A modified protocol of the ASTM normalized humidity cell test as laboratory weathering method of mill tailings

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Introduction

Acid mine drainage generation

The mining industry generates large amounts of tailings that often contain sulfide minerals

When exposed to atmospheric conditions (water and oxygen), some tailings can produce acidity accompanied by various rates of metal dissolution.

\[
\text{FeS}_2(s) + \frac{7}{2} \text{O}_2 + \text{H}_2\text{O} \rightarrow \text{Fe}^{3+} + 2\text{SO}_4^{2-} + 2\text{H}^+
\]

Acid mine drainage prediction

Static tests (Acid Base Accounting tests, "ABA") are frequently used to determine the acidity generation potential (AGP) because they are fast and inexpensive

However, static tests have an uncertainty zone where it is impossible to clearly state about the long-term AGP

When a given tailings fall in the uncertainty interval, or when there is a need for a better understanding of the future geochemical behavior, kinetic tests are recommended.

Various types of kinetic tests

All these types of kinetic tests are based on the weathering of tailings in order to evaluate their long-term geochemical behavior.
Previous laboratory works had shown opposite results in terms of AMD generation potential for a given tailings, depending on the type of kinetic test used:

1- Benzaazoua et al. (2008) noticed no acidity production when a tailings sample was submitted to humidity cell test normalized by ASTM standards: leachates pH remained neutral over 52 cycles.

2- The same sample submitted to column kinetic test becomes acidic after 1 year, which corresponds to only 13 cycles (Demers et al. 2008).

Humidity cell test run on another reactive tailings showed important observation: once the test was interrupted (Christmas holidays), the leachate’s pH became acidic and then increase to neutral for the remaining test time.

Based on these results, the present study focuses on the humidity cell test protocol (option A, ASTM D5744-07)

The humidity cell test is the most widely used method for AMD prediction

It is the only one normalized by American Society for Testing and Materials (ASTM)

Originally designed for mine wastes with particle size less than 6.3 mm (6300 µm)

To use it with the concentrator tailings characterized by a fine sized particle distribution (<200µm), a modification of the standard ASTM protocol is tentatively investigated in this work.

Material and methods

The humidity cell test is performed in a Plexiglas chamber that enables air input and output.

- The inside diameter is 20.3 cm
- The height is 10.2 cm,
- The cell is filled with 1 kg of material placed on a perforated plate covered with two geo-textile layers.
- Dry and humid air fluxes were 1 to 1.5 L/minute
- 99% air moisture level provided by humidifier with water temperature of 25-30°C
ASTM normalized humidity cell protocol

1. Rinse the sample with deionized water
2. Dry air blown over the sample
3. Humidified air
4. Flushed water

Schematically, the evolution of water content inside the sample is as follows:

- One cycle
- Dry cycle
- Humid cycle
- Rinsing day

The objective is to maintain the sample's saturation level around optimal values (40% - 60%), based on previous works:

1. A high degree of saturation (>85%) in tailings reduces the oxygen availability to the tailings: oxidation inhibition (Ouangrawa et al. 2009).
2. A low degree of saturation (<20%) reduces the water availability for the oxidation reaction (Godbout et al. 2010).

What can be modified in the protocol?

The modification allows to maintain the sample's saturation level between 40% and 60% independently of its geotechnical properties.

Materials

- The studied tailings come from the Manitou abandoned mine site (Val d'Or, Canada) having a high AGP (acid generation potential).

<table>
<thead>
<tr>
<th>Mineralogical composition by XRD (wt %)</th>
<th>Chemical analysis</th>
<th>Acid-CaCO$_3$/t</th>
</tr>
</thead>
<tbody>
<tr>
<td>Quartz 44.3</td>
<td>Albite 6.7</td>
<td>Cl~I 3.6</td>
</tr>
</tbody>
</table>

- The sample is mainly composed of quartz, muscovite, and pyrite.
- No neutralizing mineral found in the sample, which is confirmed by the ABA test (neutralizing potential NP = 0).
- The particle size distribution of the sample is typical of concentrator tailings.

Results and analysis
The kinetic test were conducted over 24 weekly cycles. For each cycle, 500 mL of deionized water was added into each cell and leachates were analyzed. Standard and modified protocols were compared for:

- pH
- Eh
- Conductivity
- Sulfate release and iron

Evolution of the water saturation during testing

In the standard protocol, saturation level decreased progressively until it became zero by the end of the test. The sample started to dry from week 12 and continued to lose water gradually until it became completely dry at week 17. Saturation is maintained between 40% and 60% in the modified protocol.

Leachate’s pH evolution

The conductivity and acidity are related to the sulfate and iron release. The weekly release rate of sulfuric acid and iron represents the pyrite oxidation rate.
Leachate chemistry evolution

Al, Mg, Ca, and Si are products of silicate mineral dissolution

**Conclusions**

- After 24 weeks of kinetic testing, the sample in the standard protocol cell released a cumulative amount of sulfate, iron, conductivity, and acidity that was much lower (4.5 x) than for the modified protocol cell.

- Results showed that the standard humidity cell created an unsuitable oxidizing environment due to its drying cycles.

- The modified protocol maintained conditions more favourable to sulfide oxidation due to the sample saturation level which was maintained at an optimal level.

**Perspectives**

- Based on these preliminary results, six humidity cells were set up for further investigations and are presently under testing:
  - 2 cell tests were conducted as duplicate of those presented in this paper, one of them was instrumented with a water content sensor for saturation measurement during the test.
  - 2 cells were set up with the same sample (1kg), but the cell diameter was reduced to 10.2 cm to evaluate simultaneously the effect of the sample thickness and ASTM protocol modification on sulfide reactivity.
  - 2 cells (20.3 cm ID) filled with a different sample which has a lower acid generation potential (AP=70 kg CaCO3/t) to evaluate the effect of sample composition on the standard and modified test protocols.

**Thank you**

The sample was initially installed in a cell with a water saturation of 50%.

The degree of saturation is monitored by weighting the humidity cells, and the targeted S is obtained by adding deionized water to the cell during the dry and moisturized cycle.

- Sample saturation during the kinetic test was deduced by calculating the water loss and water gain and comparing to its initial water content (W%), and using the geotechnical parameters of the material placed in the humidity cell: D, sample thickness, Gs, n, Initial W(%), and the initial cell weight (with sample).
$n = \frac{e}{1+e}$

Where:

$e = \frac{V_t}{V_i} - 1$

$V_i = \frac{M_s}{G_s}$

$V_i = \frac{D^2 \times \pi}{4} \times h$

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Humidity cell test experiments

Two cells were used:

- The first cell based on the standard ASTM
- The second cell based on a modified protocol