Modification and Automation of the Humidity Cell Test Protocol to Favor Tailings Reactivity

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Abstract

Previous laboratory work showed that the standard ASTM humidity cell (HC) kinetic test can be influenced by several factors that have significant impact on sulfide oxidation. This could lead to different leaching rates and, in some cases, to different acid mine drainage interpretations and predictions. In this study, modifications to the standard HC protocol are discussed. Mine tailings with a high acid-generating potential was submitted to both standard and modified ASTM HC kinetic test for a period of20 weeks. Keeping samples permanently at a degree of saturation between 40% and 60 % was the main protocol modification,a range where tailings are the most reactive, as demonstrated in previous work. Results showed that under the modified ASTM protocol, sulfide oxidation rate was different by several orders of magnitude compared to the standard protocol. But, maintaining tailings degree of saturation in a range of 40-60% during the entire test requires daily operator intervention which is expensive and timeconsuming. This regulation can be automated with the use of a controller, a moisture sensor and a computer. The setup of the automated saturation control system and its operating mode is presented in this paper. The system can simultaneously control several humidity cells.

Key Words: Acid mine drainage, prediction, ASTM humidity cell,

Introduction

Most of the active and closed mine sites are surrounded by solid wastes stored as waste rock piles and tailings impoundments which can contain significant proportions of sulfide minerals. When exposed to atmospheric conditions, thesesulfidic materialsmay undergooxidationproducing acid mine drainage (AMD)which represents a major source of acidity and metalsfor the surrounding environment (Kleinmann *et al.*,1981).Static tests have often been used to assess,as a screening step,the acid-generating potential (AGP), but sometimes, they are unable to assess the long-term AGP (due to a large uncertainty zone, Ferguson and Morin, 1991). For these uncertain materials, kinetic tests are commonly used to provide a direct measurement of acid generation, neutralization rates, and long-term AGP.

Column and humidity cell tests are the most commonly used kinetic test types to predict AGP and the lag time to AMD onset. Theyare based on sample alteration under laboratory controlled conditions simulating relatively intense weathering conditions (Bowell *et al.*,2006; Frostad *et al.*,2002; Lapakko and White ,2000; Lawrence ,1990; Priceand Kwong,1997; Sapsford *et al.,* 2009). Previous laboratory works had shown opposite results on AGP prediction for the same tailings depending on the type of kinetic test used. Benzaazoua *et al.*, (2008) noticed a certain reactivity inhibition when tailings sample from Doyon mine (Québec) were submitted to the ASTM standard normalized humidity cells.These authors reported that the pH of leachate remained neutral over 364 days corresponding to 52 cycles (Figure 1)whereas Demers *et al.,*(2008) noticed that the same sample submitted to column kinetic test became acidic after 380 days, which corresponds to only 10 cycles, as showed in Figure 1.

Moreover, Sapsford*et al.*, (2009) reported a difference in the calculated sulfate release rate (mg/kg/cycle) from a crushed waste rock submitted in duplicate to ASTM humidity cell tests. They concluded that the aeration system malfunctioned; as a result, one cell became much drier than the other during the test, depriving the waste rock of interstitial water requiredfor pyrite oxidation. Bowell *et al.*, (2006) had observed the same phenomenon when analyzing weathering of Volcanogenic

Massive Sulfide material on duplicate aerated HCs. The difference in sulfate release rates between the two HCs were attributed to an excessive drying in one cell due to an increase in air-flow through it.

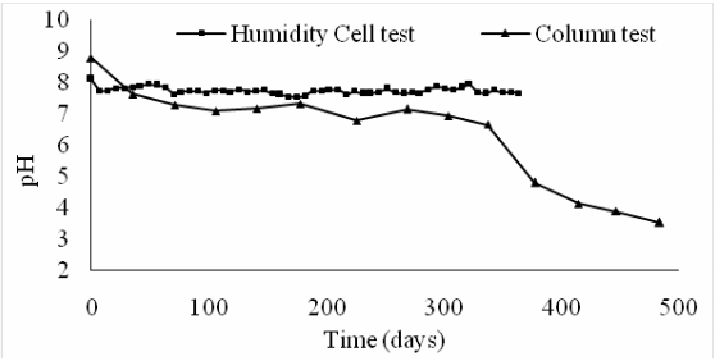


Figure 1. Evolution of leachates pH for the same sample submitted to column and humidity cell kinetic test (Data from Benzaazoua *et al.*, 2008; Demers *et al.,* 2008).

Frostad*et al.*, (2002) studied 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 for samples from the same mine waste. Their data have shownthat the standard humidity cell creates an unnatural oxidizing environment due to its extreme wetting and drying cycles, and therefore produced inconsistent results.

Based on theseobservations and findings, we have made a modification to ASTM HC kinetic test (ASTM D5744-07, option A, 2007) protocol to prevent the excessive drying and wetting of the sample and to overcome misinterpretation of the AMD (Bouzahzah *et al.*,2010; 2012). We focused on the ASTM HC, the only kinetic test normalized by the American Society for Testing and Materials (ASTM) and this method is widely used for AMD prediction (Sapsford *et al.,* 2009). In our testing, the same concentrator tailings were submitted to two HC Kinetic tests, a standard ASTM protocol and a modified protocol. In the modified protocol the sample was kept within a given range of saturation (40%-60%) by manually adding deionized water when required. Both protocols were compared with respect to S_r, cumulative dissolved ions (mg/kg/week), acidity and conductivity (Figure 2). Data shown in figure 2 indicate that sulfide oxidation rate was significantly affected using ASTM protocol as compared to standard protocol.

Evolution of the degree of saturation (S_r) inside the samples placed in two humidity cells are shown in Figure 2A. As indicated, in the modified protocol, $S_r$was maintained between 40% and 60% creatingfavorable conditions for sulfide oxidation. When using standard protocol over 24 weeks for testing, (S_r) decreased progressively to reach zero by the end of the procedure.

To maintain an optimalS_r of tailings during a kinetic test, a daily maintenance of an operator is needed in order to visually check the saturation and manually add water whentailings begin to dry. This task isexpensive and time consuming. Therefore, the need of protocol automation is necessary.

In this paper we are presenting an automated control system for saturation.This set-up is equipped with a controller, a data logger, a moisture sensor and a computer for data monitoring and recording. The sample installation in the HC, instrumentation and operating mode of the controller are described.

Materials and methods

Materials

The tailings sample selected for this study comes from the Manitou abandoned mine site located approximately at 15 km southeast of Val-d'Or (Abitibi, Canada). Manitou mine was operated from

1942 to 1979 for zinc and copper beneficiation. Presently, there are 12 million tons of tailings stored above ground and one million tons underground. Tailings recognized as being highly acid generating cover a surface of about 180 hectares. The mine tailings were sampled in the non-oxidized zone into the impoundments (1 m deep). The sample was placed immediately in a container and flooded with deionized water to prevent sulfide oxidation during transport and lab storage.

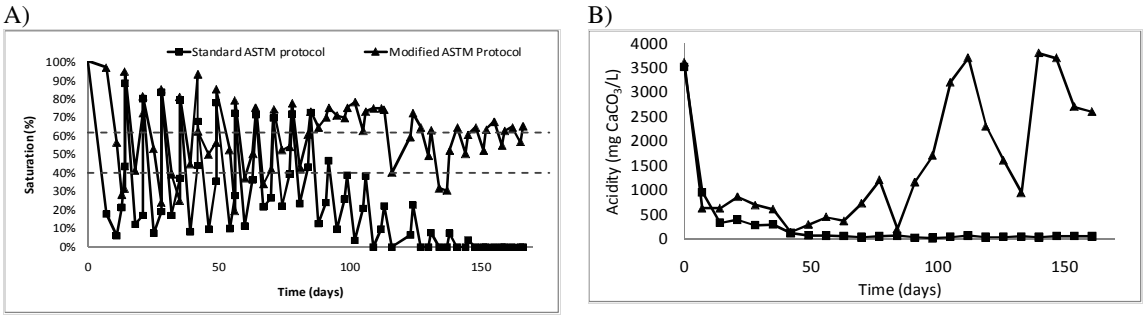


Figure 2. A) Saturation profile in two humidity cells. Two horizontal lines represent the targeted S_r values (between 40% and 60%). B) Acidity release after 160 days of kinetic test.

Methods

Material characterization

Tailings specific gravity (G_s) was determined with a Micromeritics helium pycnometer. Particle size distribution was determined using a Malvern Mastersizer laser particle size analyzer. 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 corresponding data are not reported in this study. Sulfide sulfur was determined by subtracting the sulfate sulfur (determined by 40% HCl extraction; method adapted from Sobek *et al.*, 1978) from the total sulfur (ICP-AES analysis). Analysis of both total sulfur and total inorganic carbon was performed by induction furnace (ELTRA CS-2000).

Sample mineralogy wasdetermined by optical reflected light microscopy on polished sections preparedwith the bulk samples and an epoxy resin. Scanning electron microscopy (SEM) observations using backscattered electrons (BSE) for mineral identification were made on a Hitachi S-3500N microscope equipped with an X-ray energy dispersive spectrometer (Silicon drift spectrometer X-Max 20 mm²) with INCA software (450 Energy). Sample mineralogy was also analyzed with X-ray diffraction (XRD) using a Bruker A.X.S. D8 advance X-ray diffractometer equipped with a copper anticathode, and allows angular scanning over a diffraction angle (2θ) range from 5°to 60°. The absolute precision of this quantification method is around1% (Raudsepp and Pani, 2003).

Acid-base accounting(ABA) tests were conducted using Sobek *et al.*, (1978) test modified by Lawrence and Wang, (1997). The acid-generating potential (AP) was calculated assuming that the sulfide sulfur content was exclusively expressed as pyrite and that all pyrite was available for oxidation, which is the case for the studied tailings. The neutralization potential (NP) was determined by HCl addition followed by back-titration of the excess acid to a pH 8.3 endpoint. The net neutralization potential (NNP) is calculated as the difference between the AP and NP (NNP = NP-AP) (Miller *et al.*, 1991)

Kinetic test

The following section describesstep by step humidity cells kinetic test procedure.

- *Humidity cells apparatus*:

The kinetic testwork apparatus and procedures were based on the humidity cell device and protocols given in ASTM D5744-07 (option A, 2007). HC is a chamber made of Plexiglas that provided air

input and airoutput over the solid wastes. Cells have an inside diameter of 20.3 cm and a height of 10.2 cm. The sample is placed on a perforated plate covered with two geo-textile layers (as filter) to retain sample (Figure 3-A B).

- *Moisturesensor installation:*

The wall of the HC must be perforated 1.25 cm above the perforated platelevel (Figure 3-A).The hole should have a diameter of 3.5 mm and equal to the diameter of the probe connector (Figure 3-B&C). The sensor cablemust be insertedfirstfrom the inside of the HC.Asthe hole islarger than the sensor cable, it must be filled withsiliconon both sides ofthe HC wall. The sensor must be placed vertically (Figure 3-D&E) to prevent water accumulation on it which may overestimate water content. Before installation, the sensor *Decagon ECHO EC5* must be calibrated,using the studied sample, following manufacturer instructions for volumetric water content (θ %)determination.The sensor determines the dielectric constant or permittivity(K_{ab}) by measuring the period of charge of the soil (capacitor); the K_{ab} is related to the volumetric water content of a soil (Maqsoud *et al*., 2007).

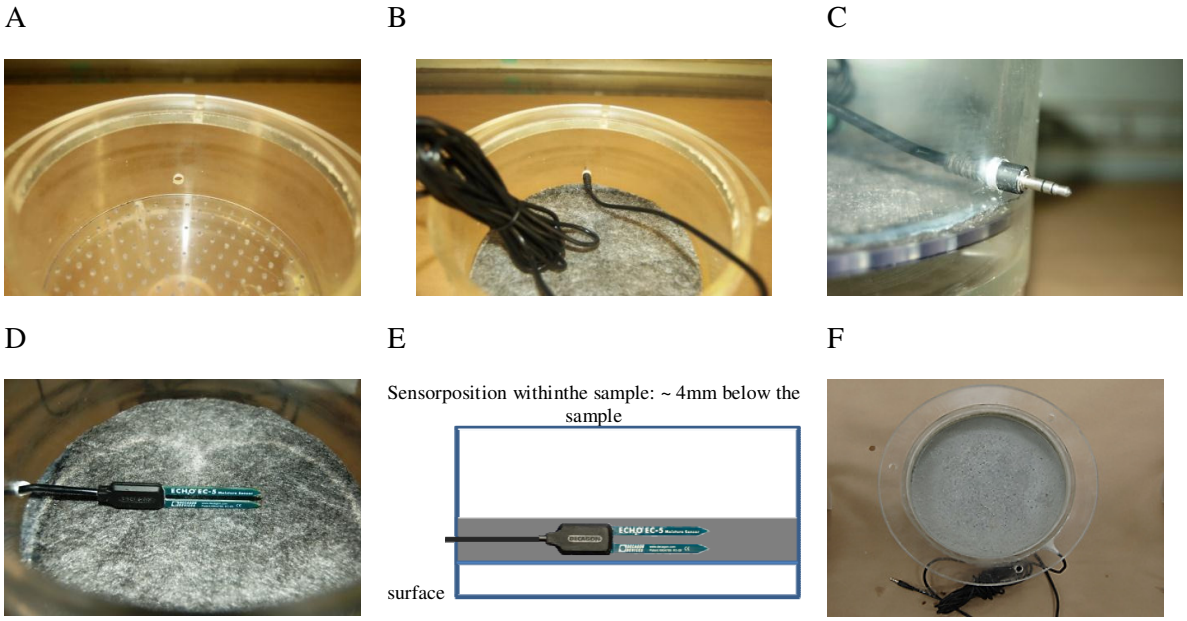


Figure 3.Humidity cellset up and water content sensor mounting.

- *Sample placement into the HC*

The sample must be thick enough to completely cover the probe(Figure 3-F) and,ideally, the material would have a void ratio (e) representative ofthe one expected in the field (see Bussière, 2007 for typical range). The following example shows how the dry masse (M_s) of the sample could be calculated to be set up with a thickness (h) of2.5 cm, a diameter (d) of 20.3 cm, a targeted porosity of 0.47 (typical values for tailings from hard rock mines; Demers *et al.,*2011).Thespecific gravity (G_s) of Manitou tailings is 3.11 g/cm^3.

$$M_s=[\pi\, d^{\,2}\,(h/4)\,(1\text{-}n)]G_s \quad (1)$$

In order to ovoid desiccation that leads to shrinkage and channeling during the kinetic test, Bouzahzah *et al.,* (2012) recommended installation of a sample in the HC with 50% initial degree of saturation. The water weight (M_w) to be added to the dry mass (M_s) to obtain 50% saturation is given by the equation 2:

$$M_w = M_s\,(W(\%)/100) \quad (2)$$

Where:

$$W(\%) = (e\ S_r)/G_s \quad (3)$$

$$e = (n/(1\text{-}n)) \quad (4)$$

n is the total porosity.

After mixing the calculated dry mass (M_s) of the sample with the calculated mass of water (M_w) to ensure sample saturation of 50%, the sample is placed in the HC and compacted tohave a thickness (h) of 2.5 cm..

- *Standard HC set up*

Tailings from Manitou wereplaced in three HCs: for the first cell (HC-1), the standard ASTM protocol was followed, while in the two other cells(HC-2 and HC-3,as duplicate), thetests were performed followingthe modified protocol. Table 1 summarizes setup details. The standard ASTM HC test protocol consists of a weekly drying-wetting cycle to ensure sulfide oxidation reactions. The first 3 days correspond to the dry period during which dry air is blown over the sample (1 to 10 L/min). The next 3-day period is the "wet" phase of the test where air is pumped through a humidifier unit supplying water-saturated air into the cell. The temperature of the water in the humidifier was maintained between 25 and 30°C during the test to keep 99 % air moisture (ASTM D5744-07, 2007). On the seventh day, the sample is flushed by adding 500 mL of deionized water for a contact time of 3 to 4 hours, after which the water is drained.

Table 1. Summary of kinetic test protocols

Description	HC-1	HC-2	HC-3
Cell diameter (d cm)	20.3	20.3	20.3
Cell height (cm)	10.2	10.2	10.2
Sample weight (Ms g)	1330	1330	1330
Sample height (h cm)	2.5	2.5	2.5
Initialdegree of saturation (S_r%)	50	50	50
Porosity (n)	0.477	0.477	0.47
ASTM protocol	Standard	Modified	Modified
Air flow (L/minute)	1 to 1.5	>2	>2
Dry air (days)	3	Dry and moist air when needed	
Moist air (days)	3		
Rinsing water volume (ml)	500	500	500

- *Modified HC set up*

HC protocol was modified by keepingthe sample S_r values at an optimal degree of saturation, 40% and 60%, allowing optimal sample reactivity (Bouzahzah *et al.,*2010).Initially the sample was placed in the HC at a degree of saturation of 50% and then maintained in the targeted$S_r$range (*Min* and *Max*) using a controller and a moisture probe. The saturation control automated system can simultaneously control up to 8 humidity cells.Figure 4 shows the setup of only one controlledcell. The operating mode is described hereafter:

- ❖ Starting test by rinsing the sample with deionized water. After rinsing, the tailing is fully saturated.
- ❖ The controller (Pico GFX-70) reads continuously the voltage (mV) coming from the moisture probe (ECHO EC-5).
- ❖ When saturation is at its *Max* value, the controller opens the valve to allow the dry air passing (default mode).

- ❖ Tailing's saturation gradually decreases until it reaches the threshold value ($MinS_r$). Then, the controller switches ONthe solenoid valve to allow moist air passing.
- ❖ By receiving the moist air, the tailing's saturation increases and when it reaches the *Max* S_r, the controller allowsthe dry air passing by switching OFFthe solenoid valve.
- ❖ Throughout the week, the controller sends dry or moist air to the tailing through the solenoid valve depending on tailing's saturation to keep the sampleatthe targeted saturation values.
- ❖ The data logger records all data with a recording interval fixed by the operator. Data are displayed and stored in a datafile by the computer. The displaying and recording software was especially developed for this study.
- ❖ The recorded signal (mV) must be converted to volumetricwater content(θ %) by equation5, and then, the degree of saturation S_r is calculated by equation 6.

$(\theta\%) = ax+b$ (5)

Where x (mV) is the recorded voltage from the sensor probe. (6)

S_r (%) = (θ/n) x 100

- *Leachates analysis*

Leachate solutions collected from the HCs were weighed and analyzed for the main geochemical 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 Accu·pHast electrode which was calibrated with commercial buffer solutions (pH 4, 7 and 10) before each set of measurements. Redox potential (Eh) was measured using a platinum ORP probe and corrected to standard hydrogen electrode (SHE). Conductivity of the samples was measured using an Accumet electrode. Acidity was determined using standard titration techniques with NaOH at 8.3 pH end point (APHA, 1995).

Results

Material characterization

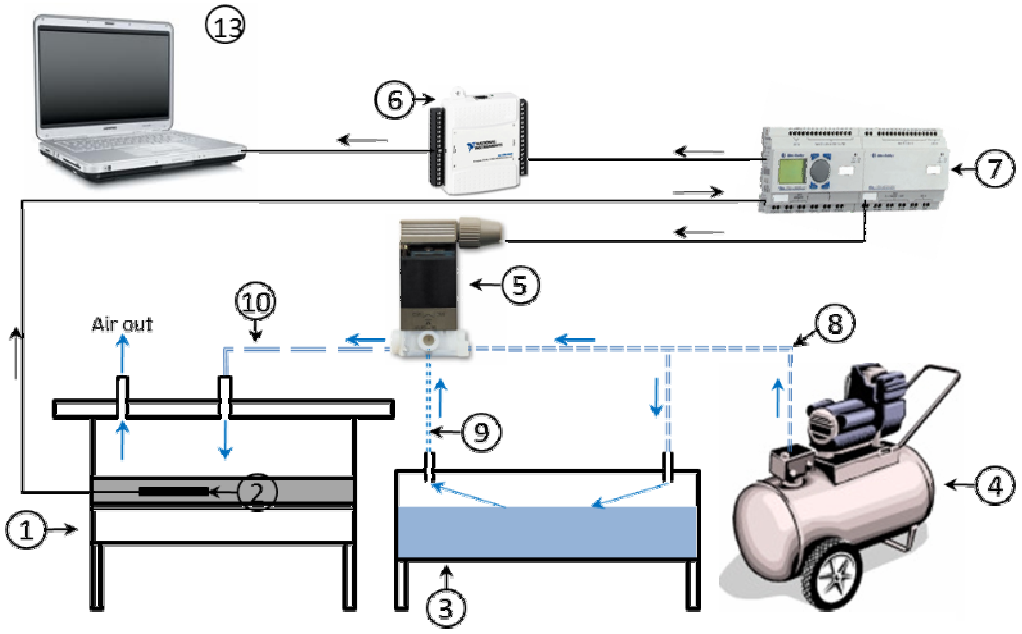
Physical, chemical and mineralogical characteristics are summarized in Table 2. The grain size parameters D_{10}, D_{50}, D_{80} and D_{90} are typical of hard rock mine tailings (Aubertin*et al.*, 2002; Bussière, 2007). Pyrite is the only sulfide detected by XRD analysis (20 wt.%), gangue minerals were mainly composed of quartz (44.3 wt.%) and muscovite (22.6 wt.%), with minor proportion of albite and chlorite at 6.7 and 3.8 wt.%, respectively. Gypsum (2.5 wt.%) is also present and possibly occurs as a secondary mineral. Sphalerite, chalcopyrite, magnetite and rutile were identified as trace phases byreflected light and electronic microscopy observations (Figure 6). As indicated in Table 2, no mineral with neutralization potential (NP=0) is present in the sample which had a negative NNP (NNP= - 401 Kg $CaCO_3$/t).

Table 2. Physical, chemical and mineralogical characteristics of Manitou Tailings

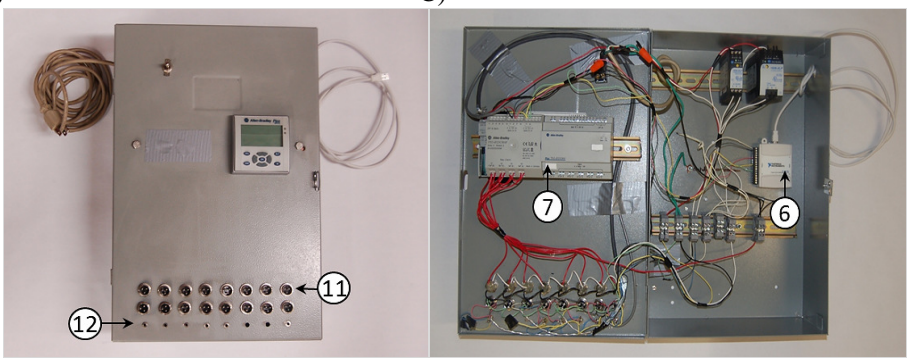
Gs (g/cm^3)	Granulometry (µm)		ICP-AES (wt.%)		Induction furnace (wt.%)		ABA test (Kg $CaCO_3$/t)		DRX (wt.%)	
3,1	D10	5,1	Al	5,17	S_{total}	13,99	NP	0	Quartz	44.3
	D50	24,3	Ca	0,74	C_{total}	0,06	AP	401	Albite	6.7
	D80	55,8	Cu	0,02			NNP	- 401	Chlorite	3.8
	D90	104,9	Fe	15,5					Muscovite	22.6
			Mg	0,73					Pyrite	20.1
			Mn	0,02					Gypsum	2.5
			Pb	0,09						
			Ti	0,05						
			Zn	0,51						
			S_{total}	13,5						
			$S_{sulfate}$	0,66						
			S_{sulfur}	12,84						

Acid-generating Potential (AP) = S_{sulfur} (wt.%)*31. 25; Neutralization Potential (NP) is determined by acid-base titration [method of Sobek*et al.*, (1978); modified by Lawrence and Wang, (1997)].

A)



B) C)



1- Humidity cell (HC)
2- Soil moisture probe (*ECHO EC-5*)
3- Humidifier
4- Air compressor
5- Solenoid valves (*Omega SV-1200*)
6- Data logger
7- Controller (*Allen-Bradley Pico GFX-70*)
8- Dry air
9- Moist air
10- Dry or moist air HC input
11- Solenoid valve connection
12- Soil moisture probe connection
13- Computer for data displayingand recording

Figure 4: A) Schematic representationof the automated system for the humidity cell saturation control. B-C) Photos of the system

Kinetic test results

Data in figure 7 shows the degree of saturation (S_r)curves of the sample inthree humidity cells (HC-1, HC-2 and HC-3). In this experiment, $S_r$was maintained between 40% and 60% by the controller when a modified protocol is used.When using standard protocol, $S_r$decreased progressively, until it became nil after 4 weeks of testing. From our observations, we noticed that sample in HC-1 was drier (lighter grey) than the samples in two HCs under modified protocol where samples were more moist and had a dark grey color (Figure 8). The excessive drying in the standard protocol is due to a progressive water loss caused by constant dry air-flow through the sample during three days of "dry air period". The humid period did not re-moist the sample.As shown in figure 7 a rapid fluctuation in sample saturation inHC-1 is observed because (i) addition of water is not controlled to allowcontinuous sample desaturation and (ii) the moisture probe is located ~4mmbelow the sample surfacedetecting near surface saturation changes (figure 3-E). In Bouzahzah (2012), the HC's saturationwas monitored by the moisture probe and by weighing the humidity cells (water loss and gain balance, 3 times a week). Saturation values were identical in bothmethods. In this present work, saturation was measured only by the moisture probe)

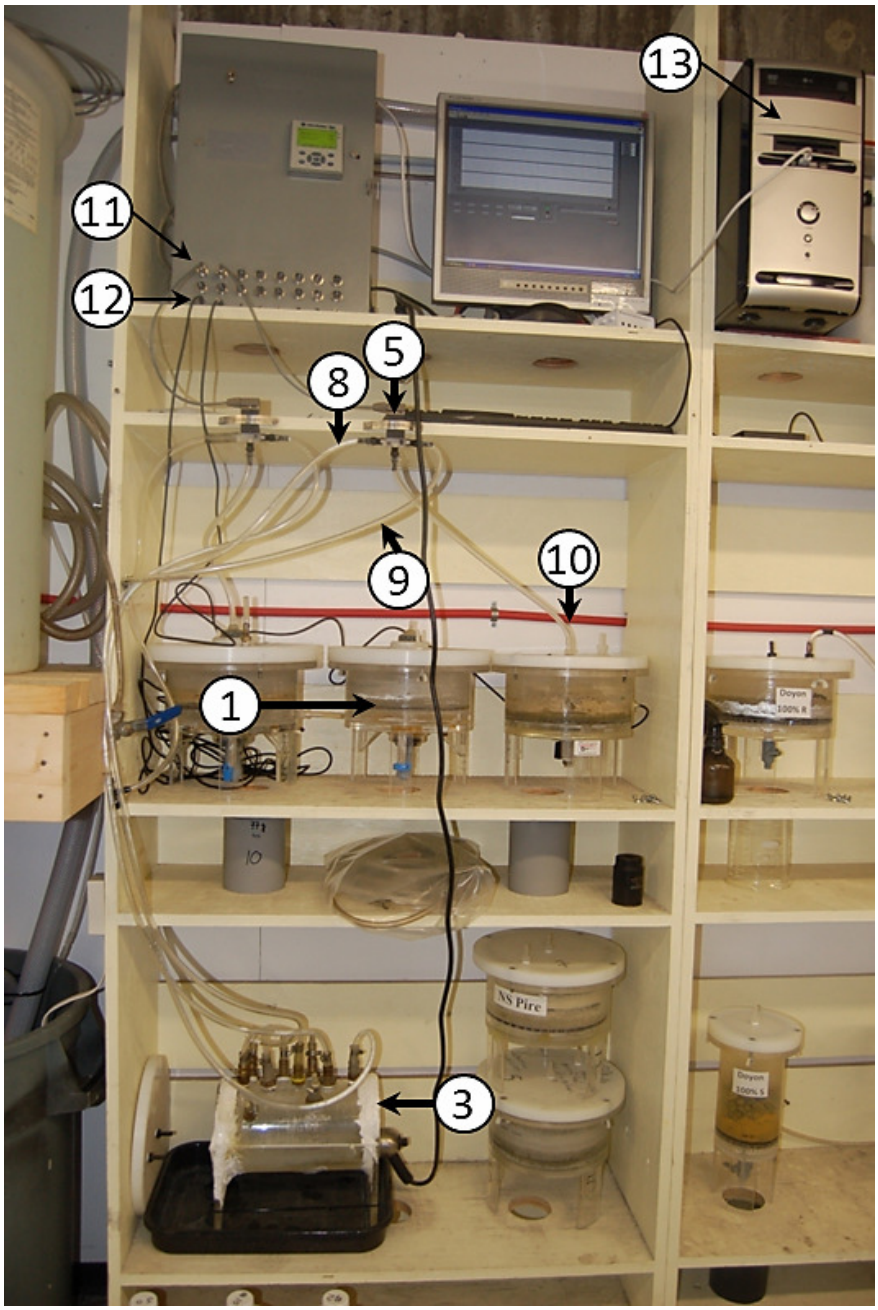
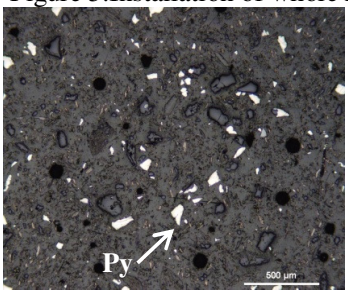
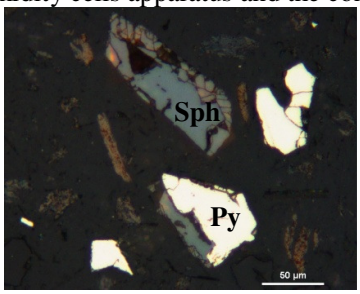
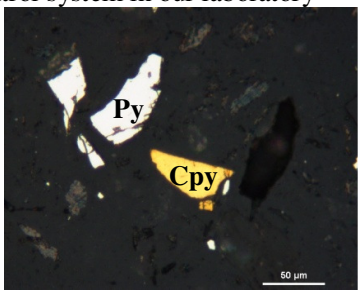


Figure 5.Installation of whole humidity cells apparatus and the control system in our laboratory

Optical microscopy







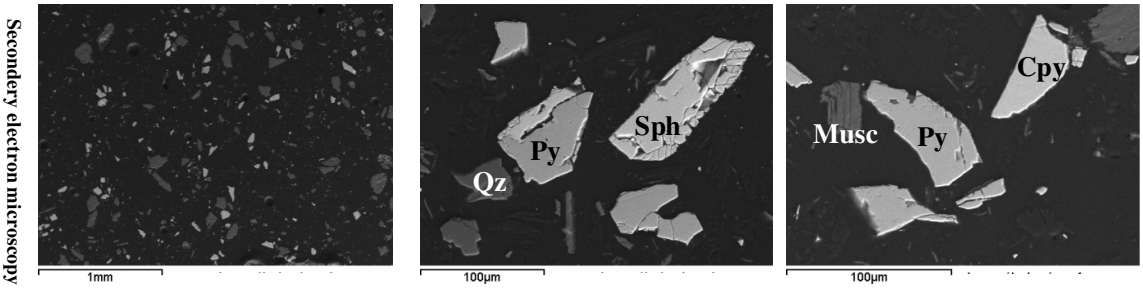


Figure6.Optical and scanning electron microscopic observations.Py: pyrite, Sph: sphalerite, Cpy: chalcopyrite, Qz: quartz, Musc: muscovite.

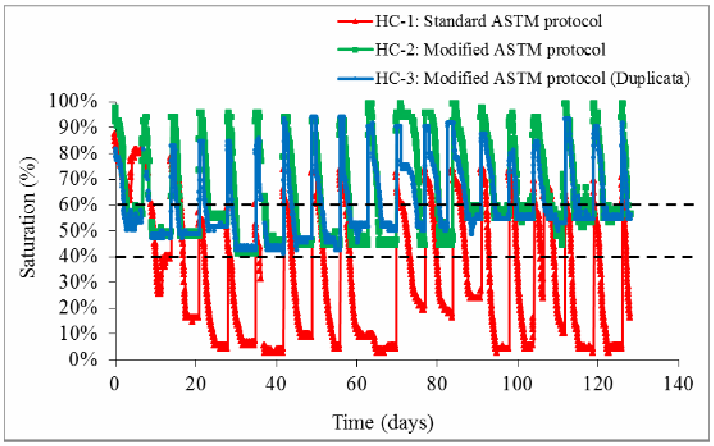


Figure 7.Evolution of saturation profilewithin the three humidity cell tests.

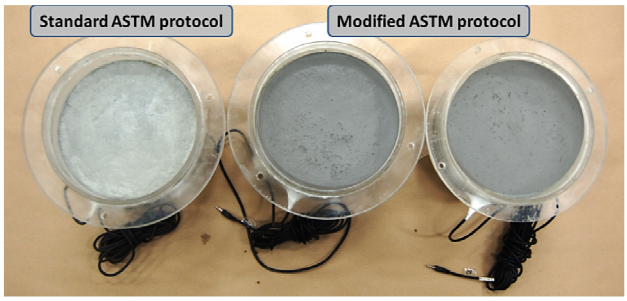


Figure 8.Sample under standard ASTM protocol is lighter grey than the dark grey moist material (darker color is related to the higher water content)

Figure 9 shows the pH, Eh, conductivity and acidity in function of time inthree cell tests.In this present work, the chemical analysis of leachates is not shown. As reported in previous work (Bouzahzah *et al.*, 2010; 2012), analyzing acidity and conductivity is sufficient to determine the difference of reactivity between standard and modified HC protocols.These two parameters, as indicated in figure 9, show similar curves as compared to the main dissolved ions.

Leachates have higher values of acidity and conductivity when using modified protocol which indicate that the concentration of total dissolved ions in the modified protocol was significantly higher as than total dissolved ions in the standard HC protocol.The lower pH and higher Eh of leachates recovered

from the modified HC are related to the higher pyrite oxidation rate for the targeted saturation conditions.

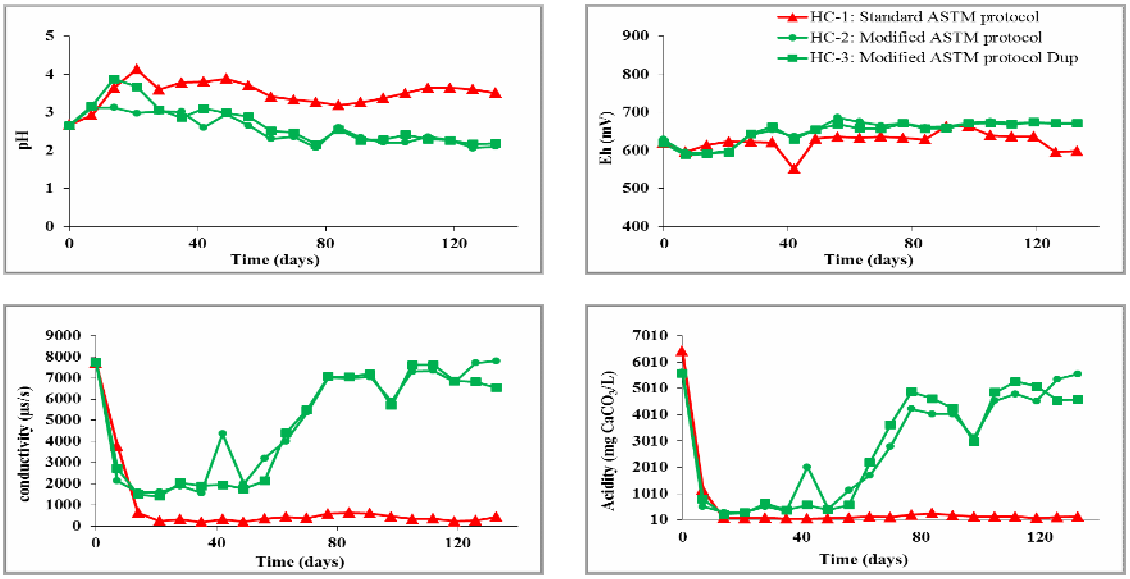


Figure 9.pH, Eh, conductivityand acidity after 20weeks of kinetic testing. The graphs demonstrate clearly the higher reactivity of tailings under the modified HC protocol.

Finally,our study indicate that reactivity of samples is higher when saturation was kept in a fixed range of values in a controlled system as compared to sample where saturation was manually controlled (Figure 10). Consequently, we conclude that an automatic saturation control is more efficient than a manual control.

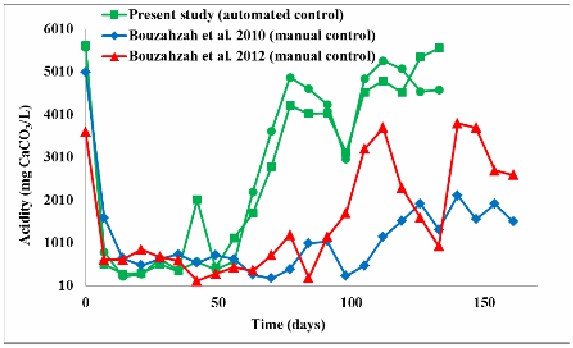


Figure 10.Effectiveness of automatic saturation control expressed by a higher acidity which reflects high tailings reactivity.

Conclusion

Previous laboratory work have shown that humidity cell kinetic tests can be influenced by some factors that may have a significant impact on leaching rates and can lead to misleading predictions of acid mine drainage. The authors previously showed that a modified ASTM HC protocol generated higher sulfide oxidation rates than the standard protocol by manually maintaining the S_r between 40 and 60 %. However, maintaining optimal$S_r$valuesduring the kinetic test requires incessant operator intervention,which can be expensive and time consuming. The objective of this study was to set up an automaticsystem to maintain sample saturation at a given range of values by using a controller, a data logger, a moisture sensor and a computer for data monitoring and recording. The setup was presented

with its operating mode. Results show that the modified ASTM protocol whenusing an automated control system had created conditions into HC that were more favorable to sulfide oxidation.The system was able to consistently maintain saturation in the targeted range of S_r values.

Acknowledgements

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