COLUMN TEST INVESTIGATION ON THE USE OF CALCIUM CARBONATE BY PRODUCT AS MOISTURE-RETAINING LAYER TO PREVENT ACID MINE DRAINAGE

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ABSTRACT
Omya calcium carbonate by-product that comes from the purification process of natural marble seems to have the appropriate properties to be an effective moisture-retaining layer (MRL) in a cover with capillary barrier effects (CCBE). To further test this option, a column test was performed where measurements of volumetric water content (θ) and oxygen concentration were performed. Also, water samples were taken at the bottom of the column after each flushing event and chemically analysed. θ measurements indicated that the MRL remained at a high degree of saturation. The water quality showed that the cover limits the production of AMD. Finally, oxygen flux through the cover estimated using two different approaches confirmed the efficiency of the cover to limit oxygen migration and, consequently, AMD generation.

RÉSUMÉ
Omya-SPS est un sous-produit provenant du processus de purification de marbre naturel. Il est constitué essentiellement de carbonate de calcium. Ce produit semble avoir les propriétés adéquates pour constituer une couche de rétention d'eau efficace dans un recouvrement de type multicouches. Pour tester cette option, des tests en colonne ont été réalisés. Durant ces essais en colonne, des mesures de la teneur en eau volumétrique (θ) et de la concentration en oxygène ont été réalisées. De plus, des échantillons d'eau ont été prélevés en bas de la colonne et sur lesquels des analyses chimiques ont été réalisées afin d'évaluer la performance de la couverture. Les mesures de θ indiquent que la couche de rétention d'eau constituée de produits Omya-SPS est restée près de la saturation complète. La qualité de l'eau de percolation a montré que le CEBC limite la production du drainage minier acide. Enfin, le flux d'oxygène à travers la couverture évalué à l'aide de deux différentes approches, confirme l'efficacité de la CEBC à limiter la migration d'oxygène et, par conséquent, la génération du DMA.

1 INTRODUCTION

When mine wastes are considered acid-generating, different management options and rehabilitation strategies are available to inhibit the acid production. In a humid climate, oxygen barriers are considered as the most viable option (e.g. SRK 1989; MEND 2001). For that purpose, an effective way to limit oxygen migration is by the use of a cover with capillary barrier effects CCBE (e.g. Nicholson et al. 1989; Aubertin et al. 1995; Bussière et al. 2003). This type of oxygen barrier relies on a high moisture content in one (or more) of its multiple layers to prevent oxygen migration (e.g. Nicholson et al. 1989; Collin and Rasmusson 1990; Yanful 1993; Aachib et al. 1993; Ricard et al. 1997; Bussière et al., 2003). The diffusion of gas through a nearly saturated porous media can be low enough to limit the influx of oxygen from the atmosphere to the sulphidic mine wastes, hence reducing the rate of oxidation to a negligible value (e.g. Nicholson et al., 1989; Rasmuson and Erikson, 1986; Aachib et al., 1993). CCBE is considered in many situations the most viable option over the other type of oxygen barrier to limit acid mine drainage production.

CCBE usually contains three to five layers, made of different materials; each layer has one (or more) function(s). The bottom layer is made of a coarse-grained material which acts as both a mechanical support and a capillary break. The fine-grained material, utilized as the moisture-retaining layer (MRL), is placed over the first layer to create the capillary barrier effect. This material should have a relatively low saturated hydraulic conductivity (k_{sat}) compared to the capillary break layer (i.e. k_{sat} 2 to 3 orders of magnitude lower that the material below it) and a significant water retention capacity. Another coarse-grained material is placed on the moisture-retaining layer to prevent water loss by evaporation and to promote lateral drainage. Usually, the properties of the material used for this layer are similar to those of the bottom layer. Other layers can be placed over these layers to act as protective layers (see Aubertin et al. 1995 for more details).

The Omya calcium carbonate by-product (called Omya-SPS material) that comes from the purification process of natural marble seems to have the appropriate properties to be an effective moisture-retaining layer (MRL) in a CCBE. To further test this option, different laboratory tests were performed and the main results are presented in this paper.

More specifically a characterization of the materials used in the column study is first presented followed by a description of the column setup used to simulate a real CCBE. Main results in term of water quality of the leachates, and volumetric water content and oxygen concentration in the CCBE are presented. Finally, oxygen fluxes through the CCBE are calculated using sulphate production and oxygen concentration measurements.

2 MATERIALS CHARACTERIZATION

The main physical, chemical, and mineralogical properties of the materials used in the column are presented in the following.

2.1 Grain size distribution and relative density of the grains

The particle size distributions of the column components, determined using laser diffraction for the Omya-SPS and the Doyon tailings, and by standard sieves for the sand are presented in *Figure 1.*

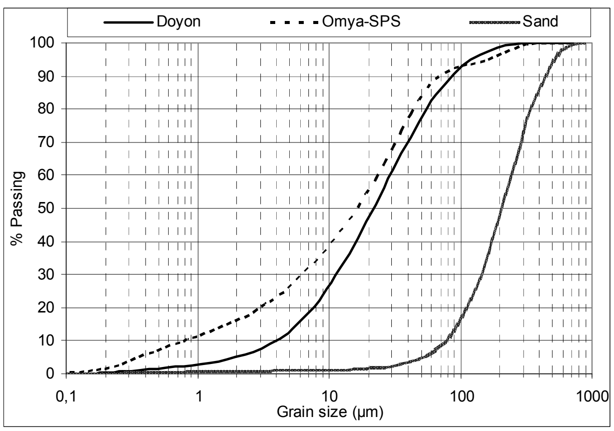

Figure 1. Grain size distribution of the materials used in the columns

This figure shows that the Doyon and Omya-SPS materials are relatively fine, while the sand is coarse compared to the other materials. These differences can be expressed using the D_{10}, D_{50}, and D_{90} parameters (diameter of particles at 10%, 50 % and 90% passing respectively). These parameters (D_{10}, D_{50} and D_{90}) are respectively 76, 200 and 445 µm for the sand, 0.8, 16 and 70 µm for the Omya-SPS, and 4, 22 and 88 µm for Doyon tailings. The measured relative density of the grains D_r (using a helium pycnometer) are 2.71, 2.73, and 2.82 respectively for sand, Omya-SPS, and Doyon tailings.

2.2 Hydraulic properties

Permeability tests were performed according to the ASTM D 5084-90 standard. For void ratios varying between 0.52 and 0.55 (porosity of 0.34 to 0.36), the saturated hydraulic conductivity (k_{sat}) of the Omya-SPS material is between 1.3E10^{-5} and 1.9E10^{-5} cm/s, typical of a silty material. A representative water retention curve (WRC) for the Omya-SPS is presented in Figure 2. For the tests performed, the air entry values (AEV) measured are between 7 and 10 m of water for void ratios between 0.46 and 0.51.

Figure 2 also shows the predicted WRC using the modified Kovács Model (Aubertin et al. 1998; 2003) for the sand where the AEV is estimated between 0.3 and 0.4 m of water. This figure shows the contrast in term of water retention properties between the Omya-SPS and the sand. These differences should give the desired capillary barrier effects in the CCBE (e.g. Bussière et al. 2003, 2007).

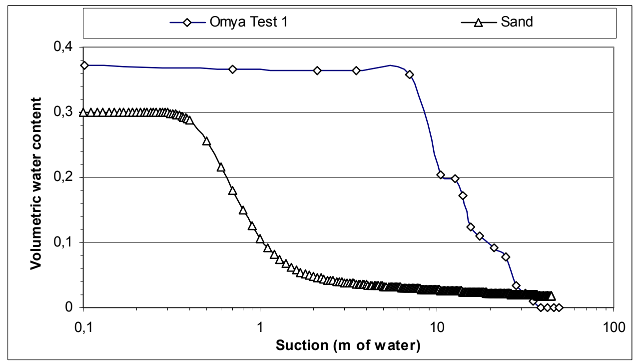

Figure 2. Typical retention curves of the tested materials

2.3 Initial chemical and mineralogical characterization

The chemical composition of the different materials used was analysed with an ICP-AES, following an acid digestion, except for sulphate sulphur (S_{SO4}) which was extracted from the solids with a 40 % HCl solution. Acid-base accounting (ABA) test was performed on the Omya-SPS materials and Doyon tailings. For the latter material neutralizing potential was estimated using the modified Sobek method proposed by Lawrence and Wang (1997) For the Omya-SPS neutralizing potential was determined based on the mineralogy. The results obtained are presented in Table 1.

The Doyon tailings show a net neutralization potential NNP (Neutralization potential NP - Acid potential AP) of -86.9 kg $CaCO_3$/t, and are therefore considered acid-generating; note that acid generation is observed on site confirming the NNP result. The Omya-SPS material shows a high NNP of almost 700 kg $CaCO_3$/t. The Omya-SPS material is mainly composed of calcium (26.0 %) with minor amounts of magnesium (1.89 %), sodium (0.622 %) and aluminium (0.448 %). The sand is composed of aluminium (4.62 %), iron (1.89 %), and calcium (1.82 %) while the Doyon tailings contain some aluminium (6.11%), iron (4.8%), sulphur (3.60%) and calcium (1.30%). For the sand and Doyon tailings, the total percentage of the elements in Table 1 is lower than 100% due to the presence of silicium in the solid (element not analysed).

Table 1. Chemical characterization and ABA test results for the studied materials

Elements	*Omya SPS*	*sand*	*Doyon*
Al (%)	0,448	4,62	6,11
Ca (%)	26,0	1,82	1,30
Cu (%)	<MDL	<MDL	0,063
Fe (%)	0,187	1,89	4,8
Mg (%)	1,89	0,732	0,716
Mn (%)	0,007	0,029	0,048
Na (%)	0,522	0,402	0,658
S (%)	0,032	0,002	3,60
S_{SO4} (%)	<MDL	<MDL	0,184
Zn (%)	0,001	<MDL	0,013
NP (kg $CaCO_3$/t)	698*	n/a	19,8
AP (kg $CaCO_3$/t)	0.06	n/a	106,7
NNP (kg $CaCO_3$/t)	697.94	n/a	-86,9

MDL: method detection limit; n/a: not analyzed

The mineralogical composition of the materials used was determined by X-Ray diffraction using the Rietveldt method (Taylor and Hinczak, 2001). Table 2 indicates that the sand is mainly composed of quartz (44.9%), anorthite (27.8%), and albite (15.1%), with minor amounts of other silicates. Those minerals have a low reactivity in the column test conditions and are therefore considered non-reactive. The Doyon tailings are mainly composed of quartz and muscovite, with almost 6% pyrite (acid-generating mineral), and 2.8 % calcite (neutralizing mineral), traces of dolomite and minor amounts of chlorite and albite. The Omya-SPS material is composed of 70% calcite, 20% quartz, and minor amounts of silicates.

Table 2. Mineralogical characterization of the column components

Mineral	Sand	Doyon	Omya-SPS
Quartz (%)	44.9	56.3	19.3
Muscovite (%)	5.7	27.0	1.8
Chlorite (%)	1.8	3.1	0.8
Albite (%)	15.1	4.7	1.5
Actinolite (%)	4.6	-	6.6
Anorthite (%)	27.8	-	-
Calcite (%)	-	2.8	69.8
Dolomite (%)	-	0.2	0.1
Pyrite (%)	-	5.9	-
Total (%)	99.9	100	99.9

3 COLUMNS SETUP

The CCBE column contains a cover with capillary barrier effects placed over 30 cm of Doyon (reactive) tailings. The CCBE is composed of a 50 cm layer of Omya-SPS material sandwiched between two 30 cm sand layers (see Figure 3). The control column only contains the Doyon tailings layer.

The CCBE column is equipped with TDR probes to measure volumetric water content, with tensiometers to measure suction, and with gas ports to measure oxygen concentration. The different equipments are located at 43, 73 and 93 cm from the bottom of the column (see Figure 3). Each TDR probes was calibrated in the laboratory with the Omya-SPS materials to improve the accuracy of the measurements (see Figure 4). Also, both columns are equipped with a ceramic plate at the bottom, used to artificially set the location of the water table (at 1.50 m below the column), and with a water outlet for water collection. More information on the column setup and on the preparation of the column test can be found in Demers and Bussière (2008; this conference).

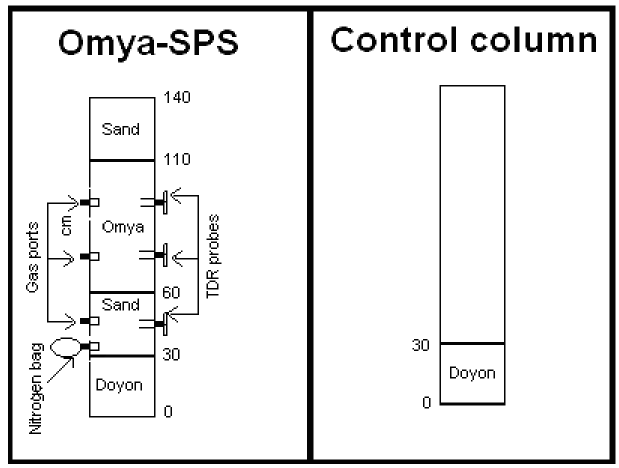

Figure 3. Columns description

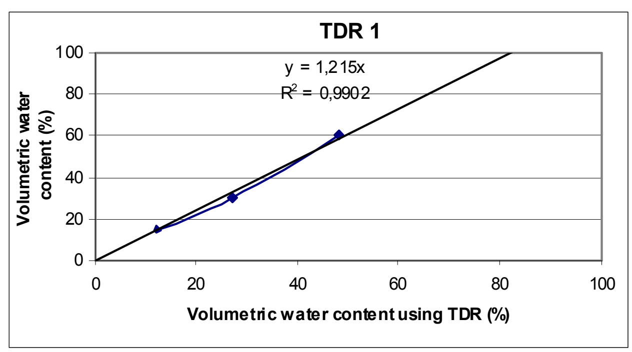

Figure 4. TDR probes calibration with the OMYA-SPS material

The flushes in both columns are done on a monthly basis. At each flush, 2L of deionised water is added at the top of the column and the water is drained out of the column after approximately 5 days. Water travelling through the cover and tailings gathers soluble elements from the solid materials, and as such could represent exfiltration water found beside a tailings impoundment. Leachate is collected at the bottom of the columns and analyzed for its quality (pH, Eh, metal content, acidity, and alkalinity). Measurements of volumetric water content and oxygen concentration were performed 7 and 28 days following each flush.

At the beginning of the tests (between the initial and 2nd flush), the column did not drain because of an air-trapping effect. Thereafter, a bag filled with nitrogen was connected to the bottom sand layer to allow drainage. Hence, the first flush was longer (46 days) but the time between two consecutive flushes remained stable after that. Also, in December 2006, a saturation of the column was performed. The objective was to simulate a wet period similar to the one observed after the snow melt in Québec. This wet period usually has a beneficial effect on the performance of a CCBE.

4 RESULTS

Results of water quality of the recovered leachates (after each flush), volumetric water content and oxygen concentration profiles are presented in the following paragraphs.

4.1 Water quality

Figure 5 shows that the CCBE column leachates had a pH greater than 7.6 after 526 days while the Doyon column had a pH less than 4. The Eh values showed a higher rising trend in the Doyon column (between 328 and 542 mV) than in the CCBE column (between 293 and 466 mV), indicating that oxidation seems to be happening more rapidly in the Doyon column. However, conductivity values were close in both columns, with values between 2500 and 4000 µmhos.cm.

Figure 6 shows the elements associated to neutralization (calcium, magnesium, and manganese), which are essentially linked to carbonate dissolution; aluminum, coming mainly from silicates dissolution is also presented.

The calcium values were similar in both columns, while magnesium, manganese, and aluminum values were much higher in the Doyon column than in the CCBE column. Aluminium appeared in very low concentrations in the water leached from the CCBE column, while in the Doyon column its concentration raised from under 1 ppm (before 300 days) to almost 62 ppm (at 484 days). This behaviour is explained by the fact that aluminum hydrolyses into $Al(OH)_3$ under pH 5.5-6.0. Since pH values in the CCBE column remained over these values throughout the experiment, aluminum values remained very low in this column. For pH values below 5, the weathering of silicate increased and no significant precipitation occurred. This phenomenon is observed in the Doyon column after 300 days.

Figure 6 also shows that copper, zinc, nickel, iron, and sulphur have lower concentrations in the CCBE column than in the Doyon column. These low concentrations in the CCBE column indicate lower sulphide oxidation rates. For the Doyon column, one can observe that the concentrations of copper, zinc, nickel, and iron increase significantly after 300 days.

This rising trend could be explained by two phenomena. First, secondary mineral precipitation happens mostly over pH 6, and the pH dropped below these values after 335 days in the Doyon column.

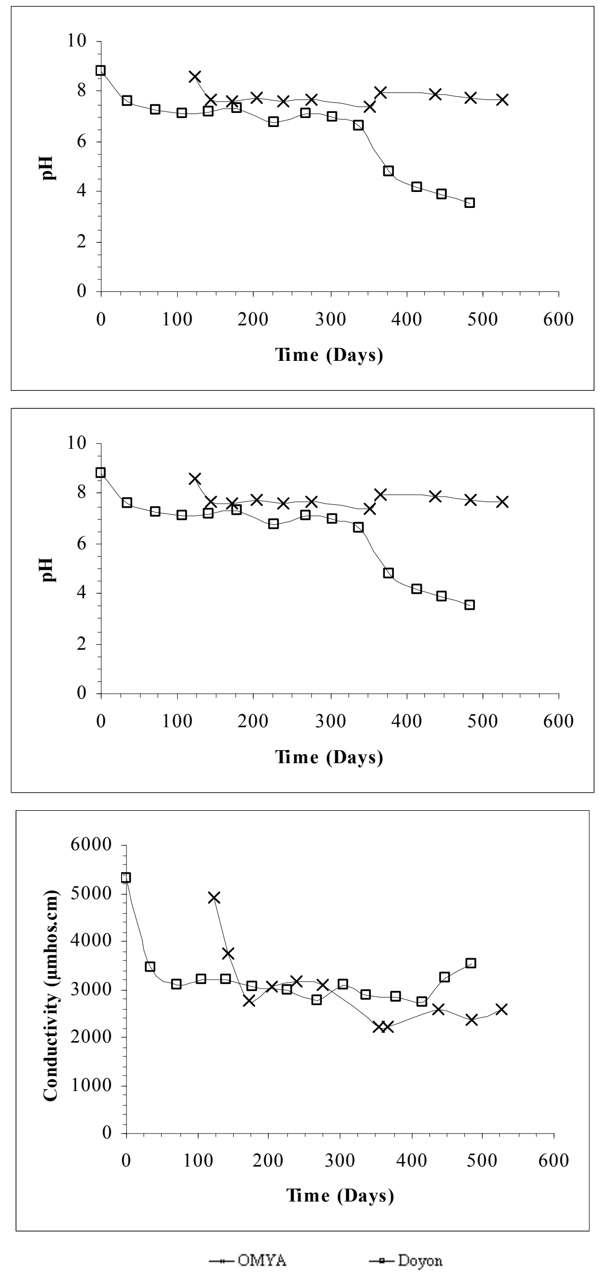

Figure 5. Evolution of pH, Eh and conductivity during test

Second, the pH drop observed after 300 days in the Doyon column accelerated sulphide oxidation and thus the leaching of these metals into the Doyon column. Although some of the released elements (copper and zinc) exceeded the authorized REMM limits (from 0.3 to 1.0 mg/L) in the early flushes of the CCBE column, they were under the accepted values for all the later flushes. Zinc, copper, iron, and sulphur concentrations show a decreasing trend during the test in the CCBE column. While sulphur levels in the Doyon column varied from 800 to 1400 mg/L, the concentrations in the CCBE column were relatively stable with a slight decreasing trend from 600 to 500 mg/L. Again, these lower values are explained by the lower oxidation rate.

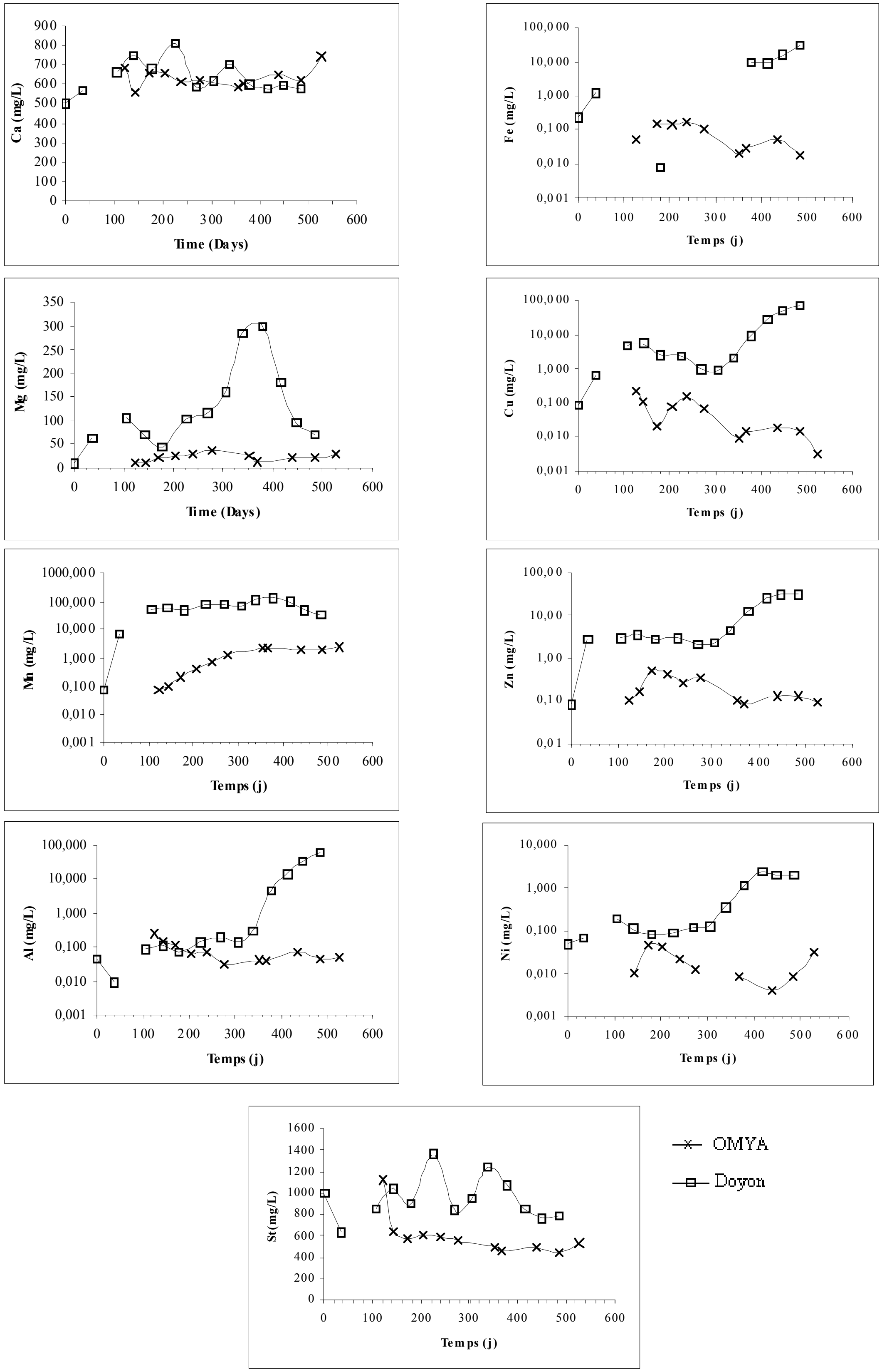

Figure 6. Evolution of the neutralization-associated elements and metal concentration during the test

4.2 Volumetric water content measurements

Volumetric water content measurements were performed at two locations (see *Figure 3*):

- 43 cm located in the middle of the sandy layer (used as capillary break layer);
- and 93 cm located near the top of the Omya-SPS material (the sensor at 73 cm was defective).

During the first flush, different problems occurred and volumetric water content measurements did not performed well. Measurements for the other flushes are presented in *Figure 7*. The volumetric water contents remained constant ($\cong$ 30%) in the Omya-SPS material which corresponds to a degree of saturation greater than 85% (assuming a porosity of 0.35). Some variations were measured in the bottom sand layer, but θ values were usually around 18 %. However, for the period following the saturation event, θ values remained relatively constant at approximately 12%. These volumetric water contents were reached rapidly after the end of the flush ($\cong$ 1 day).

This important contrast in the volumetric water content between the capillary break layer and the moisture-retaining layer is indicative of well developed capillary barrier effect.

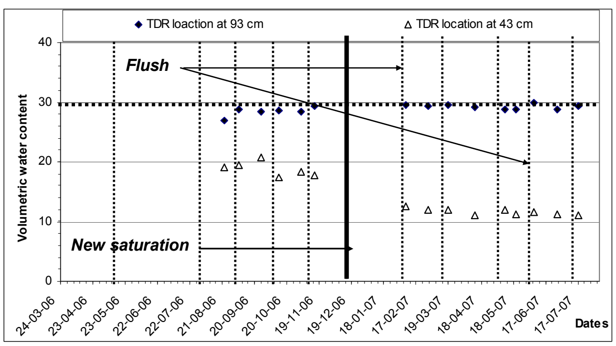

Figure 7. Volumetric water content measurements during the testing period

4.4 Oxygen concentration measurements

The oxygen measurements showed a variation between the different sampling port locations in the column with CCBE (see *Figure 8*).

Higher oxygen concentrations were measured at the location 93 cm and lower concentrations were observed at the location 43 cm, in the sand layer. For this last location, oxygen concentrations were usually lower than 5%. These measurements indicate the presence of a decreasing gradient between the top and the bottom of the CCBE; the oxygen concentration difference between the top and the bottom is between 7 and 21%.

The oxygen concentration decrease with depth is explained by oxygen consumption of the Doyon tailings at the bottom of the column. These oxygen profiles observed in this investigation are typical of others measured in covers placed over acid generating tailings (e.g. Demers 2008).

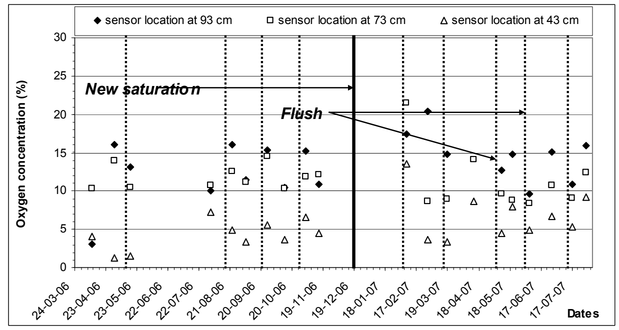

Figure 8. Oxygen concentration at the different gas sampling ports

5 PERFORMANCE OF THE CCBE TO LIMIT OXYGEN MIGRATION

The oxygen flux through the cover can be evaluated by calculating the diffusive oxygen fluxes using Fick's first law. Also, this oxygen flux can be evaluated using sulphate flux measured in the leachates.

5.1 Description of the methods used

The oxygen flux can be calculated using the Oxygen-gradient method (e.g. Elberling et al. 1994). This approach uses Fick's first law (e.g. Nicholson et al. 1989) that can be written as:

$$F(t) = -D_e \frac{\partial C(t)}{\partial z} \qquad [1]$$

where *F(t)* is the diffusive oxygen flux [$ML^{-2}T^{-1}$ or $MoleL^{-2}T^{-1}$], *C(t)* is the gas concentration in the gaseous phase at time *t* [ML^{-3} or $MoleL^{-3}$], D_e is the effective diffusion coefficient of oxygen [L^2T^{-1}] and *z* is the position [L]. In Eq.1, the oxygen gradient is calculated using oxygen concentrations measured at the two sampling points in the moisture-retaining layer made of Omya-SPS material. The accuracy of the calculated fluxes depends on the oxygen concentration measurement precision, and on the approximation of the diffusion coefficient D_e which was estimated from the following expression (Aachib et al. 2004):

$$D_e = \frac{1}{n^2}(D_a^0 \theta_a^{p_a} + H D_w^0 \theta^{p_w}) \qquad [2]$$

In this equation, *H* is the dimensionless Henry's equilibrium constant ($H \cong 0.03$ at 20°C), n is the porosity, θ_a [-] is volumetric air content in the material, θ is the volumetric water content (measured with the TDR probes), D_a^0 and D_w^0 [L^2T^{-1}] are the free oxygen diffusion coefficients in air and in water, respectively (at room temperature, $D_a^0 = 1.8\text{x}10^{-5}$ m²/s and $D_w^0 = 2.5\text{x}10^{-9}$ m²/s). Exponents p_a and p_w reflect the tortuosity of the flow path in the porous media (e.g. Aubertin et al. 2000). The value used for exponents p_a and p_w is 3.3, as proposed by Aachib et al. (2002).

Sulphate production is an indication of oxidation reactions taking place in the column. Since the OMYA-SPS contains a very small quantity of sulphur (0.032

ppm, see *Table 3*), the sulphate flux (sulphate concentration in leachate for surface area over time) measured is assumed to come from the Doyon tailings essentially. The conversion from sulphate flux to oxygen flux can be performed using the stoichiometry of the overall sulfide oxidation reaction (Kleinmann et al. 1981):

$$FeS_2(S) + \frac{7}{2}O_2(g) + H_2O \rightarrow Fe^{2+} + 2SO_4^{2-} + 2H^+ \qquad [3]$$

Eq. (3) indicates that 3.75 mol of gaseous oxygen (O_2) produce 2 mol of sulphate, which corresponds to a ratio O_2:SO_4^{2-} of 1.75. This approach of using sulphates concentrations in the leachates to estimate the oxygen flux through the CCBE is called the Sulphate-release method (Elberling et al. 1994).

5.2 Results

The calculated fluxes with the Oxygen-gradient method (using the entire test data of volumetric water content and assuming n=0.35) were between 1.3 and 15.2 mol-O_2/m^2/year (see *Figure 9*). The mean value of the calculated oxygen flux was 4.8 mol/m^2/year. Figure 9 also shows that the fluxes are usually less after the saturation event, confirming the positive effect of having such type of event on the capacity of the CCBE to limit oxygen migration. The measured fluxes are also comparable to field measurements on water and CCBE covers (e.g. Li et al., 2000; Dagenais et al. 2005; Demers 2008).

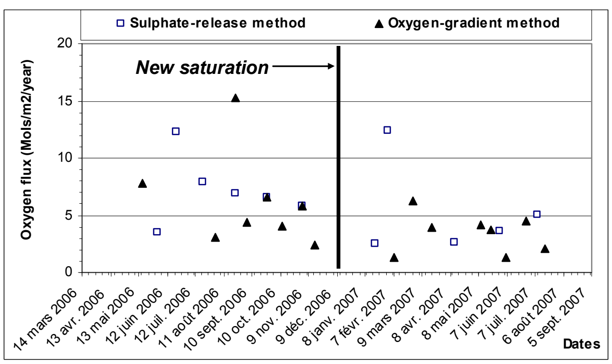

Figure 9. Evaluation of the oxygen flux at the bottom of the moisture retaining layer using Sulphate-release and Oxygen-gradient methods

Oxygen fluxes, using the Sulphate-release method, were calculated using the approach presented above. *Figure 9* shows that the oxygen flux estimated with this method are between 2.5 and 12.4 mol/m^2/year, with a mean value of 6.3 mol/m^2/year. These values are in accordance with the values obtained using the Oxygen-gradient method and with those measured on existing CCBEs (Dagenais et al. 2005; Bussière et al. 2006).

6 CONCLUDING REMARKS

The main conclusions of this experimental project are:

- The geochemical behaviour of the columns indicated that the CCBE with a moisture-retaining layer made of the Omya-SPS material limits the production of AMD; the leachates had near neutral pH values and low metal concentrations.
- Volumetric water content measurements showed that the Omya-SPS material was at a high degree of saturation during the entire testing period. The bottom sand layer remained at a relatively low volumetric water content during the test (between 10 and 20%). This water distribution in the CCBE is typical of an efficient CCBE.
- The O_2 concentrations were decreasing with depth (from top to bottom). This behaviour is explained by the oxygen consumption in the Doyon tailings at the bottom of the column.
- The oxygen fluxes through the CCBE estimated with two approaches confirm the ability of the cover to limit oxygen migration. After few months, the oxygen fluxes were usually between 1 and 5 mol/m^2/year, which correspond to measured fluxes from efficient oxygen barriers used to limit AMD generation.

This study confirms that the Omya-SPS is a good material for being used as moisture-retaining layer in a CCBE. However, more work should be done at the intermediate field scale to confirm these laboratory results.

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