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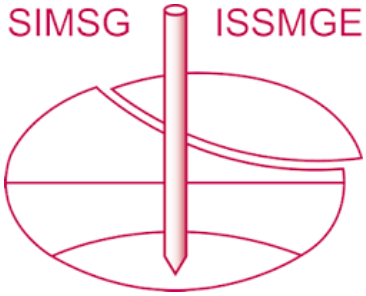

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Oit depletion in hdpe geomembranes used in contact with solutions having very high and low ph

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ABSTRACT

Heap leaching is one of several leaching methods (in-situ leaching, dump leaching, pressure leaching and tank leaching) whereby metal and other ores are leached with various chemical solutions to extract valuable minerals. Commonly HDPE is used in the pad under the ore to protect the environment and to preserve the leached solution containing the desired mineral. The most extensive application of heap leach pads is for copper ore processing. In this application, weak sulphuric acid is usually used to dissolve copper from the mineral ore. Typically copper pregnant leach solution has a pH less than 2.0 with a concentration of copper that varies from 1 to 5g/l and iron concentration up to 5g/l. A program for examining the effect on the aging of HDPE geomembranes of these and other solutions relevant to heap leach pads at both very low and high pH and to low level radioactive waste facilities where the leachate has a high pH is described. Tests are being conducted at temperatures of 40, 65, 75, 85, and 95^{o}C. The geomembrane properties and solution chemistry being used are also presented. Preliminarily antioxidant depletion rates for geomembrane incubated in a solution with a pH=0.5 are presented and a preliminary extrapolation to field temperature is provided.

RÉSUMÉ

La lixiviation en tas est l'une des nombreuses méthodes de lixiviation (lixiviation in situ, lixiviation en tas, lixiviation sous pression, lixiviation en réservoir) permettant de lessiver des minerais métalliques avec différentes solutions chimiques qui extraient des minéraux précieux. Typiquement, le PEHD est utilisé dans l'aire sous le minerai pour protéger l'environnement et contenir le lixiviat contenant le minéral désiré. L'uitlisation la plus courante d'aires de lixiviation en tas est dédiée à l'extraction du cuivre. De l'acide sulfurique peu concentré est généralement utilisé pour dissoudre le cuivre du minerai. Typiquement, le lixiviat contenant le cuivre a un pH inférieur à 2,0, et des concentrations en cuivre et en fer de 1-5g/l et 5g/l respectivement. On décrit un programme visant à examiner l'effet de ces solutions, et d'autres solutions pertinentes pour les aires de lixiviation en tas et les installations de stockage de déchets radioactifs de faible activité, sur le vieillissement de différents types de géomembranes à différentes températures (40, 65, 75, 85 et 95°C). Les propriétés de la géomembrane et la chimie des solutions utilisées sont également présentées. Les résultats préliminaires de la diminution du taux d'antioxydants pour la géomembrane incubée dans une solution à pH = 0,5 sont présentés et une extrapolation préliminaire à la température du site est fournie.

1 INTRODUCTION

Heap leaching is one of several methods (in-situ leaching, dump leaching, pressure leaching and tank leaching) whereby metal and other ores are leached with various chemical solutions that extract valuable minerals (Thiel and Smith 2004). While this technology is applied to gold, silver, copper, nitrates, uranium, nickel and other ore types, the biggest application in terms of both tonnes leached and installed leach pad area is for extracting copper from sulfide and oxide ores. In this process, copper ore from a mine (most commonly open pit) is blasted, loaded and transported usually to the primary crushers to be crushed and then often subjected to subsequent crushing and screening (though occasionally the ore is processed without crushing). The crushed ore is staked in piles ("heaps") over the leach pad and irrigated with a dilute sulfuric acid solution to dissolve the copper. The leach solution containing the dissolved copper is often called the pregnant leach solution (PLS), The pad liner is usually either a single geomembrane (GMB) or GMB with clay/geosynthetic clay liner (GCL) to act as a composite liner below a layer of permeable crushed rock drainage layer with a drainage pipe network (Thiel and Smith 2004). The PLS is drained from the bottom of the pad to a lined PLS pond. The PLS is subjected to a process called solvent extraction (SX). The SX process concentrates and purifies the copper leach solution so that copper can be recovered at a high electrical current efficiency by electrowinning (EW) cells. It does this by adding a chemical reagent to the SX tanks which selectively binds with and extracts the copper. The concentrated copper solution is then dissolved in sulfuric acid and sent to the electrolytic cells for recovery as copper plates (cathodes). The acidic solution is then pumped back to a lined raffinate pond (PLS after extracting copper) so that it can be re-used in irrigating

the expanding heap after readjusting the acid concentration.

According to Smith (2008), heap leaching can be accomplished using either permanent ("multi-stack") heaps (conventional pads) or dynamic heaps (on-off pads). In a conventional heap, one lift of ore is stacked and then leached, and then subsequent lifts stacked and leached over the prior lifts. In a dynamic heap, after leaching the spent ore is rinsed, removed and disposed of in a dump and a lift of fresh ore placed on the pad. The spent ore dump is often but not always lined.

High density polyethylene (HDPE) geomembranes are extensively used in heap leach pads in the mining industry, and linear low density polyethylene (LLDPE) is becoming popular. In copper heap leaching, oxide and sulphide ores are irrigated with weak sulphuric acid resulting in a PLS that typically has pH less than 2.0 with a copper concentration that varies from 1 to 5g/l and an iron concentration up to 5g/l. In some copper operations surfactant may be added to the PLS to aid in mineral extraction (Queja 1995; Merigold 1996).

Pilot testing of mineral extraction from uranium ores with 0.1% uranium by heap leaching in a manner similar to copper is currently in progress (Hornsey et al. 2010) and in this application the PLS typically has a pH =0.5.

According to Kappes (2002) leaching gold and silver from their ores involves the use of diluted sodium cyanide to dissolve the metals without dissolving other metals like copper and zinc. To reduce the mobilization of other minerals, the solution is maintained at pH of 9.5 to 11 by adding lime and/or caustic soda.

Heap leaching is also being applied to nickel laterite and nickel sulfide ores (Steemson, 2009). Acid usage tends to be much higher than for copper or uranium with consumption rates on the order of 500 kg of acid per tonne of ore common (compared to under 50 for copper ores). Additionally, higher temperatures are expected in nickel heap leaching, with 70ºC measured in pilot facilities and even higher temperatures are possible.

HDPE also may be used to provide containment of stabilized hazardous solid waste and low level nuclear waste landfills. Low level radioactive waste typically have a high pH in the range from 9-12.5 and have low concentrations of radioactive metals (Golder Associates Ltd, 1995).

In addition to short-term effects, an extreme pH has the potential to damage the antioxidants and other compounding agents used in geomembrane formulations, leading to eventual brittle failure of the sheet (Smith 1997).

Smith (1997) examined the suitability of several geomembranes for copper leach pads. The study used the test methodology of EPA 9090 to examine the compatibility of HDPE, very low density polyethylene (VLDPE) and polyvinyl chloride (PVC) with actual copper PLS provided by an operating SX / EW facility in Arizona. It was concluded that through this short-term testing, both HDPE and PVC are compatible with PLS used in this study while VLDPE exhibited a significant loss of physical properties.

According to Thiel and Smith (2004), laboratory tests were conducted by immersing 1.5mm HDPE GMB in 96% sulfuric acid. The result showed 2.7% loss in the tensile properties in test duration of 8 days at 23^{o}C.

Studies conducted by Gulec et al. (2004 & 2005) involved a 1.5mm HDPE geomembrane incubated in synthetic acid mine drainage (AMD), acidic water with pH=2.4, and deionized water. Their results showed a faster antioxidant depletion rate in synthetic AMD than in acidic and deionized water.

Jeon et al. (2008) incubated HDPE GMB in buffer solutions at pH 5.0 and 9.0. It was found that the OIT depleted with faster rates in the acidic solution (pH =5.0) than in the basic solution (pH =9.0).

There is a paucity of published research examining the chemical compatibility of HDPE and LLDPE GMBs with PLS from heap leach pads applications for anything but very short-term conditions. Thus the objective of this study is to investigate:

- The effect of pH and related metal concentrations on OIT depletion in HDPE GMBs,
- The service life of HDPE GMBs in heap leaching.

The following sections describe an experimental program that has been initiated and is presently ongoing. Some preliminary results are presented.

2 TEST MATRIX

2.1 Test Method

Geomembrane coupons (190 mm by 100 mm) were placed in 4 liter glass containers. The coupons were separated using 5mm glass rods to ensure that there was leachate in contact with all surfaces. The various leachates being studied (Figure 1) are added to the jars which are incubated in forced air ovens at different temperatures.

The temperatures used in this study are 40, 65, 75, 85, and 95^{o}C. High temperatures are used in recognition of the wide range of field temperatures and to obtain depletion rates in convenient time. The 95^{o}C temperature was used based on the findings of Rowe et al. (2010a) that the antioxidants depletion rates at 95^{o}C were consistent with those at lower temperatures for GMBs in both water and synthetic leachate.

The following tests are being performed both on virgin and aged GMB specimens:

- Standard and high pressure oxidative induction time (ASTM D3895 & ASTM D5885)
- Crystallinity (ASTM E794)
- Melt index (ASTM D1238)
- Tensile Properties (ASTM D6693)
- Stress crack resistance (ASTM D5397)

2.2 Test Details

2.3.1 pH Effects

The GMB samples are being incubated in six different solutions (Tables 1 & 2). The chemical concentrations presented in Table 1 (Solutions 1, 2 & 3) generally address the chemical composition and pH range relevant

to copper PLS, solution below the ore, and raffinate solution. The pH = 0.5 is examined because it is typical for Uranium and Nickel PLS solutions and highly acidic conditions are known in copper PLS especially early in the leach cycle where concentrated acid is added to the ore in a process known as “pre-curing” (see the following subsection).

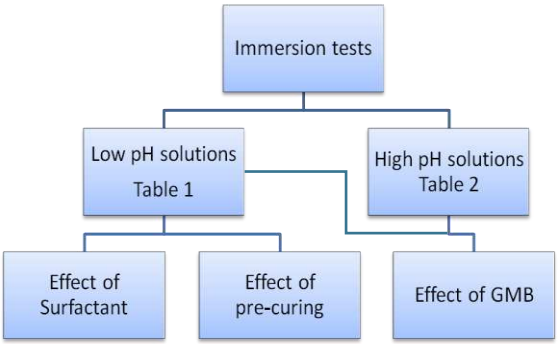

Figure 1. Flow chart showing test matrix

Table 1. Chemical constituents of the low pH solutions used in this study (mg/l except for pH)

Analyte	Sol#1	Sol#2	Sol# 3	Sol# 4	Sol# 5
pH/acid content	0.5	1.25	2.0	100g/l H_2SO_4	0.5
Cadmium	1.7	1.7	1.7	1.7	1.7
Copper	87	87	87	87	87
Calcium	550	550	550	550	550
Aluminum	4500	4500	4500	4500	4500
Cobalt	20	20	20	20	20
Iron	710	710	710	710	710
Magnesium	3300	3300	3300	3300	3300
Nickel	7.6	7.6	7.6	7.6	7.6
Zinc	62	62	62	62	62
Sodium	11	11	11	11	11
Lithium	1000	1000	1000	1000	1000
Lead	1.4	1.4	1.4	1.4	1.4
Chloride	5000	5000	5000	5000	5000
Sulphate[1]	70000	70000	70000	70000	70000
Surfactant[2]	---	---	---	---	5

[1] Sulphate concentration without taking into consideration the sulphate component in H_2SO_4 added for pH adjustment.
[2] IGEPAL Ca-720

The chemical composition for the high pH solutions used in this study (Solutions 6, 7 & 8, Table 2) simulate the PLS of gold, silver, aluminum heap leaching and low level radioactive leachate except that, for safety reasons, cyanide was not used in the leachate for gold and silver leaching and radionuclide was excluded from the low level radioactive leachate. Thus the selected solutions (Table 2) represent the low metal concentrations found in the low level radioactive waste with the range of pH found in gold, silver, aluminum heap leaching and the low level radioactive waste.

Table 2. Chemical constituents of the high pH solutions used in this study (mg/l except for pH)

Analyte	Sol# 6	Sol# 7	Sol# 8
pH	9.5	11.5	13.5
Arsenic	0.45	0.45	0.45
Copper	10.3	10.3	10.3
Silver	0.3	0.3	0.3
Aluminum	0.3	0.3	0.3
Barium	0.1	0.1	0.1
Iron	0.006	0.006	0.006
Magnesium	3	3	3
Molybdenum	0.6	0.6	0.6
Nickel	0.08	0.08	0.08
Zinc	0.02	0.02	0.02
Potassium	100	100	100
Sulphate	20	20	20
Oxides[1]	13	13	13

[1] Concentrations without taking into consideration the Sodium Hydroxide (NaOH) added for pH adjustment.

Solutions were changed every 4 weeks to ensure a constant pH during the testing period and to prevent the build-up of antioxidant concentrations in the solution.

2.3.2 Pre-curing

Copper heap leach operators have found that pre-curing the ores with concentrated sulfuric acid, commonly at 96% (Thiel and Smith 2004), is useful to satisfy the non-copper consumption and dissolve the readily soluble copper before the ore is placed on the pad during the agglomerating stage. This effectively reduces the time required to leach the metal and allows a smaller leach pad area relative to the metal production rate. Thus, irrigation of the first lift can result in high (>20g/L) copper tenor in PLS and may be accompanied by high free acid (10-20g/L), especially if the operators get over exuberant with the acid addition, which can happen at start-up Consequently, this practice raises the issue of HDPE compatibility with concentrated sulfuric acid (Thiel and Smith 2004).

To investigate such extreme effect of pre-curing, Solution 4 (Table 1) was prepared with a very high acid content (100g/l) and the base GMB was incubated in this solution at 85 and 65^{o}C for 2 weeks before being removed and incubated in the Solution 2 for ten weeks to simulate the conditions in the field. This incubation cycle is repeated every three months.

2.3.3 Surfactant

Solution 5 is the same as Solution 1 except that also contains 5mg/L of IGEPAL Ca-720 (Table 1). GMB_2 (Table 3) is being incubated at 65, 75, 85 and 95^{o}C in this solution to address the combined effect of surfactant and low pH on the service life of HDPE.

2.3.4 Geomembranes

GMBs with different HDPE resins, thicknesses and antioxidant packages are being examined (Table 3):

- GMB_2: was used for most tests (base case GMB). It is being incubated in the eight solutions defined in Tables 1 and 2 at 40, 65, 75, 85, and 95°C,
- GMB_3 is being incubated in Solutions 1 and 8 (Tables 1 & 2) at 75, 85 and 95°C.
- GMB_1: was previously used for incubation in both jar immersion (Rowe et al. 2010a, b) and composite liner configuration (having MSW leachate on the top of GM and GCL on the bottom) as described by Rowe et al. (2010c). It is examined in this study by immersion in Solutions 1 and 8 (Tables 1 & 2) at 75, 85 and 95°C Rowe et al. (2010c). This will eventually provide insight regarding composite liner performance of GMB_1 in Solutions 1 and 8 based on the relation presented in Rowe et al. (2010c) between jar immersion and composite liner configuration.

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3 PRELIMINARY RESULTS

3.1 Introduction:

This paper presents preliminary results for antioxidant depletion using standard oxidative induction time (Std-OIT). The change in the OIT value is considered to give a good indication for the amount of antioxidants depleted from the GMB with time due to ageing (Hsuan and Koerner 1998; Sangam and Rowe 2002). The Std-OIT is evaluated in accordance with ASTM D3895 using a TA Instruments Q-100 series differential scanning calorimeter (DSC).

Results for the incubation of GM_1 in Solution 1 (Table 1) are presented at three different temperatures (75, 85 and 95°C). To allow comparison, results at 85°C incubated in Solution 1 will be compared to those obtained for incubation of the same GMB in both distilled water (Rowe et al. 2010a) and municipal solid waste landfill synthetic leachate (Rowe et al. 2010b). GMB_1 used in current study is the same GMB studied by Rowe et al. (2010a & b).

A first order exponential decay relationship (Hsuan and Koerner 1998) is used to describe antioxidant depletion rates in terms of Std –OIT depletion:

$$OIT_T = OIT_o\, e^{(-st)} \quad [1]$$

or, by taking the natural logarithm on both sides:

$$ln\,(OIT_T) = -st + ln(OIT_o) \quad [2]$$

where OIT_t is the *OIT* remaining at any time *t* (min), OIT_o is the initial OIT (min), *s* is the antioxidant depletion rate ($month^{-1}$), and *t* is the ageing time (month).

Table 3. Properties of geomembranes examined in this study

Property	GMB_1	GMB_2	GMB_3
Nominal thickness (mm) (ASTM D5199)	1.5	1.5	1.0
Std-OIT (minutes) (ASTM D3895)	100 ± 2.0**	168 ± 3.0	168 ± 3.0
HP-OIT (minutes) (ASTM D5885)	273 ± 16	960 ± 25.5*	960 ± 25.5*
Suspected HALS***	No	Yes	Yes
Crystallinity (%) (ASTM E794)	48	46	52
Density (ASTM D1505)	0.947	0.936	0.936
MFI (g/10min) (ASTM D1238)	14.3 ± 0.8	11.5*	11.32 *
SCR (hours) (ASTM D5397)	800 ± 190	830 ± 130*	700 ± 50*
Strength at yield MD (kN/m) (ASTM D6693)	27.0 ± 1	28.0 ± 1.0*	18.5 ± 0.5*
Strength at break MD (kN/m)	46.0 ± 5.0	50.0 ± 3.0*	34.0 ± 1.0*
Strain at yield MD(%)	24.0 ± 2	21.0± 0.7*	25.0 ± 1.1*
Strain at break MD (%)	825 ± 80	820 ± 18*	785 ± 14*
Strength at yield XD (kN/m)	29.0 ± 0.5	29.0 ± 1.3*	20.0 ± 0.6*
Strength at break XD (kN/m)	44.0 ± 6.0	51.0 ± 1.5*	36.0 ± 0.9*
Strain at yield XD(%)	19.0 ± 0.4	18.0 ± 0.7*	19.0 ±1.2*
Strain at break XD(%)	830 ± 95	860 ± 23*	853 ± 38*

*properties obtained by A.R. Ewais; ** Virgin OIT presented here differs from that reported in Rowe et al 2010 (a,b&c) due to ageing of the roll in room temperature between the time of their tests and these tests;*** Hindered Amine light stabilizers;
MD: Machine direction; XD: Cross machine direction
GMB_2& GMB_3 are same resin same production lot

3.2 OIT Depletion Results

The variation of *ln*(OIT) with incubation time at 75, 85 and 95°C is presented in Figure 2 for GMB_1 (Table 3) in Solution 1 (Table 1). The relation between *ln*(OIT*)* and time is linear, verifying that the relation is first order as previously observed for different leachates by various investigators (Hsuan and Koerner 1998; Gulec et al. 2004; Rowe et al. 2008; Rowe and Rimal 2008; Rowe et al. 2009; Rowe et al. 2010b). In addition, Figure 3 shows that; for incubation in Solution 1, the depletion rate at 95°C is consistent with that at the lower temperatures since there is a linear relationship between the depletion rates (*ln* s) and inverse of temperature (1/T). This implies that the high temperature used (95°C) in this study is not causing any polymeric change that affects the antioxidant depletion behaviour. This is consistent with the earlier findings of Rowe et al. (2010a).

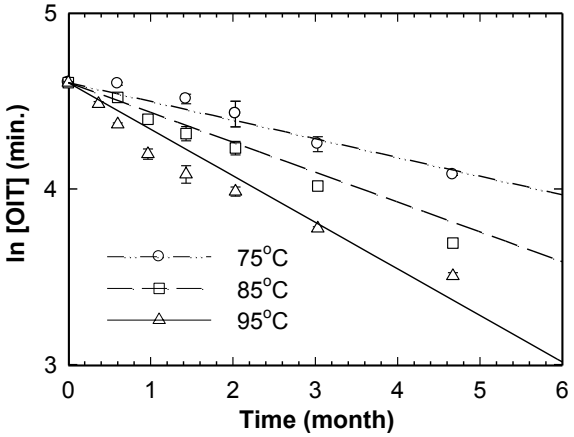

Figure 2. Antioxidant depletion rates (Std-OIT) for GMB_1 in solution 1 at three temperatures

To allow extrapolation of the antioxidant depletion rates to field temperatures; a time temperature superposition model (Arrhenius model) is commonly used. The Arrhenius equation presented by Hsuan and Koerner (1998) can be written as:

$$s = Ae^{(-Ea/(RT))} \quad [3]$$

or, by taking the natural logarithm on both sides:

$$ln\ s = ln(A) - (Ea/R)\ (1/T) \quad [4]$$

where s = antioxidant depletion rate ($month^{-1}$), Ea = activation energy (J.mol-1), R = universal gas constant (8.314 $J.mol^{-1}.K^{-1}$), T = absolute temperature (K), and A = a constant often called as collision factor.

The antioxidant depletion rate *(s)* can be obtained from the slope of the regression line in antioxidant depletion curves (Figure 2 and Eq. 2). Equation 4 can be plotted to obtain the Arrhenius plot as presented in Figure 3.

The antioxidant depletion time is extrapolated at several field temperatures as presented in Table 4.

To illustrate how the low pH leachate can affect antioxidant depletion of this HDPE GMB, Figure 4 shows the antioxidant depletion at 85°C in Solution 1 (Table 1), distilled water (Rowe et al. 2010a) and MSW synthetic leachate (Rowe et al. 2010b). It can be seen that the antioxidant depletion rates in MSW leachate is the fastest. There is only a slight difference between GMB_1 incubated in Solution 1 (Table 1) and distilled water for the incubation time presented in this paper.

Table 5 shows the antioxidant depletion rates are different in different incubation solutions. It can be seen that the depletion rate in MSW leachate and distilled water is 6.5 and 1.5 times the depletion rate in Solution 1 of pH=0.5 respectively.

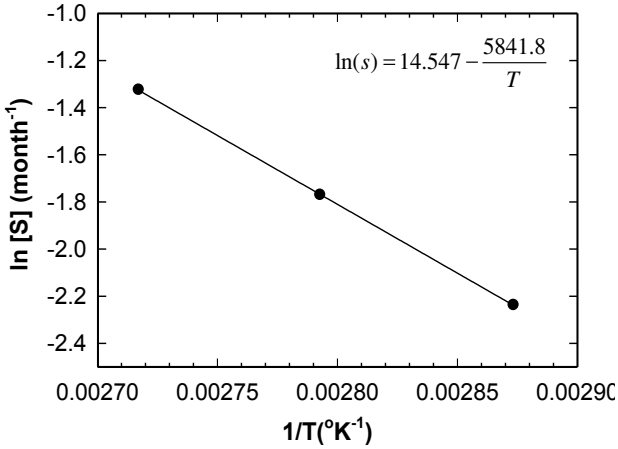

Figure 3. Arrhenius Plot of the antioxidant depletion for GMB_1 immersed in Solution 1 (Table 1)

Table 4. Predicted antioxidant depletion times based on Std-OIT for GMB_1 incubated in Solution 1

Temperature (°C)	Antioxidant depletion time (years)
50	16
40	28
30	50
20	100

Table 5. Antioxidant depletion rates in different incubation solutions

Incubation Solution	Antioxidant depletion rate ($month^{-1}$)
Solution 1 pH=0.5	0.17
Distilled water	0.26
MSW leachate	1.11

4 CONCLUSIONS

An investigation into the effect of different types of GMBs in solutions representative of a number of mining applications has been described. Three different HDPE GMBs with different resins, thicknesses and antioxidant packages are being examined.

Preliminary antioxidant depletion results based on Std-OIT depletion for one HDPE GMB incubated in a solution with and extremely low pH were presented. Based on the data available up to the time of writing, the following can be tentatively concluded:

- The antioxidant depletion stage of the GMB at field temperature, when incubated in a solution of pH of 0.5 varied from 16 years at 50°C to 100 years at 20°C.

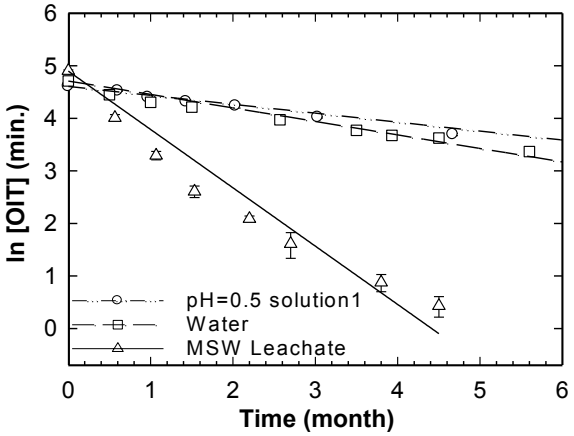

Figure 4. Std-OIT depletion for GMB_1 in different immersion fluids at 85°C (water data from Rowe et al. 2010a and MSW leachate date from Rowe et al. 2010b).

- At least for this geomembrane and solution (with no surfactant) over the limited period of the test, the extremely low pH solution had less effect on antioxidant depletion (as detected by the Std-OIT test) than the simulated MSW leachate which contained surfactant.

These conclusions only apply to the GMB, antioxidants detected by the Std-OIT test, and solutions examined over the time period examined. Since the tests are ongoing and HP-OIT tests are also in progress, these conclusions may be revised as more information becomes available in the future.

The ongoing testing will provided an indication of how rapidly antioxidants deplete for a number of different geomembranes and a range of solution relevant to heap leaching using low pH solutions and also high pH solutions relevant low level radioactive waste, stabilized hazardous waste and some heap leaching applications. Updated results will be presented in the oral presentation at the conference. The full set of results will be published in a subsequent paper when they have been run a sufficient time to draw clear conclusions.

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