist material, which remained thoroughly wetted during the entire test when the modified protocol was used (Fig. 3b). The excessive drying observed in the standard protocol was due to progressive water loss caused by air-flow through the sample (evaporation). Some difficulties were encountered in maintaining equal and uniform airflow through the test, causing a drop of the S_r in the HC-2 by week #12 (day 81), week #16 (day 116), and week #19 (day 134), indicated by an arrow in Fig. 3. The variation in air flow was due to instability of the laboratory compressed air system.

The pH, Eh, conductivity, and acidity of the leachates are plotted against time, showing their trends in the two cells throughout the experiments (Fig. 4). The leachates associated with the modified HC were more acidic, with a pH of 2.6, higher Eh (664 mV), higher acidity (1,473 kg $CaCO_3$/mL), and higher conductivity (3,000 µS/cm). Leachates from the standard protocol showed higher pH and lower values for the other parameters (average values: pH 3.8, Eh 584 mV, acidity: 93 kg $CaCO_3$/mL, and conductivity: 711 µS/cm). As shown in Fig. 4, after 160 days of testing, the modified protocol cell released cumulative amounts of SO_4^{2-} ($\approx$6,400 mg/kg), Fe ($\approx$4,000 mg/kg), Zn ($\approx$675 mg/kg), Al ($\approx$340 mg/kg), Mg ($\approx$200 mg/kg), Ca ($\approx$2,000 mg/kg) and Si ($\approx$340 mg/kg) that were much higher than the corresponding values measured in the standard protocol (SO_4^{2-}: 1,430 mg/kg, Fe: 330 mg/kg, Zn: 110 mg/kg, Al: 100 mg/kg, Mg: 40 mg/kg, Ca: 1,160 mg/kg, and Si: 110 mg/kg). At first sight, this difference could be explained by secondary minerals precipitating in the standard HC, leading to lesser leaching rates. In fact, if reaction rates were faster than the removal of dissolved constituents in the interstitial water, secondary mineral precipitation could explain the observed difference between the standard and modified protocols. Secondary sulfate mineral precipitation (like gypsum and jarosite at more acidic pHs) can remove sulfate from solution and give the impression that sulfide oxidation rates are slower (Bowell et al. 2006). Morin and Hutt (1998) demonstrated how gypsum precipitation has led to erroneous interpretations of weathering rates in many humidity cell studies. To be sure that the differences in leachate loads between HCs under the standard and the modified protocols were not due to sulfate precipitation, the kinetic tests were dismantled at the end of the kinetic tests and analyzed for sulfate. The results showed negligible sulfate content, suggesting that no secondary sulfate mineral precipitation had occurred. Geochemical modeling using Visuel MINTEQ ver. 3.0 (Felmy et al. 1984) also confirmed that no secondary sulfate minerals should have precipitated in the conditions of the kinetic tests. The HC flushing ensured nearly complete removal of dissolution products and that all sulfate released by pyrite oxidation was removed by the rinsing. Therefore, the weekly release rates of sulfate reported as mg SO_4/kg/week represent the actual pyrite oxidation rate and can be used to compare the standard and modified protocols (Fig. 4). The cumulative sulfate released at the end of the test in the modified HC was 6,370 mg/kg/week, almost 4.5 times higher than that calculated for the standard protocol (Table 4).

Moreover, in acidic conditions ($pH < 3.5$), Zn and Fe are mobile (Delmas-Gadras 2000; Dold and Fontboté 2002; Dold et al. 2005; Dold 2006; Gräfe and Sparks 2005; Smuda et al. 2007; Warwick et al. 1998) and can also be used as markers for sulfide oxidation rates. Saturation indices calculated with Visual MINTEQ (ver. 3.0) indicated that leachates from both HC-1 and HC-2 were undersaturated with respect to Fe–Zn-bearing minerals.

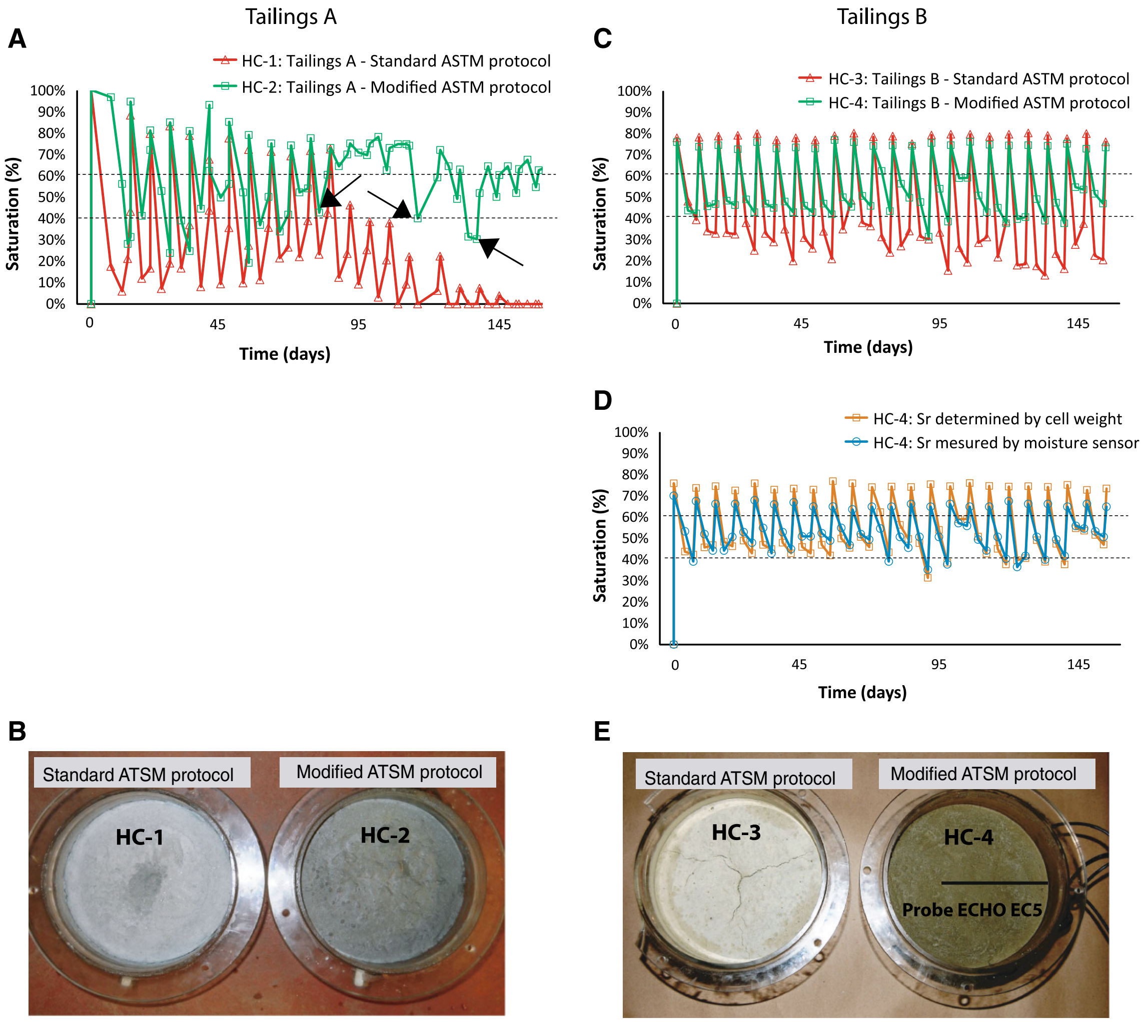


Fig. 3 Standard and modified HC comparison: **a**, **c** saturation profile in tailings A and B humidity cells corresponding to the modified and standard protocol (the *two horizontal lines* represent the targeted S_r values between 40 and 60 %). **d** Saturation profiles determined by HC weighing and moisture sensor in tailings B. **b**, **e** Pictures showing the difference in sample moisture under standard and modified HC protocol (*darker color* (HC-2 & HC-4) is related to the higher water content)

Using Zn and Fe as comparison parameters, the modified protocol generated 6–12 times more dissolved metals than the standard protocol (Table 4).

Tailings B: The S_r of tailings B was monitored over the testing period by weighing the HCs with the samples (similar to tailings A) and by a soil moisture probe (only for tailings B). Figure 3c shows the S_r profile of the two HCs under the standard (HC-3) and the modified protocol (HC-4). Saturation curves determined with a moisture sensor and by HC weighing are very similar (Fig. 3d). In the modified protocol, the saturation was maintained between 40 and 60 %, while it ranged between 10 and 35 % in the standard protocol.

Despite a significant saturation difference between samples in the HC-3 and HC-4, the average pH, conductivity, Eh, acidity, and alkalinity values were relatively similar between the two HCs under standard (pH 7.7, Eh 500 mV, alkalinity: 6 kg $CaCO_3$/mL, conductivity: 790 µS/cm) and modified protocols (pH 7.6, Eh 500 mV, alkalinity: 6.5 kg $CaCO_3$/mL, conductivity: 875 µS/cm) (Fig. 4). The cumulative SO_4^{2-}, Mg, and Ca leached over the 22 cycles were also similar between the two cells (Table 4). However, the cumulative values of zinc (4 mg/kg), Mn (8.5 mg/kg), and Si (113 mg/kg) were 1.5 times higher for the modified protocol than for the standard protocol (Zn: 2.5 mg/kg, Mn: 5.6 mg/kg, Si: 79 mg/kg). Using Zn as a comparative parameter due to its relatively high mobility in these pH conditions, the HC modified protocol allowed more favorable oxidation conditions, but the difference between the two HC oxidation rates was low, compared to tailings A.

The higher leaching rates under the modified protocol, observed in the HCs for both tailings A and B, and higher

Fig. 4 Evolution of pH, Eh, conductivity, acidity and total cumulative leached SO_4^{2-}, Fe, Zn, Al, Ca, Mg, Mn and Si after 160 days of HC testing

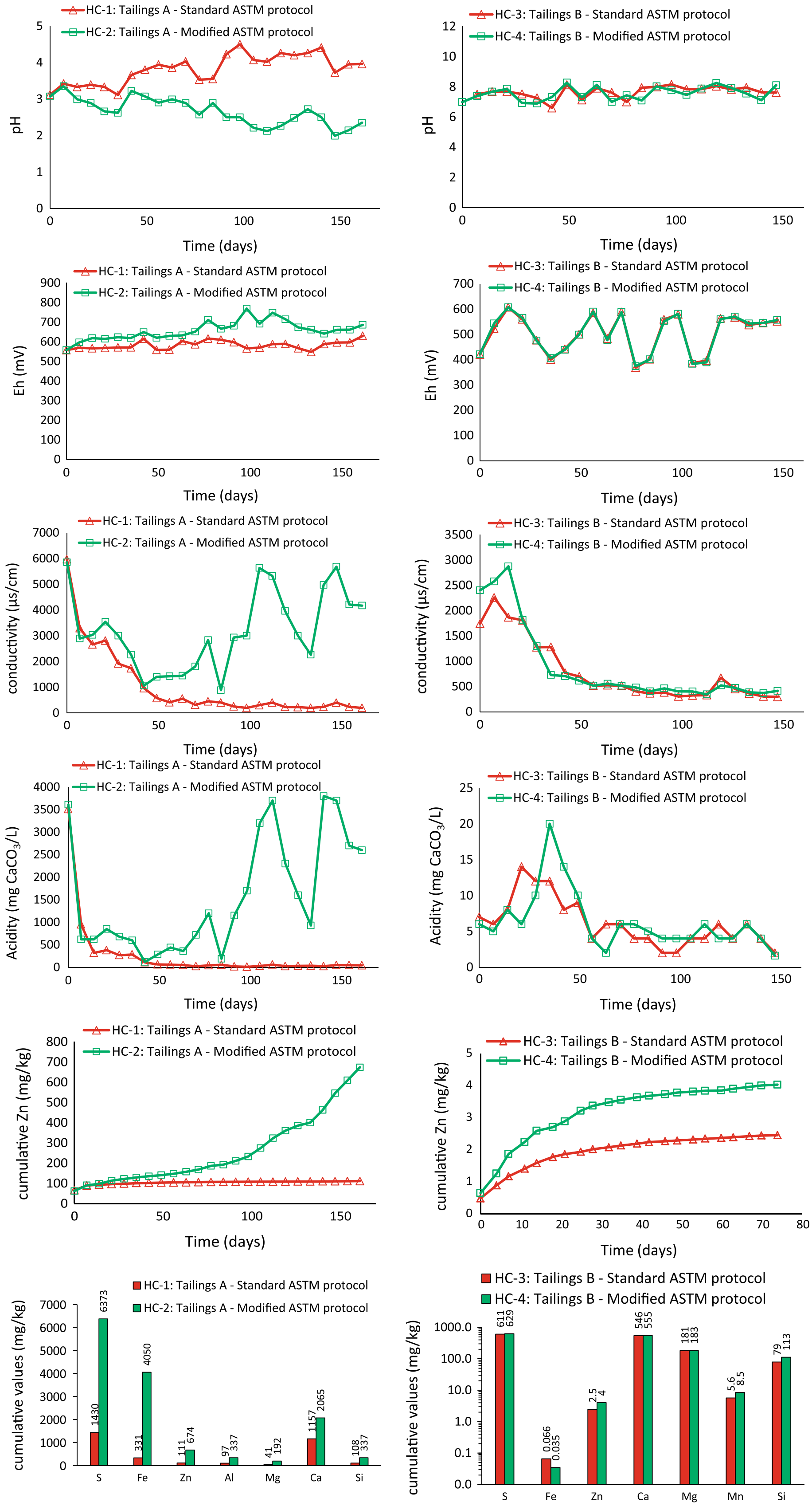


Table 4 Ratios of cumulative values of dissolved main elements from standard and modified HC tests (mg/kg/week)

	Humidity cell protocol	S	Fe	Zn	Al	Mg	Ca	Mg	Mn	Si
HC-1	TA—standard ASTM protocol	1,430	331	111	97	41	1157			108
HC-2	TA—modified ASTM protocol	6,373	4,050	674	337	192	2,065			337
Ratios		4.5	12.2	6.1	3.5	4.7	1.8			3.1
HC-3	TB—standard ASTM protocol	611	0.066	2.5			546	181	5.6	79
HC-4	TB—modified ASTM protocol	629	0.035	4			555	183	8.5	113
Ratios		1.0	0.5	1.6			1.0	1.0	1.5	1.4
HC-5	TA—standard ASTM protocol	2,321	856	328	265	137	1,027			107
HC-6	TA—modified ASTM protocol	5,889	2,805	474	362	200	3,113			431
Ratios		2.5	3.3	1.4	1.4	1.5	3.0			4.0
HC-7	TA—standard ASTM protocol	4,446	1,553	303	315	169	2,763			420
HC-8	TA—modified ASTM protocol	5,863	2,053	338	255	137	4,085			450
Ratios		1.3	1.3	1.1	0.8	0.8	1.5			1.1
HC-9	TB—standard ASTM protocol	965	0.032	2.3			915	256	14.4	83
HC-10	TB—modified ASTM protocol	954	0.184	2.8			926	261	15.3	51
Ratios		0.99	5.8	1.2			1.0	1.0	1.1	0.6

TA tailings A, *TB* tailings B

pH, Eh, conductivity, and acidity (especially for tailings A), are attributed to the higher sulfide oxidation rates when using the modified HC procedure. This higher oxidation rate is attributed to the S_r, which was maintained between 40 and 60–70 % by manually and continuously adding deionized water to the sample. Conversely, under the standard HC test protocol, the lower tailings reactivity is related to the insufficient water supply, and consequently, to a too low saturation of the sample, which is unsuitable for sustaining a high sulfide oxidation rate. The low reactivity difference of tailings B between the standard and modified HC, compared to tailings A, is probably related to its lower acid generating potential (Table 2). This difference may also be related to the ability of tailings B to retain some moisture from humid air during the moist cycle. In fact, tailings B is sandy and coarse-grained ($D_{80} = 145$ µm) whereas tailings A is fine-grained and clayey ($D_{80} = 56$ µm).

Influence of the Combined Initial Porosity, Saturation, and Weekly Dry–Wet Cycles on Sample Reactivity

The tailings A sample was used for this part of the study because of its higher reactivity, as observed by the difference in leachate chemistry between the standard and modified protocol (HC-1 and 2). The experiment was performed with two cells: the first followed the standard ASTM protocol (HC-5) and the second followed a modified ASTM protocol (HC-6), in which the sample was kept at S_r values between 40 and 60 %. The difference from the previous experiment was that the sample was compacted to reduce porosity and had an initial S_r of 50 %. The targeted porosity was 0.47, which is a typical value for tailings from hard rock mines (Bussière 2007; Demers et al. 2011). During the test, sample saturation was monitored by a soil moisture probe (ECHO EC5, only on HC-6 under modified protocol) and by weighing the humidity cells (HC-5 and 6). The experimental procedure was the same as the previous experiments (HC-1 to HC-4).

Figure 5b shows the water saturation (S_r) profile in the two humidity cells using the standard (HC-5) and modified protocols (HC-6). Saturation curves determined by moisture sensor and by HC weighing were very similar (Fig. 5c). In the modified protocol, the saturation was maintained between 40 and 60 %, while it was between 10 and 30 % in the standard protocol. As opposed to HC-1, the sample in HC-5 was not completely dry at the end of the test.

The leachate chemistry (Eh, pH, conductivity, acidity, metal loads) shows that the difference between the standard and modified protocol (HC-5 and HC-6) was less noticeable (Fig. 6) than in the first series, when samples were initially installed in a dry state. The cumulative values of Zn (475 mg/kg), Fe (2,800 mg/kg), S (5,890 mg/kg), Al (360 mg/kg), Mg (200 mg/kg), and Ca (3,110 mg/kg) were higher for the modified protocol compared to those from the standard protocol (Zn: 330 mg/kg, Fe: 850 mg/kg, SO_4: 2,320 mg/kg, Al: 265 mg/kg, Mg: 140 mg/kg, Ca: 1,030 mg/kg). Using Zn as a marker for oxidation rates, one can see that the modified protocol generates only 1.4 times more Zn than the standard protocol (HC-5,6) while the modified protocol generated 6 times more Zn when the sample was initially installed in a dry state (HC-1, 2; Table 1).

This difference is probably related to the sample behavior in the HC-1 as opposed to its behavior in HC-5. It was observed that as tailings A in HC-1 (under the standard protocol) dried during the testing period (Fig. 3a), it

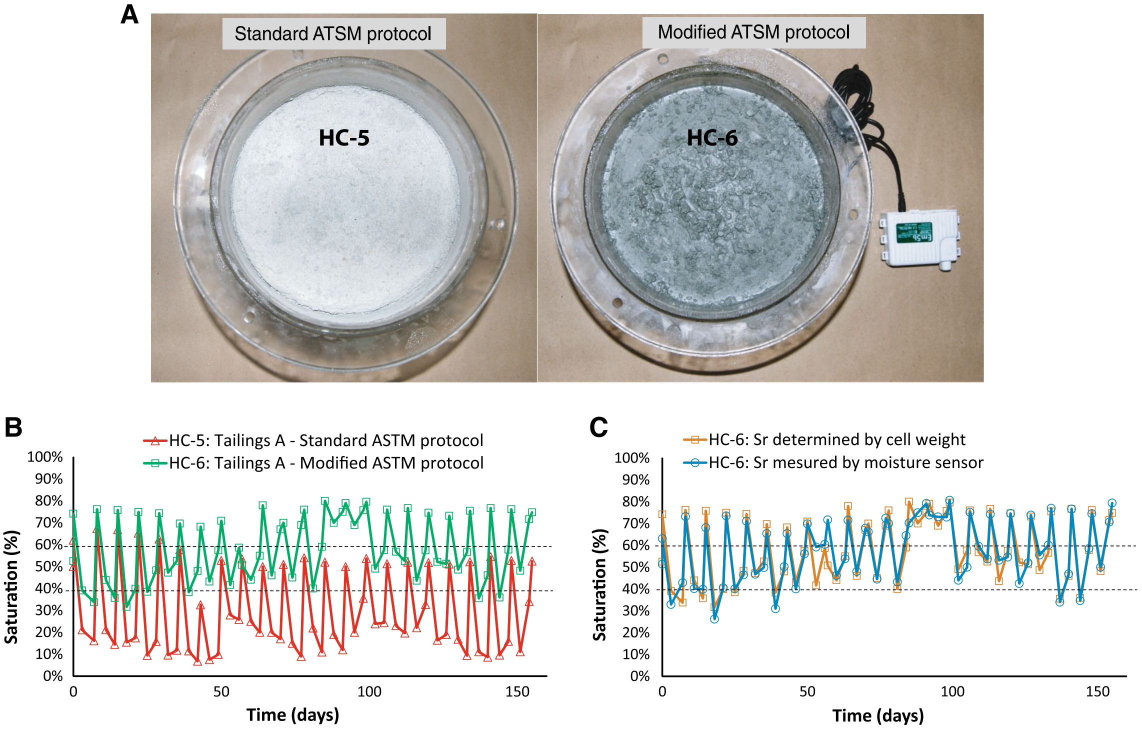


Fig. 5 Standard and modified HC comparison in tailings A: **a** Pictures showing the difference in sample moisture under standard and modified HC protocol (*darker color* (HC-6) is related to the higher water content). **b** Saturation profile corresponding to the modified and standard protocol. **c** Saturation profiles determined by HC weighing and moisture sensor in HC-6

gradually became hydrophobic between weeks 4 and 6. In fact, it was noticed at the end of every moist cycle that a thin film of water droplets accumulated on top of the sample surface (Fig. 7b). Throughout the test, the sample in HC-1 became increasingly dry and retracted from the cell wall; shrinkage gradually increased (Fig. 7a). Consequently, and as observed, water flowed between the sample and the cell wall without contacting all of the tailings, and therefore was less loaded with oxidation products. The rinse water exited the cell faster than in the cell using the modified protocol.

Influence of the Combined Sample Thickness and the Weekly Dry–Wet Cycles on Sample Reactivity

Maintaining sample saturation in a fixed range of values for a better reactivity requires incessant operator intervention during the kinetic test, which can be expensive and time-consuming. To assess the sample thickness influence on its weathering behavior if it can avoid protocol modification and operator intervention, cells having dimensions of 10.2 cm ID by 20.3 cm height, usually required for the coarser samples, were used. The tailings used for this investigation were tailings A (HC-7/HC-8) and B (HC-9/HC-10), installed with initial thickness and S_r of 5 cm and 50 %, respectively. For each sample, the kinetic test was performed with two cells: the first cell followed the standard protocol, while the second one followed a modified protocol. The experimental procedure of both protocols was the same as for HC-1 to HC-4 with the same sample weight.

Figure 8 shows the S_r evolution during the kinetic tests. For both modified HC (HC-8 and HC-10) in both A and B tailings, S_r was maintained between 40 and 60 %, while it was around 10–30 % in the standard HCs (HC-7 and HC-9) over the testing period. Saturation curves for HC-8 determined by moisture sensor and by HC weighing are similar. Figure 9 summarizes the leachate's chemistry obtained from each cell and shows that:

Tailings A: Leachate characteristics from the modified and standard protocol are similar. The average values for pH (2.7), Eh (628 mV), conductivity (3,826 µS/cm), and acidity (913 kg $CaCO_3$/mL) recorded from the modified protocol are similar to those of the standard protocol (pH 2.8, Eh 609 mV, conductivity: 3,374 µS/cm, acidity: 780 kg $CaCO_3$/mL). Using Zn as a tracer for oxidation rates, the standard protocol (HC-7) generated similar Zn loads (cumulative value: 303 mg/kg) to the modified protocol (cumulative value: 338 mg/kg), as seen in Table 4.

Tailings B: There are no differences between the standard and modified HC protocol results (Fig. 9). The Eh,

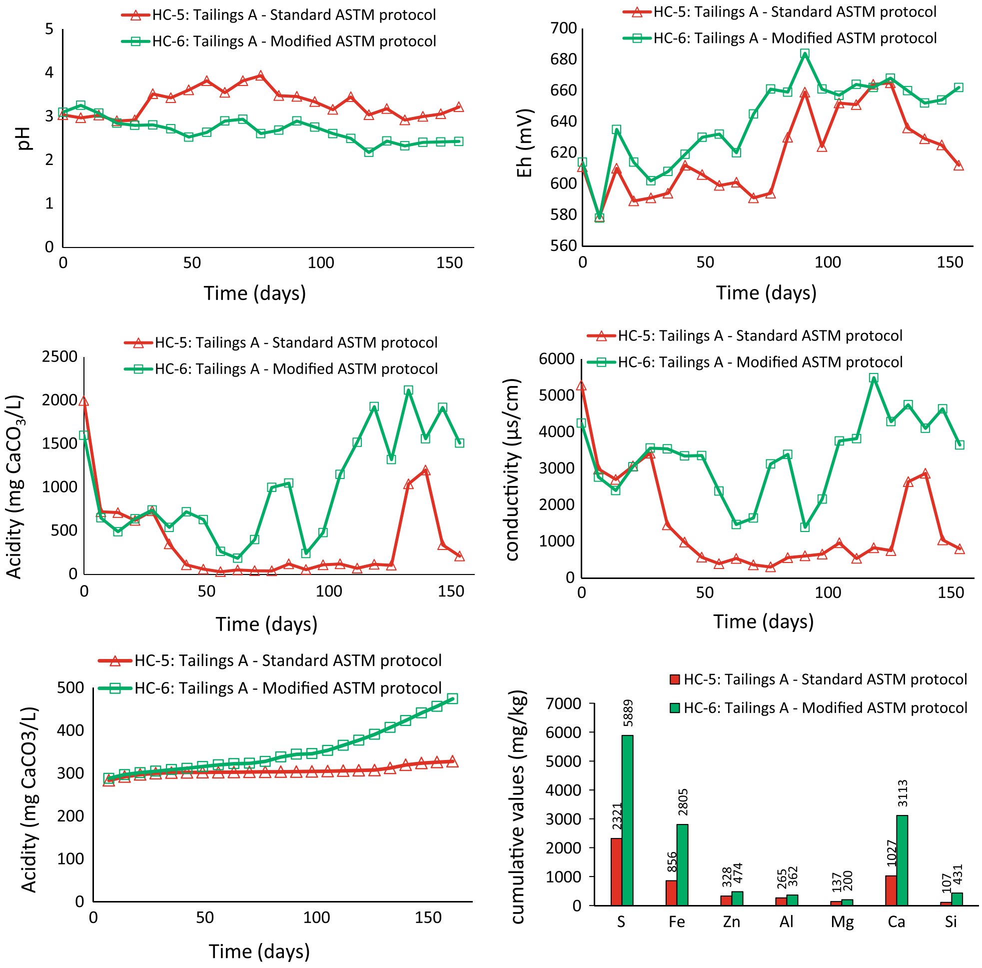


Fig. 6 *Tailings A* evolution of pH, Eh, conductivity, acidity and total cumulative leached SO_4^{2-}, Fe, Zn, Al, Mg, Ca, and Si released over a period of HC testing

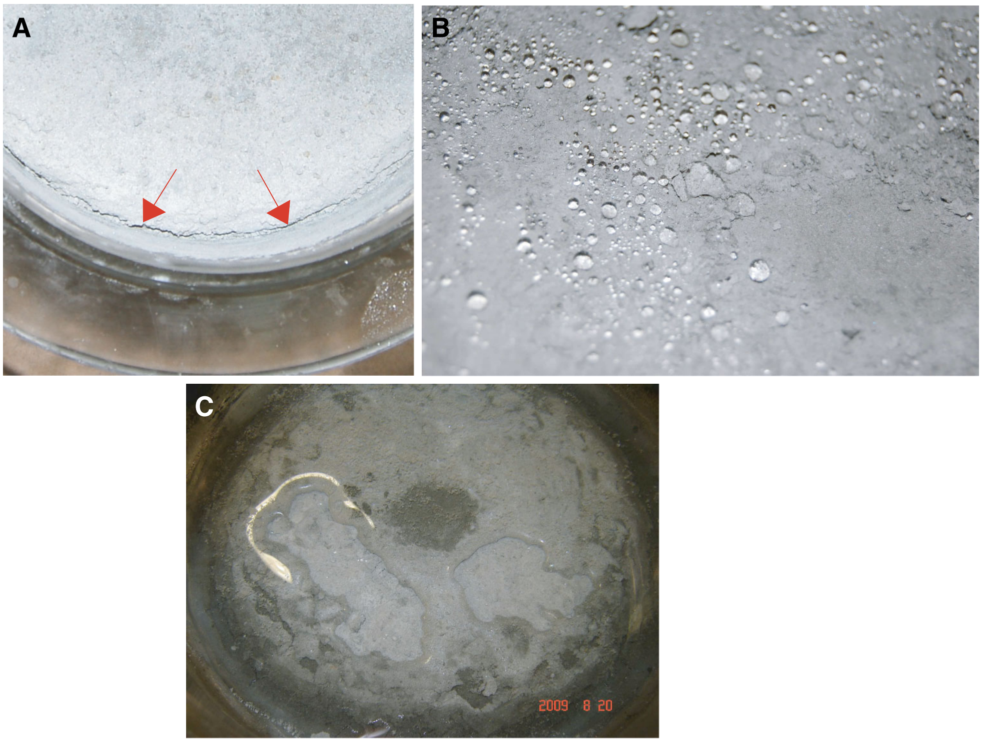
pH, conductivity, acidity, alkalinity, and dissolved metals show the same trends between the modified (pH 7.5, Eh 491 mV, conductivity: 735 µS/cm, alkalinity: 48 kg $CaCO_3$/mL) and standard protocol (pH 7.6, Eh 482 mV, conductivity: 728 µS/cm, alkalinity: 48 kg $CaCO_3$/mL). The leachate Zn loads showed that the modified protocol generated similar cumulative Zn loads (2.3 mg/kg) to the modified protocol (2.8 mg/kg), as seen in Table 4.

For both tailings A and B, the same characteristics of the leachates between the standard and modified protocol suggest that increasing sample thickness in the HC can enhance the sample reactivity, thereby reducing errors related to the standard ASTM protocol (as seen with HC-1 and HC-5). Despite these satisfactory results, it was observed during the testing period that the rinse water could not easily percolate through the entire thickness of the sample in the HCs. Consequently, it was necessary to apply air pressure above the sample overnight to recover the 500 mL of flushing water. This air pressure caused compaction of the sample, which increased with each HC cycle, and caused a gradual increase in percolation time. It was necessary to disaggregate the sample in both HC (standard and modified protocol) every 3 or 4 rinses to facilitate sample flushing to provide a maximum leaching rate. Since the sample was thicker, its bottom part was always moister than the upper part; then, the disaggregation mixed the moist lower part with the driest upper part, bringing the entire sample to a saturation value that promoted its reactivity, even in the standard protocol. This influenced the results of both protocols, leading to their similarity, even if saturation between the standard and modified protocols was different. Tailings B (HC-9 and HC-10) showed less resistance to water percolation, probably due to it sandy nature.

Summary and Conclusions

Previous laboratory work showed that results of humidity cell kinetic test can be influenced by factors that may have a significant impact on leaching rates and may lead to erroneous acid-generating potential prediction. The main

Fig. 7 Behavior of tailings A under standard HC test (HC-1). **a** sample shrinkage, **b**, **c** sample hydrophobicity to the rinse water



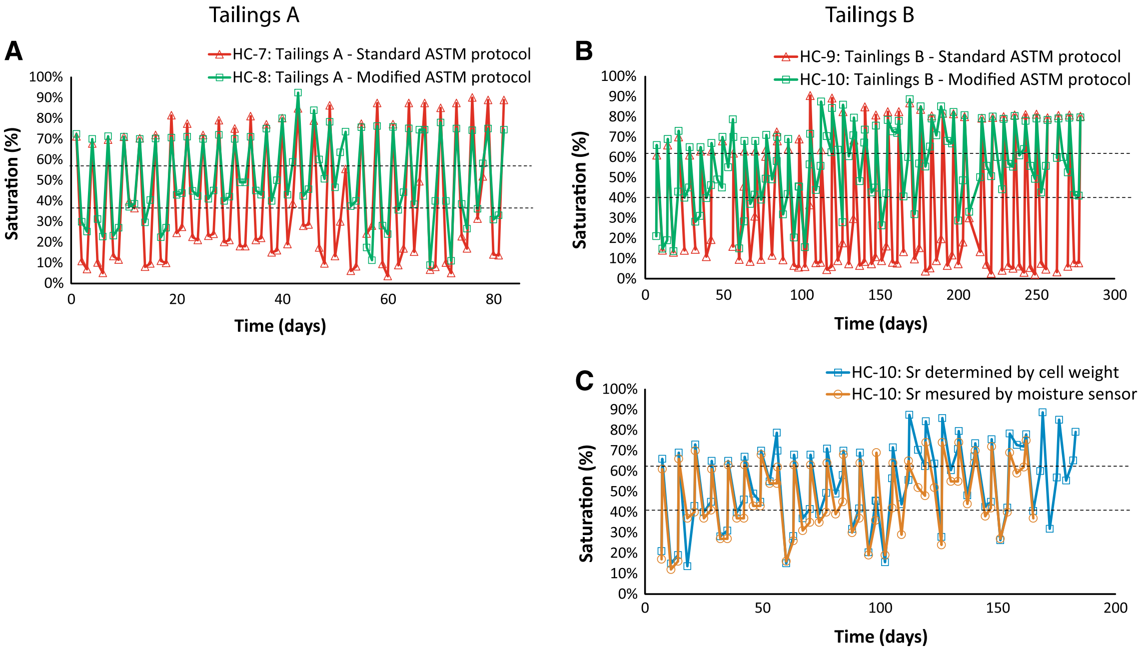
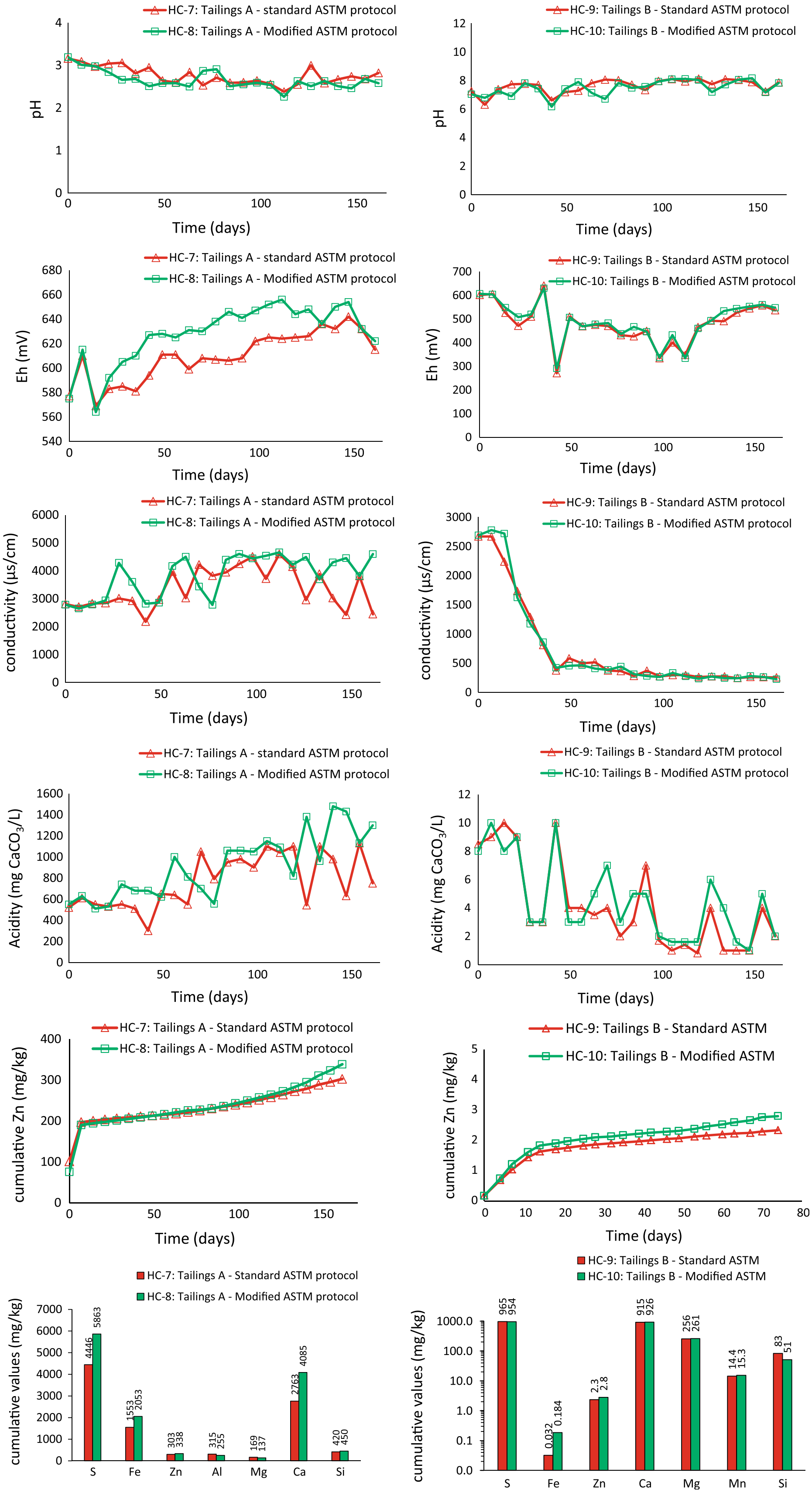


Fig. 8 Saturation profile corresponding to the modified and standard HC protocols (**a**, **b**). **c** Saturation profiles determined by HC weighing and moisture sensor in tailings B

objective of this study was to evaluate the effect of protocol modification on the ASTM humidity cell results. The main protocol modification consisted of keeping the two different mines tailings at a continuous degree of saturation between 40 and 60 %, where sulfide oxidation was optimal. The influence of the initial sample porosity and thickness was also investigated. For each parameter, two HCs were set up for each tailings sample. The first one followed the standard ASTM protocol and the second one followed the modified protocol.

Fig. 9 Evolution of pH, Eh, conductivity, acidity and total cumulative leached SO_4^{2-}, Fe, Zn, Al, Mg, Ca, and Si released over a period of HC testing



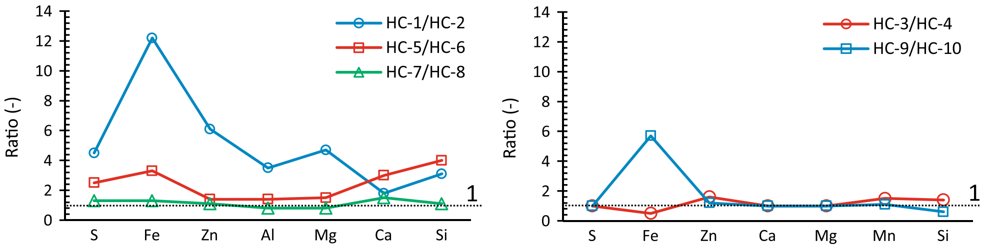


Fig. 10 Ratios of cumulative values of main dissolved elements from all standard and modified HC configuration set ups

Sulfide oxidation rates with the modified ASTM protocol were several times greater than with the standard protocol. The modified protocol created conditions that were more favorable to sulfide oxidation due to the moisture content, which was maintained at an optimal level for oxidation. Figure 10 shows the ratios of cumulative S, Fe, Zn, Al, Si, Ca, Mg, and Mn loads recovered from all of the HC tests. For tailings A, when installed dry, the ratio was high, indicating a noticeable difference in reactivity of samples between the standard and modified protocols. The difference in reactivity of samples submitted to the standard and modified HC protocols decreased when the sample was installed at 50 % S_r and compacted, or when the sample was installed thicker. However, tailings B was less sensitive to these parameters and the ratio was quite similar for the standard and modified protocols, regardless of initial set up conditions. The very different behaviors of tailings A and B relative to the HC protocols can be attributed to sample reactivity and mineralogy. In fact, tailings B is sandy, coarse-grained and not very reactive, and became uniformly humidified after each flush. In contrast, tailings A is highly acid-generating and the difference between standard and modified protocols was sometimes very noticeable, depending on HC protocol and set up. This difference is attributed to sample shrinkage when submitted to the standard protocol and to its hydrophobic behavior, probably due to its fine particle size distribution and silty/clayey nature.

Based on the results obtained in this study, the best protocol and HC dimension for concentrator tailings that allow a maximum reactivity and a more conservative AMD prediction is:

- to install the sample (≈1 kg) with 50 % saturation,
- with a porosity representative of the one expected in the field, with the typical values for tailings from hard rock mines being ≈0.47.
- The 1 kg sample must be compacted into the HC (20.3 cm ID and 10.2 cm height) to obtain the targeted porosity (see Bouzahzah et al. 2012 for more details).
- The sample must be maintained within an optimal range of saturation values for better sulfide oxidation (between 40 and 60 %) throughout the test.

This study showed that keeping the degree of saturation of the tailings within a range of 40–60 % can overcome erroneous results related to excessive sample drying during the dry period of the kinetic test. However, maintaining optimal sample saturation requires daily operator intervention to verify the saturation and to manually add water when the sample begins to dry out. The time and expense of these tasks can be minimized by automation with a controller, a data logger, a moisture sensor, and a computer (see Bouzahzah et al. 2012).

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