Humidity Cell Tests: Effect of Air Flow and Cycle Duration in the Quality of Leachates[1]

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ABSTRACT

A factorial experimental design was used for humidity cell tests conducted for 10 cycles on tailings containing 71% pyrite to determine if the rate of air flow (3 and 6 $Lmin^{-1}$) and cycle duration (14 and 21 days) affected leachate pH, electrical conductivity, and Eh. The tailings composite sample for the tests was taken from Tizapa, a massive sulfide mine located southwest of Mexico City, Central Mexico. The method ASTM D 5744-96 was taken as the basis for the design of the cells with two modifications: 1) air was circulated across the surface of the tailings and 2) water was dropped directly emulating rain on a tailings dam. Examining drainage quality data by cycles indicated the four treatments did not significantly affect the pH, but it was found that the air flow rate had a significant effect in the quality of leachates. The higher air flow delayed acidification, probably due to excessive water loss that inhibited oxidation reactions.

Key words: Massive sulfide ore, tailings, AMD prediction, kinetic tests, leachate quality, humidity cell

INTRODUCTION

Static and kinetic tests are commonly conducted to predict the acid generating behavior of mine wastes and classify these wastes based on the predictions. Static tests predict the quality of the drainage by comparison of the potentials of the sample to produce and neutralize acid. Kinetic tests take a larger volume of sample and time. They provide information about the rates of mineral dissolution and, therefore, about the acid production and quality of the drainage. The humidity cell test is the kinetic tests most widely used for predicting the quality of drainage from mine wastes. The development of this test can be traced back to 1962 with the application of kinetic tests to coal-mine wastes (Lapakko, 2003) and it was further adapted by Sobek et al. (1978). In 1996, the ASTM D 5744-96-Standard Test Method for Accelerated Weathering of Solid Materials Using a Modified Humidity Cell was approved (ASTM, 2000).

The Sobek humidity cell test, as well as the ASTM method for waste rock introduce air flow through the cell from the bottom and a cycle of seven days composed by two periods. First, a three-day dry period for dry air circulation through the tailing or waste bed and a second three-day period during which humid air is circulated through the waste. On the seventh day, the tailing or waste bed is leached with a fixed volume of water left in contact with the solids for one hour. Parameters such as pH, electrical conductivity, redox potential, sulfate and metals concentrations,

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etc., are determined in the resulting leachate. Modifications to the humidity cell test continue to be made based on additional research (Bucknam et al., 2009).

It has been reported that differences in protocol variables affect the leachate quality. Frostad et al. (2002) performed a comparative study using five kinetic test protocols (standardized humidity cells, non-aerated cells and others). These authors indicated that the standardized humidity cell creates an unnatural oxidation environment due to the extreme humidity and dry periods, thus producing erratic results. They further reported that non-aerated cells (but naturally ventilated) have an oxidation environment closer to natural conditions, produce more consistent results and therefore, better estimate the rate of sulfide oxidation in the field. Sapsford and Williams (2005) conducted a study based on the ASTM method and compared aerated and non-aerated cells. They found that variations in air flow affected the leachate characteristics, and that less time was required to reach the peak iron and sulfates concentrations in the aerated cells.

Climatic conditions must also be considered in extrapolation of humidity cell results to the field. Schwartz et al. (2006) concluded that humidity cells are adequate to estimate the quality of leachates in humid climates but may be inadequate for arid climates. This scope was considered in the previous job developed in our research group and described in the following section.

BACKGROUND TO THE STUDY

González-Sandoval (2006) used humidity cell tests to assess the effects of hydrologic variables (drought, rainy season duration, precipitation) on pyrite oxidation rate at the surface of a non-flooded tailings dam. The humidity cells were based on the Sobek et al. (1978) design. To study the effects of wet period duration, periods of 1, 4, and 7 days were considered and the test cycle duration was extended from the standard 7 days to 14 days. During the wet period, water was carefully dropped on the surface of the tailings instead of applying humidified air, emulating rain. The effect of the volume of water (10, 30 and 50 mL) added daily during the wet period on pH was also assessed. Air (1 $Lmin^{-1}$) was circulated over the surface of the 250 g-tailings samples. The cells were flooded on day 14 with 250 mL of deionized water for one hour; pH, electrical conductivity and other parameters were determined in the collected leachates. The experimental duration was 20 weeks.

The results showed that there was a significant difference between the treatments for the wet period duration factor during the first twelve weeks and no significant difference for the volume of water used to drop on the tailings as "rain". The 1-day wet period cell, and therefore, the longest dry period for cycles, combined with the lowest water volume, resulted in the fastest pH drop among the different treatments (pH=3.1 on week 16) whereas the cells that remained more saturated with water held pH values above 4. These conclusions seem to be in agreement with field results reported by Álvarez-Valero (2008) for Mediterranean weather, where long drought periods and short rainy season cause the generation of AMD during all year.

Based on these results and the previous studies mentioned in the introduction, the present research proposes to determine the effects of air flow and cycle duration in the performance of adapted humidity cells for tailings from a massive sulfide ore, measuring pH and other parameters in the leachates obtained.

METHODS

Study area

The tailings used in the test were taken from Tizapa, a massive sulfide mine located southwest of Mexico City, in the State of Mexico, near the town of San Juan Zacazonapan and approximately at 120 km from the city of Toluca, the capital of the state (Figure 1). The altitude is 1200 m.a.s.l., and the predominant climate is from temperate to warm with dry winters and rainy summers. During the months of July, August and September, the region receives the maximum precipitation. The annual average precipitation is 1,500 mm. The temperature in summer exceeds 30ºC, and the temperature in winter oscillates from 10 to 16ºC. The mine produces concentrates of zinc, lead and copper sulfides by flotation.

Sample preparation and characterization

The composite sample of essentially unweathered tailings was collected from two samplings at the process discharge pumping station to the tailings dam. The samples were taken to the Environmental Chemical Engineering Laboratory of the Faculty of Chemistry of the National Autonomous University of Mexico (UNAM) and dried at room temperature. The samples were screened and homogenized according to recommendations of the Mexican standard NMX-AA-015-1985 (DOF, 1985) and Richardson (1993) and divided with a Jones riffle Splitter (Gerlach et al., 2007). Two equal amounts of each sample were weighed and incorporated following the former procedure. Finally, 1-kg samples for each cell were packed in hermetically sealed plastic bags. The composite sample was analyzed in the mining company laboratories by the following techniques: The sample was digested according to EPA method 3051 (1991) and analyzed for total metals concentrations by inductively coupled plasma-optical emission spectrometry (ICP-OES, Perkin Elmer 2002 mod. Optima 4300 DV); semiquantitative analysis by X-ray fluorescence (XRF) and mineralogical content by X-ray diffraction (XRD). The sulfates in the tailings were extracted according to Mexican standard NOM-141-SEMARNAT-2003 (2004) and then quantified by EPA 9038 turbidimetric method (1986).
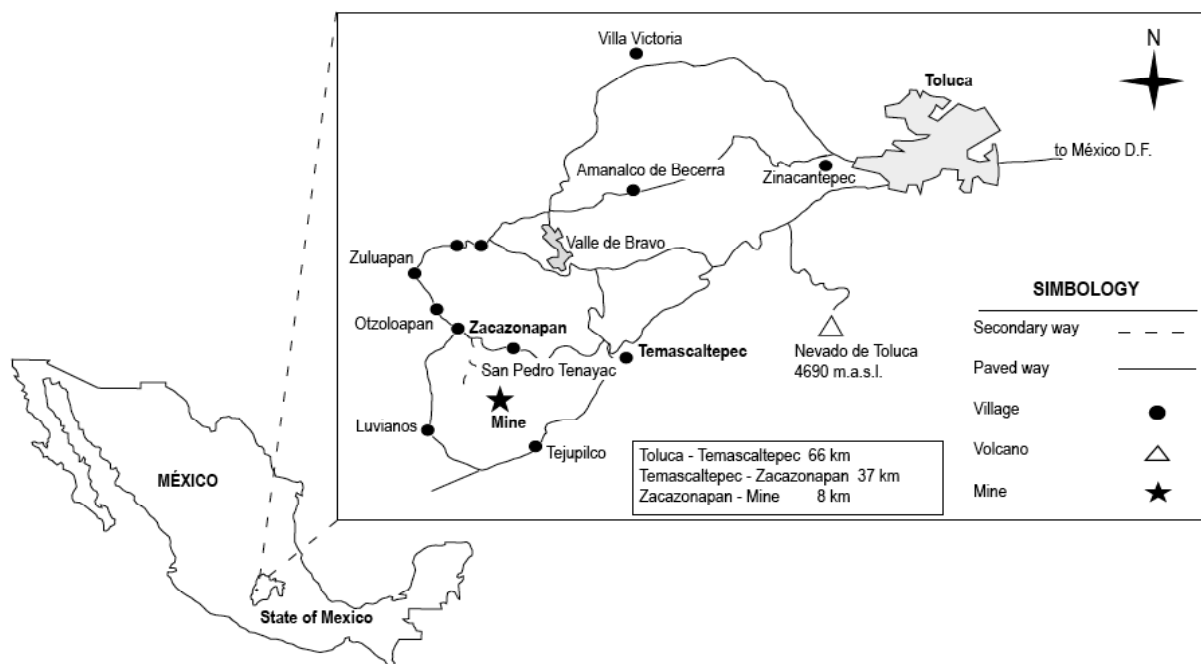

Figure 1. Mine location (Lizárraga Mendiola et al, 2008)

Humidity cells construction

The cells were dimensioned according to method ASTM D 5744-96 (2000). They were constructed from polyethylene, as shown in Figure 2. Polyethylene mesh and compressed cotton were used above the perforated plate due to availability and because it was verified that cotton did not alter the pH and electrical conductivity of solutions (González-Sandoval, 2006). Air was supplied with Hagen Maxima air pump connected to a superficial round shape hose diffuser. Each cell was loaded with one kilogram of tailings (average bed height = 2 cm) and flood rinsed with 1 L of deionized water, which was left in contact with the tailings for one hour at the beginning of the tests.

Experimental Design

In order to assess how the cycle duration and the air flow of humidity cells can affect the quality of the leachates, mainly pH and, by inference, the rate of pyrite oxidation, a 2^2 factorial experimental design was implemented in which the levels of the factors were: (1) cycle duration: 14 (as in previous study) and 21 days, were selected based on the observation that long dry periods favor pyrite oxidation and, (2) air flow rates above the tailings bed: 3 and 6 $Lmin^{-1}$ were used, considering the air flow rate interval stated in the method ASTM D 5744-96 (2000), 1 to 10 $Lmin^{-1}$. The analyzed response variable was pH, electrical conductivity (EC) and redox potential (Eh), sulfate and metals concentrations were also quantified. Each treatment combination was replicated twice.

Operation conditions

The operation conditions were defined based on the study conducted by González-Sandoval (2006) as described in the background to the study. The treatments used were as follows: (1) Cell operated with 14-d cycles and a constant air flow rate of 3 $Lmin^{-1}$, (2) Cell operated with 14-d cycles and a constant air flow rate of 6 $Lmin^{-1}$, (3) Cell operated with 21-d cycles and a constant air flow rate of 3 $Lmin^{-1}$ and (4) Cell operated with 21-d cycles and a constant air flow rate of 6 $Lmin^{-1}$.

Air was circulated continuously across the surface of the tailings, the room temperature ranged from 20 to 25 ºC, and the room humidity was not controlled. The wet period of all cells consisted of one day prior to leaching, in which 50 mL where evenly dropped on the surface of the tailings (it was observed in the previous study that the shortest the wet period, the faster pH drops). On day 14, the cells were flooded with 1 L of deionized water for a contact time of one hour after which the leachate was collected in a polyethylene vessel. The main response variable considered was pH as an indicator of pyrite oxidation. The values of pH and electrical conductivity were measured in the leachates with a Corning Checkmate II meter and redox potential with a Thermo-Orion electrode model 9678BN. The samples were split for sulfates and total metals analysis, acidified with concentrated nitric acid to pH 2 and preserved under refrigeration at 4ºC. Sulfate concentrations were determined by turbidimetry (EPA, 1986). Samples were digested according to EPA method 3005A (1992) and analyzed by inductively coupled plasma-optical emission spectrometry (ICP-OES, Perkin Elmer 2002 mod. Optima 4300 DV) at the Department of Metallurgical Chemical Engineering of the Faculty of Chemistry, UNAM.

Statistical analysis

The analysis of variance of the results was performed using Statgraphics 5.1 software for the leachate pH for a multifactorial design at a confidence level of 95%.
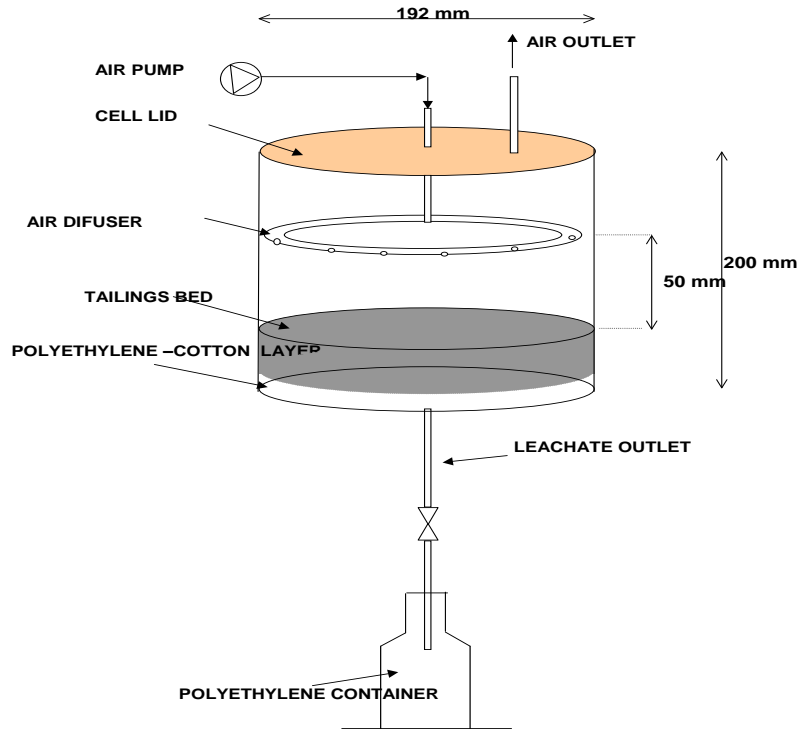
Figure 2. Humidity cell scheme

RESULTS AND DISCUSSION

Tailings characterization

Table 1 presents the mineralogical composition of the sample used in the cells. It is evident that the tailings have low buffering capacity from carbonates (2% calcite) relative to the pyrite content (71%). Samples of these tailings were previously analyzed by acid-base accounting static test (González-Sandoval, 2006), which showed their high potential to generate acid drainage (NNP<0.03). Table 2 shows the elemental composition of the tailings determined both by XRF and ICP-OES. It is expected that metals present in the tailings would likely occur in the leachates (e.g. zinc). The sulfate content of the tailings (0.32%) is attributed to slight oxidation of the sample and the use of zinc and copper sulfates in the flotation process.

Table 1. Mineralogical content of the tailings

MINERAL PHASE	COMPOSITION
FeS_2 (Pyrite)	71%
ZnS (Sphalerite)	5%
SiO_2 (Quartz)	10%
$(Mg,Fe)_6(Si,Al)_4O_{10}(OH)_8$ (Clinochlore)	5%
$(K,Na)(Al,Mg,Fe)(Si_{3.1}Al_{0.09})O_{10}(OH)$ (Muscovite)	3%
$CaCO_3$ (Calcite)	2%
$CaSO_4.2H_2O$ (Gypsum)	1%

Table 2. Elemental composition of the tailings determined by XRF and ICP-OES (weight %)

ELEMENT	XRF (%)	ICP-OES (%)
Aluminum (Al)	0.51	0.539
Arsenic (As)	0.3	0.281
Boron (B)	-	0.004
Cadmium (Cd)	0.06	0.027
Calcium (Ca)	0.94	0.461
Copper (Cu)	0.24	0.249
Iron (Fe)	35.9	37.05
Lead (Pb)	0.41	0.404
Lithium (Li)	-	<0.001
Magnesium (Mg)	0.21	0.293
Manganese (Mn)	-	0.022
Nickel (Ni)	0.002	0.002
Oxygen (O)	10.1	-
Potassium (K)	0.37	0.146
Silicon (Si)	7.3	-
Sodium (Na)	-	0.057
Sulfur (S)	41.5	-
Zinc (Zn)	3.1	3.33

(-) Not determined

Leachates quality

The values of the parameters measured in the leachates are shown in Table 3. The pH differences between duplicates range from 0 to 1.65 in the most extreme case, probably due to the dissolution of some mineral particles within the tailings. Regarding the evolution of pH, in the first four leaching cycles, pH decreased faster in the 14-days cycle cell with low air flow (4.80 to 4.48), but increased slightly in the cells with the same cycle duration and high air flow rate (4.79 to 4.96) while in the approximately equivalent time, up to the third leaching cycles of the 21-days cycle units, the pH was higher (4.7). From this point on, the acidification process was apparently triggered in all the cells, obtaining the lowest pH value in the 21-days cycles cells operated with lower air flow.

The analysis of variance, ANOVA, for the multifactorial design at an interval of confidence of 95%, for the factors of cycle duration and air flow rate and pH as response variable, showed there is no significant difference between the treatments. As a consequence, there was no significant difference for EC and redox potential since these parameters are pH dependant.

Considering that the leachings of the 14-day cycle cells and the 21-day cycle cells are performed at different times, two additional ANOVAS were calculated for 14-day cycles cells and the 21-day cycle cells, independently, taking air-flow rate and time as the controlled factors. These ANOVAS showed that the air flow had a significant effect on pH in the course of time, so that the lower air flow rate increased the oxidation of pyrite.

Table 3. Parameters measured in the leachates (mean sulfate and elemental concentrations, mg/L)

Cycle no.	Date of leaching	pH a	pH b	Δ |pHa-pHb|	pH mean	SO_4^{2-} (mgL^{-1})	E.C., µS/cm	Eh, mV	Al	As	Ca	Cd	Cu	Fe	K	Mg	Na	Pb	Zn
Results for cells with 14-day cycles, 3 Lmin^{-1} air flow rate																			
1	3/6/08	5.03	4.93	0.1	4.98	2663	3793	171.5	0.15	0.13	410.70	2.43	2.72	4.61	1.95	156.10	-	2.21	200.20
2	17/6/08	4.96	5.08	0.12	5.02	1702	2555	173.2	0.00	0.04	328.65	0.50	0.34	0.01	1.61	108.98	1.52	1.57	49.57
3	1/7/08	4.95	4.28	0.67	4.615	2441	2893	222.55	-	-	-	-	-	-	-	-	-	-	-
4	15/7/08	4.61	4.34	0.27	4.475	1521	2785	245.85	0.00	0.13	302.30	0.60	0.47	0.42	2.85	154.10	0.53	2.20	72.95
5	29/7/08	4.43	4.09	0.34	4.26	2689	3263	266.05	0.00	0.10	92.55	0.23	0.23	0.52	1.02	49.31	0.08	0.79	21.45
6	12/8/08	4.00	4.02	0.02	4.01	1752	3075	276.6	0.14	0.45	295.40	1.14	1.53	4.20	0.45	143.40	0.38	3.10	112.99
7	26/8/08	3.89	4.07	0.18	3.98	1904	2670	261.5	0.00	0.04	19.27	0.07	0.00	0.10	0.03	14.49	0.00	0.05	4.71
8	9/9/08	5.46	3.81	*1.65*	4.635	1716	2460	340.75	0.23	0.38	229.10	1.08	1.11	6.31	0.05	104.74	0.29	1.81	95.09
9	23/9/08	3.52	3.6	0.08	3.56	1975	2840	383.50	0.46	0.36	194.65	2.02	1.09	10.84	0.00	79.01	0.13	0.84	167.40
10	7/10/08	3.46	3.6	0.14	3.53	1547	2625	398.95	-	-	-	-	-	-	-	-	-	-	-
Results for cells with 14-day cycles, 6 Lmin^{-1} air flow rate																			
1	3/6/08	4.77	4.83	0.06	4.80	2175	3293	197.0	-	-	-	-	-	-	-	-	-	-	-
2	17/6/08	5.13	5.00	0.13	5.07	1879	3115	193.4	0.00	0.10	538.85	1.21	1.50	0.55	0.23	138.10	1.87	3.47	122.50
3	1/7/08	5.93	6.07	0.14	6.00	2071	3148	143.8	-	-	-	-	-	-	-	-	-	-	-
4	15/7/08	4.85	4.90	0.05	4.88	1266	2558	207.4	0.19	0.15	387.35	1.02	2.96	7.17	1.03	121.45	0.50	4.32	102.70
5	29/7/08	4.66	4.78	0.12	4.72	1466	2535	221.0	1.73	0.18	386.50	3.01	5.22	26.04	0.77	152.50	0.57	2.08	242.20
6	12/8/08	4.49	4.97	*0.48*	4.73	1573	2773	230.5	0.11	0.20	284.75	0.73	1.46	5.46	0.20	152.35	0.49	3.02	86.98
7	26/8/08	4.42	4.72	0.30	4.57	1573	2695	215.5	0.00	0.13	95.30	0.35	0.49	1.07	0.00	66.37	0.10	1.00	33.16
8	9/9/08	4.2	4.42	0.22	4.31	1208	2848	283.4	0.13	0.35	165.00	1.09	0.66	4.39	0.02	104.38	0.27	1.37	96.01
9	23/9/08	3.81	3.87	0.06	3.84	1573	2993	300.0	0.27	0.26	187.15	1.12	1.76	12.83	0.00	117.85	0.22	2.13	91.58
10	7/10/08	3.75	3.77	0.02	3.76	1466	2340	371.1	-	-	-	-	-	-	-	-	-	-	-

(-) Not determined

Table 3. Parameters measured in the leachates (mean sulfate and elemental concentrations, mg/L) (Continued)

Cycle no.	Date of leaching	pH a	pH b	Δ $|pHa-pHb|$	pH mean	SO_4^{2-} (mgL^{-1})	E.C., μS/cm	Eh, mV	Al	As	Ca	Cd	Cu	Fe	K	Mg	Na	Pb	Zn
Results for cells with 21-day cycles, 3 Lmin^{-1} air flow rate																			
1	10/6/08	4.52	4.69	0.17	4.61	2307	2943	221.8	0.62	0.10	362.10	2.27	5.88	14.81	0.64	134.05	4.25	4.55	179.05
2	1/7/08	4.62	4.71	0.09	4.67	3756	3335	206.8	-	-	-	-	-	-	-	-	-	-	-
3	22/7/08	4.75	4.64	0.11	4.70	3091	3908	216.3	0.38	0.19	408.40	1.31	3.29	14.12	0.11	165.20	0.61	4.57	121.42
4	12/8/08	4.68	4.50	0.18	4.59	3511	3770	241.2	0.07	0.23	286.15	1.16	0.86	2.04	1.70	159.45	1.58	1.62	115.40
5	2/9/08	4.16	3.96	0.20	4.06	3011	3790	288.7	0.00	0.03	0.10	0.01	0.00	0.00	0.00	0.22	0.00	0.00	0.09
6	23/9/08	3.71	3.54	0.17	3.63	2886	3765	339.0	0.55	0.37	302.05	1.55	2.11	28.20	0.00	123.40	0.25	2.43	112.00
7	14/10/08	3.67	3.73	0.06	3.70	2752	3290	338.6	-	-	-	-	-	-	-	-	-	-	-
8	5/11/08	4.83	3.33	1.50	4.08	2154	2880	320.2	0.98	0.68	791.00	2.65	3.60	52.40	0.00	171.10	0.62	3.03	349.70
9	25/11/08	3.57	3.27	0.30	3.42	1957	3045	347.4	1.75	1.13	630.20	3.97	4.35	60.31	0.00	159.50	0.10	2.61	419.80
10	16/12/08	3.29	3.32	0.03	3.31	2368	2985	374.8	-	-	-	-	-	-	-	-	-	-	-
Results for cells with 21-day cycles, 6 Lmin^{-1} air flow rate																			
1	10/6/08	4.69	4.89	0.2	4.79	2361	3538	230.1	0.60	0.11	593.65	3.58	6.62	10.23	1.09	156.35	-	2.59	295.40
2	1/7/08	5.01	4.92	0.09	4.97	3877	3485	206.4	-	-	-	-	-	-	-	-	-	-	-
3	22/7/08	4.7	4.78	0.08	4.74	2511	3208	215.3	0.48	0.24	552.70	1.70	3.81	17.35	0.57	197.00	1.05	5.68	167.00
4	12/8/08	4.99	4.94	0.05	4.96	2315	3345	220.9	0.04	0.19	242.15	0.74	0.82	1.56	1.06	132.55	0.38	2.02	67.48
5	2/9/08	4.53	4.53	0.0	4.53	2199	3160	255.5	0.00	0.03	0.04	0.01	0.00	0.00	0.00	0.10	0.00	0.00	0.22
6	23/9/08	4.13	4.04	0.09	4.09	2511	3325	287.5	0.25	0.03	38.35	0.02	0.04	0.45	2.54	38.18	-	0.00	0.41
7	14/10/08	4.19	4.36	0.17	4.28	1238	3020	278.2	-	-	-	-	-	-	-	-	-	-	-
8	5/11/08	3.96	3.75	0.21	3.86	2243	3053	305.0	0.76	1.16	705.60	2.67	3.38	38.47	0.00	269.15	0.64	6.72	282.55
9	25/11/08	3.74	3.58	0.16	3.66	2315	2815	333.9	0.82	1.10	694.50	2.59	3.36	44.58	0.00	226.70	0.45	5.95	269.70
10	16/12/08	3.62	3.51	0.11	3.57	1457	2360	357.1	-	-	-	-	-	-	-	-	-	-	-

(-) Not determined

Sulfate and metals concentrations had a similar behavior in all cells, being high in the first leaching cycles, due to the dissolution of the oxidation products remaining in the tailings. Most concentrations decreased in the intermediate cycles and increased in the last ones. It is considered that these increases correspond to the oxidation of sulfides and the dissolution of carbonates and probably aluminosilicates, as inferred by the presence of Al in the leachates. Zinc, iron and sulfate concentrations were higher in the 21-day cycle tests.

CONCLUSIONS

The results obtained in this study demonstrate that there is no significant difference between the treatments tested, but it was possible to confirm that the air flow rate has a significant effect on the pH evolution. For the air flows used in the performed tests, 3 and 6 Lmin^{-1}, the lower flow accelerated acidification. The inhibition of pyrite oxidation at the higher air flow is probably due to excessive water loss. This apparently limits the oxidation reactions, even though greater air diffusion through the tailings would be expected under the dryer conditions. It is important to continue research in kinetic test methods in order to obtain reliable results that predict with accuracy the long-term behavior of mining waste leachates under the environmental conditions of each particular site.

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