

## Modeling solubility of a phosphogypsum-clay liner in contact with different pore liquids

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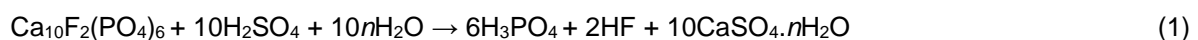
### ABSTRACT

Phosphogypsum is a by-product of the fertilizer industry, composed mainly of gypsite, whose suitable behavior in mixtures with soils has encouraged its use as a material for the construction of liners for sanitary landfills. This study investigated, through geochemical modeling, carried out by the PHREEQC software, how the solubility of a phosphogypsum-clay liner is affected by the interaction with water and leachate typical of sanitary landfills. The simulations were carried out assuming a 1 m thick liner subjected to the flow of water and leachate corresponding to the acidic, methanogenic, and mature phases of a sanitary landfill. The results indicated that the dissolution of gypsite was faster in the presence of leachate than in water, with accumulation of precipitated material in the lower layers and increased porosity in the upper layers of the liner. It was estimated that 50% of the gypsum content in the liner would dissolve after 48 pore volume of leachate flow and after 64 pore volume of water flow, as well as a 14% increase in total porosity after complete dissolution of the gypsite, leading to the conclusion that solubility must definitely be taken into account for PG to be employed in liners of sanitary landfills or in geotechnical layers where flow occurs.

*Keywords: geochemical modeling, gypsite, dissolution, PHREEQC, sanitary landfill.*

### 1 INTRODUCTION

The exploitation of phosphate rocks is an important source of raw materials for the fertilizer industry, which consumes about 71% of this kind of rock mined all over the world (IAEA, 2013). In this process, represented by the idealized chemical reaction presented in Equation 1 (Hull & Burnett, 1996), the phosphate rock ( $\text{Ca}_{10}\text{F}_2(\text{PO}_4)_6$ ) reacts with sulfuric acid ( $\text{H}_2\text{SO}_4$ ), forming two products: phosphoric acid ( $\text{H}_3\text{PO}_4$ ), hydrofluoric acid (HF), and one by-product, the phosphogypsum (PG) ( $\text{CaSO}_4 \cdot n\text{H}_2\text{O}$ ).



In Equation 1,  $n$  may be 0, 0.5, or 2, depending on the industrial process employed. In the wet process,  $n$  is equal to 2, producing the di-hydrated form of PG ( $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ ), a material similar to gypsite, except for the presence of impurities, such as fluorides, phosphates, organic matter, heavy metals (Mascarenha et al., 2018), and radionuclides of uranium and thorium (Nisti et al., 2015).

This process generates about 2.5 tons of PG per ton of phosphate rock (Pérez-Moreno et al., 2018) resulting in a global production of 200 million tons of this by-product per year (Saadaoui et al., 2017). In Brazil, 5 million tons of phosphate rock exploited per year (BRAZIL, 2019) generate about 12.5 million tons of PG. Only 15% of the global production of PG is reused – mainly in agriculture – while 85% is disposed in stockpiles and water bodies, representing a potential risk of environmental contamination (IAEA, 2013; Rashad, 2017). To reduce this volume of stocked material, several studies aiming at reuse have been carried out, such as addition in cements, mortars, concretes, and bricks (Rashad, 2017). In geotechnical engineering, PG has been used, especially, for road construction (Amrani et al., 2020; de Rezende et al., 2017; Li et al., 2020; Silva et al., 2019) and backfilling (Chen et al., 2017; Jiang et al., 2018; Li et al., 2020).

The satisfactory hydromechanical behavior of mixtures of Brazilian lateritic soils and di-hydrated PG (Mascarenha et al., 2018; Matos, 2011) has encouraged studies with the aim of using these mixtures as materials for liners of municipal solid waste (MSW) landfills. These studies have focused on the hydraulic conductivity and mechanical strength requirements (Borges, 2019; Mascarenha et al., 2018; Ribeiro et al., 2018). However, the behavior of PG in contact with the chemicals present in MSW leachate, especially in terms of solubility, should also be investigated.

The physical-chemical characteristics of MSW landfill leachates change over the years due to biogeochemical processes that may include redox, dissolution, precipitation, and complexation reactions, in addition to the action of microorganisms that degrade the waste (Souto, 2009). In the early stages, oxygen available in the waste is consumed by aerobic bacteria. However, as the concentration of this gas decreases, the anaerobic conditions promote the growth of fermentative bacteria, which are responsible for the hydrolysis of organic matter and production of acids, alcohols, and carbon dioxide (CO<sub>2</sub>). When the anaerobic conditions are fully established, this environment becomes conducive to the appearance of acidogenic bacteria, that consume the products of fermentative bacteria and produce carboxylic acids (including acetic acid), hydrogen and CO<sub>2</sub>. This low pH environment favors the solubilization of heavy metals and other inorganic substances from the solid phase. This stage is often called the acidogenic or acidic phase and lasts a few months (Alves & Bertolo, 2012; Fannin & Roberts, 2006; Pohland & Harper, 1985).

In the next stage, acetic acid and hydrogen formed in the acidic phase are converted into methane and CO<sub>2</sub> by methanogenic bacteria and, therefore, the leachate pH increases. Under these conditions, metals present in the dissolved phase may precipitate and have their concentration in the leachate reduced. This stage is called the methanogenic phase and may last for several years. Finally, the landfill reaches the mature phase, when the organic materials are almost totally consumed due to the formation of methane and CO<sub>2</sub>; the leachate presents neutral pH values and low concentrations of organic and inorganic substances (Alves & Bertolo, 2012; Fannin & Roberts, 2006; Pohland & Harper, 1985).

The interaction between liner and pores liquid, from the geomechanical standpoint, is often named compatibility and should be always carefully analyzed, since adverse reactions may cause degradation of the barrier and, consequently, an unexpected release of contaminants to the environment (Shackelford, 1994). This is notably true for soluble materials. For instance, experimental studies carried out with soils containing gypsite demonstrated that the solubility of this mineral was much higher when the soils were percolated by saline solutions than by distilled water, with a significant increase in the void ratios of the samples (Asghari et al., 2018; Azam, 2000; Ismael, 1993).

This study investigated, by means of geochemical modeling, how the solubility of di-hydrated PG is affected by the interaction with water and typical leachates found in MSW landfills, with a view to applying soil-PG mixtures for liner construction.

## 2 GEOCHEMICAL MODELING

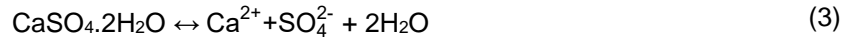
The simulations performed in this study were carried out with PHREEQC version 3, a computer program designed to perform aqueous geochemical calculations, such as speciation and saturation index calculations, batch reactions, one dimensional transport and inverse modeling (Parkhurst & Appelo, 2013). The LLNL.DAT thermodynamic database was employed.

PHREEQC was used to investigate how the solubility of PG, in a soil-PG compacted layer (liner), is affected by the flow of water and leachate from sanitary landfills at different biodegradation stages. Matos (2011) tested different compositions for soil-PG mixtures and demonstrated that the best hydromechanical performance was achieved by a mixture containing 90% soil and 10% PG in dry weight. Therefore, this composition was used in the simulations.

When compacted at the Proctor standard effort and optimum water content, this mixture presented a dry unit weight of 15.3 kN/m<sup>3</sup> and a porosity of 0.43 (Matos, 2011). Considering that PG is composed of 97.4% gypsite and that gypsite has a molecular mass of 172.2 g/mol, the initial concentration of this mineral (mol/L) in such a soil-PG layer can be calculated according to Equation 2.

$$(1530 \text{ kg}/1000 \text{ L}) \times 10\% \text{ PG} \times 97.4\% \text{ gypsite} \div (0.1722 \text{ kg}/\text{mol}) \div 0.43 = 2.012 \text{ mol}/\text{L} \quad (2)$$

The dissolution of gypsum can be written according to the following equilibrium equation (3):



At 25°C, the equilibrium constant ( $K_{\text{gypsite}}$ ) of Equation 3 is given by the solubility product of Equation 4 (Appelo & Postma, 2005):

$$K_{\text{gypsite}} = [\text{Ca}^{2+}] + [\text{SO}_4^{2-}] = 10^{-4.60} \quad (4)$$

The total amount of  $\text{Ca}^{+2}$  and  $\text{SO}_4^{2-}$  in solution is equal to the summation of the molalities ( $m$ ) of all the aqueous complexes of these species with the species in Table 1:

$$\sum \text{Ca}^{2+} = m_{\text{Ca}^{2+}} + m_{\text{CaHCO}_3^+} + m_{\text{CaSO}_4^0} + m_{\text{CaCl}^+} + m_{\text{CaCO}_3} + m_{\text{CaCl}_2} + m_{\text{CaOH}^+} + m_{\text{CaNO}_3^+} \quad (5)$$

$$\sum \text{SO}_4^{2-} = m_{\text{SO}_4^{2-}} + m_{\text{MgSO}_4} + m_{\text{CaSO}_4} + m_{\text{FeSO}_4} + m_{\text{NaSO}_4^-} + m_{\text{MnSO}_4} + m_{\text{ZnSO}_4} + m_{\text{KSO}_4^-} + m_{\text{NiSO}_4} + m_{\text{CuSO}_4} + m_{\text{HSO}_4^-} + m_{\text{AlSO}_4} + m_{\text{CdSO}_4} + m_{\text{Al}(\text{SO}_4)_2^-} + m_{\text{KHSO}_4} + m_{\text{H}_2\text{SO}_4} + m_{\text{NH}_4\text{SO}_4^-} \quad (6)$$

The amount of each specie is calculated iteratively in order to ensure the mass and electrical balances.

The Soil-PG layer was assumed to be 1.0 m thick, divided into ten sublayers (or cells) of 0.1 m each, as shown in Figure 1. The leachate composition was assumed to change over the years according to the landfill phases represented in Table 1. Thus, the liner was sequentially percolated by one pore volume (PV) of solution representing the acidic (AC) phase, 5 PV of solution representing the methanogenic (ME) and, finally, 5 PV of solution representing the mature (MA) phase.

**Table 1.** Composition of leachate in the acidic, methanogenic, and mature phases.

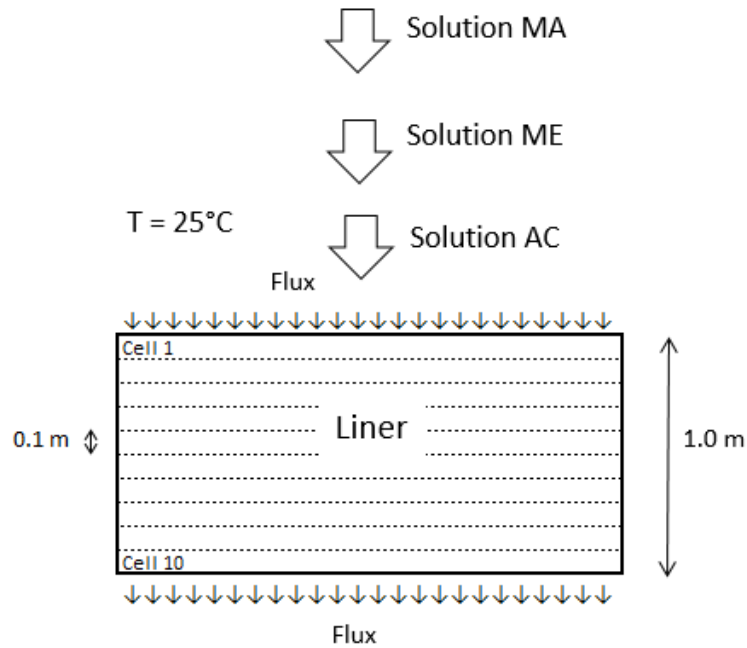
Parameter	Solution					
	AC		ME		MA	
	SIM	Reference	SIM	Reference	SIM	Reference
pH	5.8	4.4 – 8.4 <sup>a</sup>	7.8	5.9 – 9.2 <sup>a</sup>	7.1	7.1 – 8.8 <sup>b</sup>
Total Alkalinity	140.0	140 – 9,650 <sup>b</sup>	2,900.0	760-5,050 <sup>b</sup>	1377.2	200-3,520 <sup>b</sup>
Ammoniacal Nitrogen	12.0	1.7-3,000 <sup>a</sup>	200.0	0.5 – 5,000 <sup>a</sup>	415.0	6 – 430 <sup>b</sup>
Nitrate	0.5	Nd – 45 <sup>a</sup>	0.0	Nd – 270 <sup>a</sup>	0.0	0.5 – 0.6 <sup>b</sup>
Chloride	551.6	275-4700 <sup>a</sup>	975.5	20-6900 <sup>a</sup>	690.0	30 -5000 <sup>b</sup>
Sulphate	10.0	10 – 3,240 <sup>b</sup>	0.0	Nd <sup>b</sup>	5.0	5 – 40 <sup>b</sup>
Sodium	190.7	20 – 7,600 <sup>b</sup>	910.0	-	455.0	-
Calcium	49.2	10 – 2500 <sup>c</sup>	120.5	20 – 600 <sup>c</sup>	64.5	-
Potassium	37.25	35 - 2,300 <sup>b</sup>	680.8	35 - 2,300 <sup>b</sup>	480.0	35 - 2,300 <sup>b</sup>
Magnesium	49.3	3 – 1,140 <sup>b</sup>	90.1	81 – 505 <sup>b</sup>	81.0	81 – 190 <sup>b</sup>
Iron**	72.3	Nd – 1,400 <sup>a</sup>	35.8	0.01– 720 <sup>a</sup>	5.0	4 – 20 <sup>b</sup>
Manganese	19.4	Nd – 115 <sup>a</sup>	8.5	Nd – 30 <sup>a</sup>	0.6	0.6 <sup>b</sup>
Cadmium	0.1	Nd – 0.1 <sup>a</sup>	0.06	Nd – 0.6 <sup>a</sup>	0.01	-
Copper	0.3	Nd – 0.8 <sup>a</sup>	0.1	Nd – 0.6 <sup>a</sup>	0.01	-
Chrome	0.4	Nd – 1.2 <sup>a</sup>	0.1	Nd – 1.0 <sup>a</sup>	0.01	-
Nickel	0.8	Nd – 6.5 <sup>a</sup>	0.3	Nd – 1.4 <sup>a</sup>	0.2	-
Zinc	5.0	Nd – 27 <sup>a</sup>	3.5	Nd – 35 <sup>a</sup>	1.5	-
Lead	0.7	Nd – 1.1 <sup>a</sup>	0.4	Nd – 6.7 <sup>a</sup>	0.2	-

<sup>a</sup>(Souto, 2009); <sup>b</sup>(Pohland & Harper, 1985); <sup>c</sup>(Ehrig, 1983); Nd (Non-detected); \*\*Fe(II); SIM: Value used in the simulation, estimated to ensure the electric balance of the solutions; Reference: range of values, according to literature review. Concentration in mg/L.

The number of PV is calculated in Phreeqc according to Equation 7 (Appelo & Postma, 2005; Parkhurst & Appelo, 2013):

$$\text{PV} = \frac{(\text{number of shifts} + 0.5)}{\text{number of cells}} \quad (7)$$

In Phreeqc terminology, the number of shifts means how many times the solutions passed through the cells. This software calculates concentrations in the middle of the cells. So, to make the PV correspond to the full length of the layer, 0.5 is added to the numerator of Equation 7.



**Figure 1.** Simulation setting

A flux boundary condition (Cauchy type) was applied to the upper and lower faces of the liner, with a Darcy velocity of  $1.9 \times 10^{-8}$  m/s. This value was calculated considering the hydraulic conductivity coefficient and a hydraulic gradient equal to 2, according to the hydraulic conductivity tests performed by Matos (2011), and an effective porosity comprising 15% of the total porosity. The dispersivity was assumed to be 0.01 m (10% of the cell length) as suggested by Appelo and Postma (2005). The diffusion coefficient of calcium was adopted as that of calcium in water ( $7.93 \times 10^{-10}$  m<sup>2</sup>/s) at 25°C (Yuan-Hui & Gregory, 1974) multiplied by a tortuosity factor of 0.76 (Millington & Quirk, 1961). Chemical interactions with the exchangeable cations present in the soil (Table 2) were also considered in the modeling.

**Table 2.** Exchangeable cations in soil (adapted from Matos, 2011)

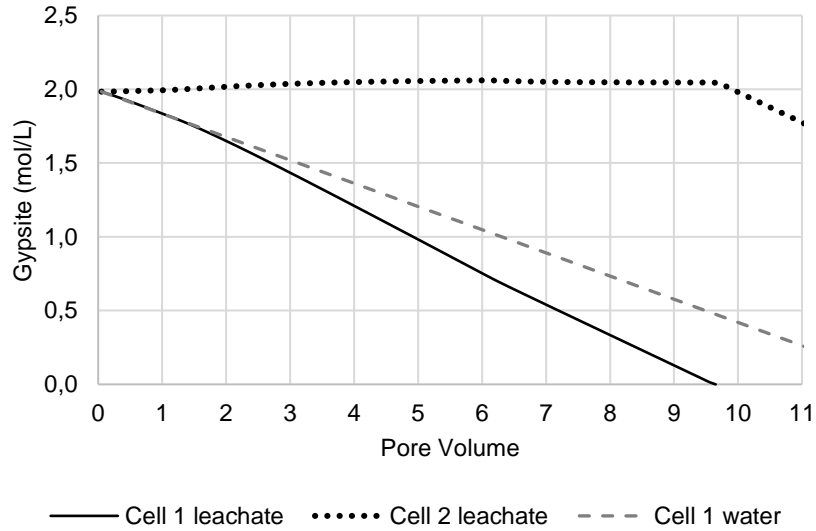
Exchangeable cations (mEq/L)				
Ca <sup>+2</sup>	Mg <sup>+2</sup>	K <sup>+</sup>	Na <sup>+</sup>	Al <sup>+3</sup>
64.90	0.20	0.06	0.39	0.40

mEq/L: milliequivalent per liter

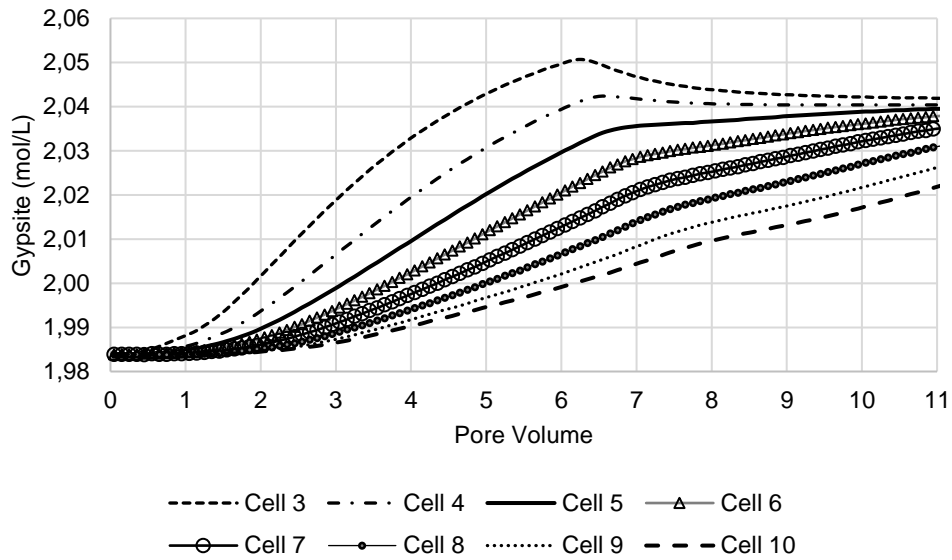
For comparison purposes, the same simulations were performed using water as percolating fluid.

### 3 RESULTS AND DISCUSSION

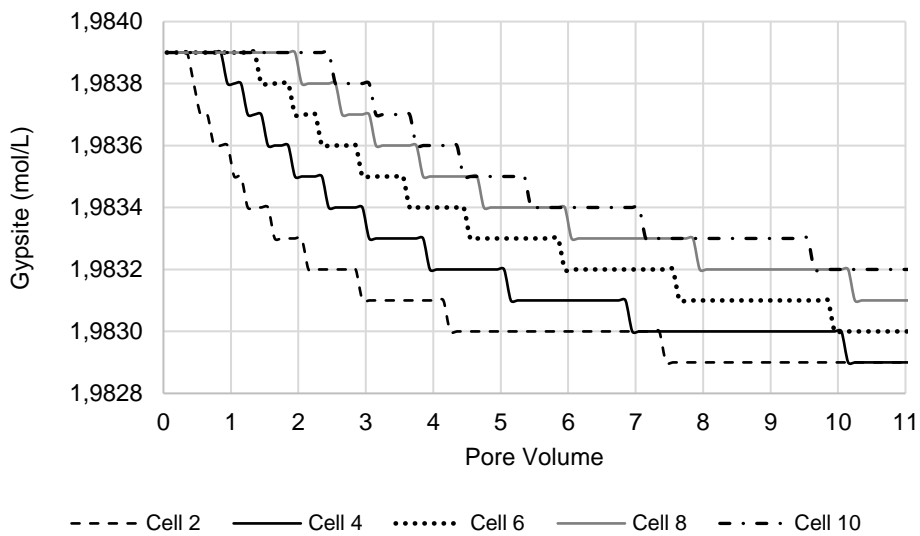
Figure 2 presents the concentration of gypsum in cells 1 and 2 (first 20 cm deep) during the flow of water and leachate. The concentration in other sublayers (cells 3 to 10) are presented in Figure 3 (leachate) and Figure 4 (water).



**Figure 2.** Concentration of gypsite in cells 1 and 2 (first 20 cm deep)



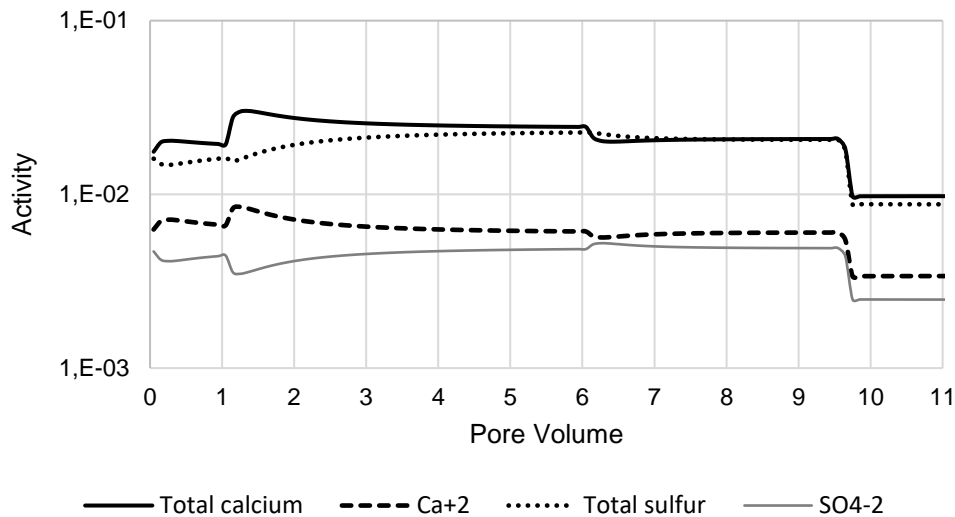
**Figure 3.** Concentration of gypsite in cells 2 to 10: leachate flow



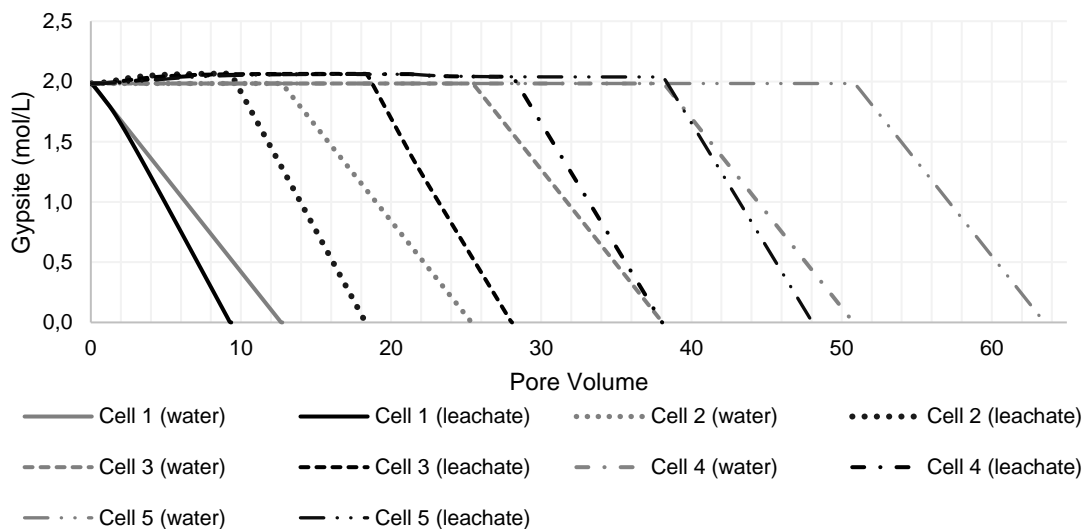
**Figure 4.** Concentration of gypsite in cells 2 to 10: water flow

The initial concentration of gypsum in Figures 2, 3 and 4 is 1.984 mol/L, while the concentration in the soil-PG mixture was 2.012 mol/L (Eq. 2), indicating that  $2.8 \times 10^{-2}$  mol/L of this mineral were dissolved to reach the equilibrium with the pore water in the compacted layer. This value is higher than the solubility of pure gypsum in water ( $1.571 \times 10^{-2}$  mol/L) and occurs because the cationic exchange process requires an additional amount of gypsum to be dissolved to provide calcium for the exchange with  $Mg^{+2}$ ,  $Na^{+1}$ ,  $K^{+1}$  and  $Al^{+3}$  present in the soil-PG mixture.

Figure 2 shows that the top of the layer (cell 1) was the most affected by the flow of water and leachate, and that the gypsum dissolution rate was higher for the simulation carried out with leachate than with water. Due to the high concentration of dissolved ions in the leachate, more chemical species are available to form aqueous complexes and therefore a greater amount of gypsum is dissolved to provide calcium and sulphate for these reactions. This is shown in Figure 5, where it is possible to see that the activity of total calcium and total sulfur is higher than that of  $Ca^{+2}$  e  $SO_4^{-2}$  ions. This difference occurs because part of calcium and sulfur are available in form of aqueous complexes with other chemical elements or even of other calcium sulphate minerals such as anhydrite ( $CaSO_4$ ) and bassanite ( $CaSO_4 \cdot 0.5H_2O$ ).



**Figure 5.** Activity of total calcium, total sulfur,  $Ca^{+2}$  and  $SO_4^{-2}$  (cell 1)



**Figure 6.** Dissolution of gypsum per layer

On the other hand, as calcium and sulphate are transported by the advective and diffusive mechanisms, the chemical equilibrium of the layers that receive these elements is shifted toward the formation of

gypsite. This process increases the amount of gypsite initially present in the inner layers of the liner, as shown in Figure 3. When the water is the percolating fluid (Figure 4), the concentration of gypsite in the pores practically does not change with time. The amount of gypsite present in all layers will decrease slowly as the dissolved material is removed by the advective flow.

The dimensionless time (PV) required for the complete dissolution of the gypsite present in the first five cells, which corresponds to 50% of the gypsite available in the liner, is presented in Figure 6. The simulation was carried out for a long-term sequence, comprising 1 PV of solution AC, 20 PV of solution ME and up to 20 PV of solution MA (until gypsite is completely dissolved). These predictions show that 50% of the gypsite present in the liner will be dissolved after 48 PV of leachate percolation, and after 64 PV when percolated by water.

Since the mixture analyzed in this study contains 10% PG or 9.74% gypsite, the dissolution of this mineral will reduce the apparent dry weight of the layers from 1,530 kg/m<sup>3</sup> to 1,390 kg/m<sup>3</sup>, which means that the total porosity will increase from 0.43 to 0.49, i.e., 14%, if the total volume of the layer does not change along the flow. Matos (2011) reported that the soil-PG mixture may reach hydraulic conductivity coefficient lower than 1x10<sup>-9</sup> m/s (Matos, 2011) when compacted at the Proctor Standard effort and optimum water content, a value generally acceptable for liners. However, as the gypsite dissolves this value will certainly increase. Furthermore, increased porosity may result in additional, non-predicted settlements, if the consolidation tests do not take into account the dissolution rate of gypsite

#### 4 CONCLUSION

The results showed how the solubility of PG can be affected by the chemical composition of the percolating fluid. In solutions of high ionic strength, such as the leachates commonly found in MSW landfills, the dissolved species tend to form aqueous complexes with other ions available in the pore liquid, which increases the solubility of the solid phase, compared to the solubility reported when the media is surrounded only by water.

The modeling also indicated that 50% of the gypsite initially available the soil-PG layer would dissolve after the percolation of 48 pore volumes of leachate and 64 pore volumes of water, while complete dissolution would result in a 14% increase in total porosity, with possible implications for the hydraulic conductivity and settlements of the layer.

Therefore, solubility must definitely be taken into account for PG to be employed in liners of sanitary landfills or in geotechnical layers where flow occurs. It is also recommended that a model verification be performed by comparing the results of the modeling with data obtained through laboratory tests.

#### 5 ACKNOWLEDGEMENTS

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