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ANTIOXIDANT DEPLETION FROM HDPE AND LLDPE GEOMEMBRANES WITHOUT HALS IN AN EXTREMELY LOW pH SOLUTION

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ABSTRACT
For the last three decades, high density polyethylene (HDPE) geomembranes (GMBs) have played a crucial rule as the primary barrier in municipal solid waste (MSW) landfills. In parallel, there have been extensive investigations into the service life of HDPE GMBs in the landfills. More recently, HDPE and linear low density polyethylene (LLDPE) GMBs, have been introduced to wide range of applications in the mining industry. Mining applications (especially heap leach pads) are introducing extreme exposure conditions especially in terms of the pH of the solution in contact with the GMB liners. Leach solutions from copper, uranium and nickel heap leaching may have a pH between 0.5 and 2.0. To provide insight regarding the durability of HDPE and LLDPE GMBs in leachate relevant to some mining applications, a study has been imitated to examine the effects of: pH, GMB type, antioxidant package and GMB thickness on the service life of GMB in mining applications. In this paper, antioxidant depletion rates for a LLDPE GMB incubated in a solution with a pH of 0.5 are presented and a preliminary extrapolation to field temperature is provided. A comparison is made between the antioxidant depletion results for the LLDPE GMB with those obtained under similar conditions for an HDPE GMB.

1. INTRODUCTION

1.1 Heap leach pads

Heap leaching is a leaching method where the ore of a desired mineral (most commonly metallic) is placed in heaps on a lined pad and irrigated with an acid or a base to extract the mineral. Consequently, solutions rich with the mineral, usually called the pregnant leach solution (PLS) may have an extreme pH and high metal concentration. The PLS is collected in a lined pond prior to processing for mineral extraction. One common heap leach technique (static or conventional heaps) involves lifts of new ore being placed over prior lifts of leached ore, sometimes with a thin liner and drain pipes between lifts. In an alternative heap leaching technique (called dynamic heaps or on/off pads), after leaching the spent ore is rinsed and then disposed in a dump and fresh ore is placed on the pad (Smith 2008).

Copper heap leaching is the most common application of heap leaching. Generally, weak sulfuric acid is used in the irrigation of the heap resulting in a PLS with a pH less than 2. According to Hornsey et al. (2010), pilot testing of mineral extraction from uranium ores with 0.1% uranium by heap leaching currently in progress are producing PLS that have a similar pH (i.e. 0.5) to copper. Extractions of nickel from nickel laterite and nickel sulfide ores are also an application of extremely low pH heap leaching (Steemson et al. 2009). The pH of Nickel PLS can be lower than that of either copper or uranium PLS due to the use of more acid (Abdelaal et al. 2011). According to Abdelaal et al. (2011), the high acid usage, in addition to other factors, can increase the liner temperature to 70^{o}C or more.

Pre-curing the ore with concentrated sulfuric acid (typically 96% according to Thiel and Smith 2004) before stacking on the pad (usually during the agglomeration stage) has been found to improve copper extraction and create a (sometimes temporary) improvement in ore permeability through agglomeration of fines on the larger particles. This usually results in a PLS of extremely low pH and temperatures exceeding 50^{o}C, especially at the start-up of the operation, and long-term thermodynamic modeling of a large copper heap in Chile predicted stabilized temperatures at the base of the heap of 45^{o}C. PLS pH moves to its normal levels with the next irrigation cycles for a static heap. Pre-curing is becoming almost standard practice for copper and is being adopted at many uranium and nickel heap leaching operations.

1.2 Geomembranes role in heap leach pads

The ore is usually stacked on a liner pad comprised of a GMB as a primary liner and in many cases a low hydraulic conductivity layer (either compacted clay liner or geosynthetic clay liner). In addition, PLS collection ponds, raffinate ponds and dumps for the spent ore (from on/off pads) are usually lined with a double GMB. Table 1 shows some applications for GMB liners in heap leach pads in different parts of the world.

Typically, the PE GMB used in heap leach applications will have a 94-96% polyethylene resin, 2-3% carbon black, and 0.25-3% antioxidants and stabilizers (Koerner et al. 2005). GMBs are produced with densities varying from 0.85 to 0.96

g/cm^3, with the difference in resin density being related to the manipulation of the length of the side chains that consequently control polyethylene chain packing (Scheirs 2009). High density polyethylene (HDPE) has scarce branching with a highly packed chain structure that results in high crystallinity and a density greater than or equal to 0.94 g/cm^3. Linear low density polyethylene (LLDPE) is to a large extent a linear polymer, produced by copolymerization of ethylene as a monomer and short-chain alpha-olefins (e.g. 1-butene, 1-hexene and 1-octene) as a commoner which results in significant numbers of short branches and a typical density less than 0.939 g/cm^3 (Scheirs 2009). The less packed structure of LLDPE results in lower crystallinity compared to HDPE. This explains the higher flexibility of LLDPE and lower susceptibility to environmental stress cracking (ESC); however, this makes the LLDPE more prone to chemical degradation than HDPE (Scheirs 2009; Islam et al. 2011).

HDPE GMBs with a thickness of 1.5mm are the most commonly used GMB for heap leach pads. Nevertheless, there is a significant and increasing use of LLDPE GMBs. In landfill applications, where contaminants are aggressive and the expected vertical pressures are relatively low (250~500 kPa), HDPE has been the commonly used GMB. In heap leaching, in addition to extreme pH and elevated temperatures, the pressure applied on the liner may reach 2,000~5,000 kPa (an order of magnitude higher than commonly seen in landfill applications); modern large-scale heaps are commonly deeper than 100m (with densities of 1.75 kg/m^3 common) and several projects have been designed in the 140 to 160m range. The increasing use of LLDPE in heap leach applications arises from LLDPE's perceived generally better puncture resistance and the fact that the higher applied stresses may result in higher tensile strains in the GMB and it has been thought that a more flexible GMB that is less prone to stress cracking (i.e. LLDPE) may be a more suitable GMB for this application; however as yet there is little data in the literature examining the relative merits of HDPE and LLDPE for this applications.

Table 1. GMB liner in different heap leach pad projects.

Heap Leach Project	Type	Location	Status	Leach Pad Liner (**)
*	Valley fill	Argentina	*	2.0 & 2.5mm HDPE
*	Conventional	Brazil	*	2.0mm HDPE or LLDPE
*	Dynamic On/off	Chile	*	2.0mm HDPE
*	Dump	Chile	*	1.5mm HDPE
*	Conventional	Chile	*	2.0mm LLDPE
*	Conventional	Chile	*	0.75 & 1.0mm PVC
*	Dynamic On/off	Peru	*	2.0mm HDPE
*	Valley	Peru	*	1.5mmHDPE
*	Valley	Peru	*	2.0 & 2.5mm HDPE
*	Valley	Peru	*	2.0 & 2.5mm LLDPE
Uranium	Dynamic On/off	Southern Africa	Feasibility study	2.0mm LLDPE
Trekopje Uranium	Static heap	Namibia	Construction	1.5mm HDPE
Uranium	Dynamic On/off	Australia	Feasibility	2.0mm HDPE
Nickel	Static heap	Indonesia	Pre-feasibility	2.0mm LLDPE
Spence Mine, Copper	Dynamic On/off	Chile	Operations	1.5mm HDPE
Acoje Nickel	Static heap	Philippines	Feasibility	1.5mm HDPE
Caldag Nickel	Static heap	Turkey	Permitting	1.5mm HDPE
Esperanza, Copper	Static heap	Chile	Operations	1.5mm HDPE
Alexander gold	Static heap	Chile	Operations	1.5mm HDPE
Los Alamos gold	Static heap	Mexico	Operations	1.5mm HDPE
Poland Nickel	Dynamic On/off	Poland	Pre-feasibility	2.0mm LLDPE
Corani Silver	Static heap	Peru	Feasibility	1.5mm HDPE
Cerro Verde Pad 5 copper	Static heap	Peru	Detailed design	1.5mm HDPE
Werter Copper	-	Indonesia	Construction	1.5mm HDPE
Nickel	Static heap	South America	Pre-feasibility	2.0mm LLDPE
Carlota copper	Valley fill	Arizona, USA	Operations	2.0mm Textured LLDPE

* Data from Thiel & Smith (2004) where this information is not provided

** Textured bottom side is commonly used in the outer stability zone and not necessarily reflected in this table; where multiple thicknesses are reported, the thicker liner is used where the ore is deepest.

Islam et al. (2011) reviewed the performance of exposed 1.5 mm HDPE and 1.0 mm LLDPE based on laboratory ageing tests and field studies. They concluded that the antioxidants in stabilized LLDPE GMBs were susceptible to faster depletion than those in stabilized HDPE GMB, however there is limited data to suggest that LLDPE may encounter slower degradation than HDPE after the depletion of antioxidants (Islam et al. 2011). More research is required to confirm this inference.

1.3 Scope of the study

For the last three decades, HDPE GMBs have played a crucial rule as the primary barrier in municipal solid waste (MSW) landfills. Over the past 20 years, there has been extensive research into the service life of HDPE GMBs in landfill liners. This research has been directed at estimating the length of three stages of the GMBs service life. Meanwhile, over the last decade, HDPE GMBs have been introduced in wide range of applications in mining industry. Mining applications (especially heap leach pads) are introducing extreme exposure conditions especially with respect to the pH of the solution in contact with the GMB liners. However, there is a paucity of published research examining the chemical compatibility of HDPE with PLS from heap leach pads applications for anything but very short-term conditions. Moreover, the long term performance of LLDPE GMBs has not been yet addressed or quantified.

In 2010 a study was initiated by the GeoEngineering Centre at Queen's-RMC directed at investigating the long term performance of GMBs in heap leach pad applications. The objective of this study is to investigate:

- the effect of pH and related metal concentrations on antioxidant depletion in different GMBs, and
- the service lives of HDPE GMBs in different heap leach applications.

The initial test matrix, properties of the HDPE GMBs investigated, and the chemistry of solutions used to simulate different heap leach pad applications were presented by Abdelaal et al. (2011). This study involves the immersion of GMBs in different solutions at different temperatures to simulate the ageing of GMBs and allow extrapolations of service lives to different field temperatures.

Due to the increasing role of LLDPE in the heap leaching industry, five different LLDPE GMBs were added to the test matrix presented by Abdelaal et al. (2011). The properties of all GMBs, including three different HDPEs and five different LLDPEs, are presented in Table 2.

LLDPEs denoted GMB_4, GMB_5 and GMB_6 in Table 2 are standard production GMBs. Based on the low value of the high pressure oxidative induction time (HP-OIT) and the linear relationship between the standard OIT (Std-OIT) and HP-OIT depletion results, GMB_4 and GMB_6 do not have hindered amine light stabilizers (HALS) as part of their antioxidant package. On the other hand, high HP-OIT values for GMB_7 and GMB_8 suggest that HALS are part of the antioxidant package for these GMBs. It is known from the manufacturer that GMB_5 has a small amount of HALS as a result of some mixing during the change over in production runs, which explains the difference in OIT results between GMB_5 and the results for GMB_4 and GMB_6.

In this paper, emphasis is placed on the aging of the 1.5mm thick LLDPE (GMB_5 from Table 2) immersed in PLS with pH = 0.5 with constituents representing the chemistry of copper, uranium and nickel heap leaching. A comparison between antioxidant depletion in solutions of pH =0.5, water and synthetic municipal solid waste landfill leachate (Table 3) is presented. Finally, a preliminary comparison of the antioxidant depletion for 1.5mm HDPE (GMB_1 from Table 2) and 1.5mm LLDPE (GMB_5 from Table 2) is also presented.

2. RESULTS AND DISCUSSIONS

2.1 Quantification of the Antioxidant depletion stage

Antioxidant depletion is the first of the three stages of polyolefin GMB service life as defined by Hsuan and Koerner (1998). Typically, antioxidants depletion is followed by degradation due to oxidation, with this chemical ageing leading to a change in properties such as break strength, elongation at break and stress crack resistance. A differential scanning calorimeter (DSC) is used to obtain the standard oxidative induction time (Std-OIT) for the GMB specimen in accordance to ASTM D3895. However, the value of the Std-OIT does not indicate either the type or the quantity of antioxidants the GMB. As an index test, initial Std-OIT can be considered as a bench mark whereby the rate of depletion of antioxidants with time due to ageing can be evaluated. A first order exponential decay relationship (Hsuan and Koerner 1998) is used to describe antioxidant depletion rates in terms of Std–OIT depletion:

$$(\text{Std-OIT})_T = (\text{Std-OIT})_o\, e^{(-st)} \qquad [1]$$

or, by taking the natural logarithm on both sides:

$$ln\ (\text{Std-OIT}_T) = -st + ln(\text{Std-OIT}_o) \quad [2]$$

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

Table 2. Properties of GMBs used in the study (mean ± std dev.).

Property	GMB_1	GMB_2	GMB_3	GMB_4	GMB_5	GMB_6	GMB_7	GMB_8
Type	HDPE	HDPE	HDPE	LLDPE	LLDPE	LLDPE	LLDPE	LLDPE
Nominal thickness (mm) (ASTM D5199)	1.5	1.5	1.0	1.0	1.5	2.0	1.0	1.5
Std-OIT (minutes) (ASTM D3895)	100 ±2	168 ± 3	168 ± 3	99 ± 2	181 ± 2	105 ± 1	148 ± 1	148 ± 1
HP-OIT (minutes) (ASTM D5885)	273 ± 16	960 ± 25	960±25	260±12	350±13	240±13	885±25	885±25
Suspected HALS*	No	Yes	Yes	No	Traces	No	Yes	Yes
Crystallinity (%) (ASTM E794)	48	46	52	41	38	35	38	37
Density (g/cc) (ASTM D1505)	0.947	0.936	0.936	UI	UI	UI	UI	UI
MFI (g/10min) (ASTM D1238)	14.3±0.8	11.5	11.32	UI	UI	UI	UI	UI
SCR (hours) (ASTM D5397)	800±190	830±130	700±50	UI	UI	UI	UI	UI
Strength at yield MD (kN/m) (ASTM D6693)	27.0 ± 1	28.0±1.0	18.5±0.5	13.8±0.1	22.4± 0.5	29.8 ±0.6	14.8±0.3	22.3±0.3
Strength at break MD (kN/m)	46.0±5.0	50.0±3.0	34.0±1.0	35.4±2.0	51.8 ±7.5	66.2±3.0	36.8±2.6	53.7 ± 3.1
Strain at yield MD(%)	24.0±2	21.0±0.7	25.0±1.1	24.2± 0.6	23.0 ± 0.5	24.2± 0.9	24.0±0.7	22.4± 0.5
Strain at break MD(%)	825±80	820 ± 18	785 ± 14	880 ± 64	880 ± 104	940±30	880 ± 60	920 ± 38
Strength at yield XD (kN/m)	29.0±0.5	29.0±1.3	20.0±0.6	15.3±0.3	23.2 ± 0.1	30± 0.25	15.6±0.3	23.1± 0.3
Strength at break XD (kN/m)	44.0±6.0	51.0±1.5	36.0±0.9	34.2 ± 2.2	54.5 ± 1.8	70.0±2.1	37.7±1.0	53.2 ± 3.8
Strain at yield XD(%)	19.0±0.4	18.0±0.7	19.0 ±1.2	18.5±0.36	19.7±0.34	21.2±0.3	19.1±0.3	20.3 ± 0.4
Strain at break XD(%)	830 ± 95	860 ± 23*	853 ± 38	920 ± 56	980± 34	1020±38	1040± 32	980 ± 92

*Hindered Amine light stabilizers;

UI: Under investigation; MD: Machine direction; XD: Cross machine direction

GMB_2 & GMB_3 are same resin same production lot

Properties for GMB_1, GMB_2 & GMB_3 are from Abdelaal et al. (2011).

2.2 Std-OIT depletion of 1.5mm LLDPE (GMB_5)

The variation of *ln*(Std-OIT) with incubation time at 65, 75 and 85^{o}C is presented in Figure 1 for GMB_5 in Solution 1. The relation between *ln*(Std-OIT*)* and time is linear, verifying that the relation is first order as previously observed for different leachates and GMBs by various investigators (Hsuan and Koerner 1998; Sangam and Rowe 2002; Gulec et al. 2004; Rowe and Rimal 2008 a & b; Rowe et al. 2008; Rowe et al. 2009; Rowe et al. 2010 a, b & c; Abdelaal et al. 2011). The antioxidant depletion rates will be used later in this paper to extrapolate the antioxidant depletion time at field temperatures.

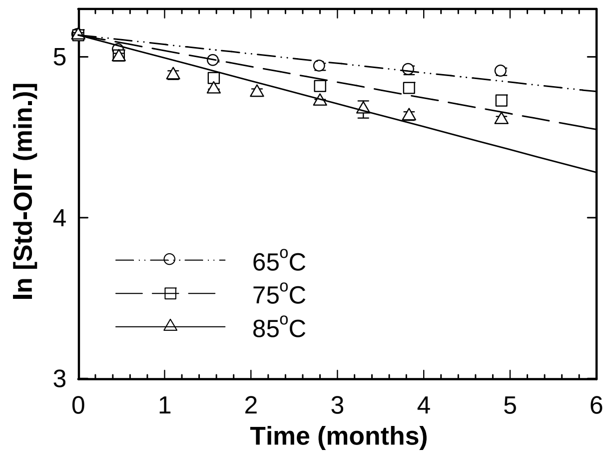


Figure 1. Antioxidant depletion (Std-OIT) for GMB_5 in Solution 1 at three different temperatures.

Table 3. Chemistry of different immersion solutions presented in this paper (unless otherwise noted, concentrations in mg/l except for pH).

Analyte	Water[1]	MSW leachate[3]	Solution 1[4]
pH/acid content	~7.0	~6.0	0.5
Aluminum	<1.0	0.0013	4500
Ammonium	--	0.00073	--
Cadmium	<0.025	--	1.7
Calcium	0.10~0.30	--	550
Cobalt	<0.02	0.031	20
Copper	<0.2	0.01	87
Iron	<0.05	0.4	710
Lead	<0.03	--	1.4
Lithium	--	--	1000
Magnesium	<0.05	--	3300
Manganese	<0.05	0.163	750
Nickel	<0.3	0.111	7.6
Potassium	0.2~0.6	--	--
Sodium	1.0~1.6	0.086	11
Zinc	<0.01	0.011	62
Chloride	--	--	5000
Sulphate	--	3.04	~70000
Surfactant[2]	---	5ml/l	---

[1] Reverse osmosis water; also used as water an in the preparation of MSW leachate and Solution 1.
[2] IGEPAL Ca-720
[3] Calculated from Rowe et al. (2010b)
[4] Abdelaal et al. (2011) calculated values

2.3 Std-OIT depletion of 1.5mm LLDPE (GMB_5) in different incubation solutions

To illustrate how low pH leachate can affect antioxidant depletion of this LLDPE GMB, Figure 2 shows the antioxidant depletion at 85°C in Solution 1, water and MSW synthetic leachate. Table 3 also provides the details of these other solutions. The antioxidant depletion rate in MSW leachate is the fastest. There is only a slight difference between the antioxidant depletion rates of GMB_5 incubated in Solution 1 and water for the incubation time presented in this paper.

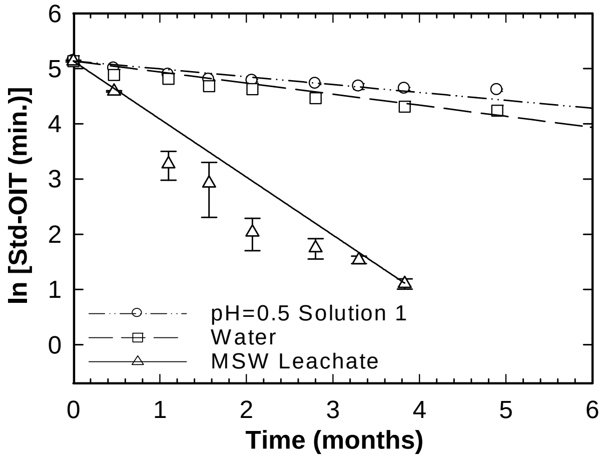
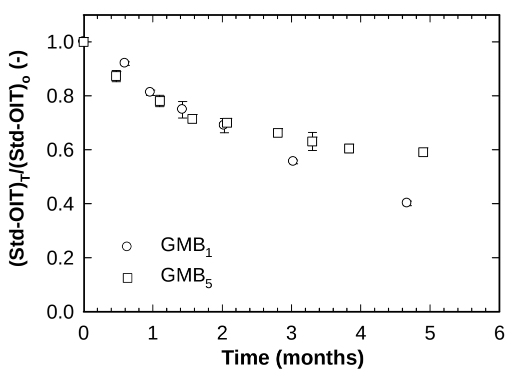


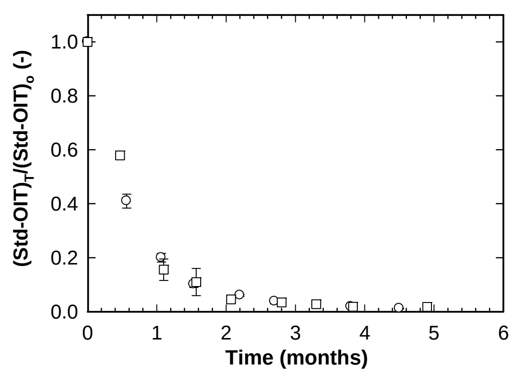
Figure 2. Std-OIT depletion in different incubation solutions at 85°C

2.4 Comparison between the Std-OIT depletion of 1.5mm LLDPE (GMB_5) and 1.5mm HDPE (GMB_1) in different incubation solutions

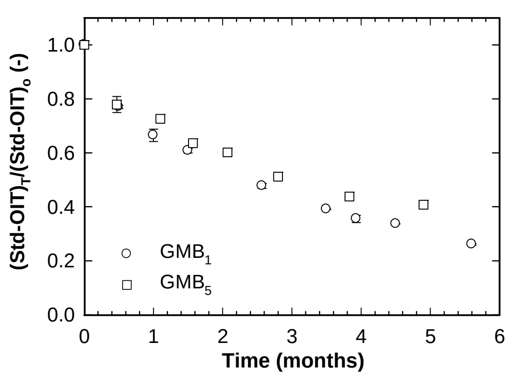
Figure 3 shows the comparison between the antioxidant depletion rates of GMB_1 (HDPE) and GMB_5 (LLDPE) plotted as normalized values to mitigate the difference in initial values of Std-OIT of the two GMBs. Results are shown for specimens incubated in water (Figure 3a), MSW leachate (Figure 3b) and mining Solution 1 with pH=0.5 (Figure 3c). The antioxidant depletion rate for both GMBs is dependent on the fluid in which it is immersed. The depletion rates for both GMBs were generally fairly similar for a given immersion fluid, but the antioxidant depletion rate for GMB_5 (1.5mm LLDPE) was a little slower than for GMB_1 (1.5mm HDPE) for all three immersion fluids.



a) Water



b) MSW leachate



c) Solution 1 pH=0.5

Figure 3. Comparison of Std-OIT depletion for GMB_1 & GMB_5 Data for GMB_1 is from Abdelaal et al. (2011).

2.5 Extrapolation of the antioxidant depletion at field 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:

$$s = A \exp(-E_a / (RT)) \quad [3]$$

or, by taking the natural logarithm on both sides:

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

where s = antioxidant depletion rate (month^{-1}), E_a = 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 the collision factor.

The antioxidant depletion rate(s) can be obtained from the slope of the regression line in antioxidant depletion curves (Figure 1 and Eq. 2). Equation 4 can be plotted to obtain the Arrhenius plot as presented in Figure 4.

Figure 4 presents the Arrhenius plot together with the Arrhenius equation for both GMB_1 and GMB_5. It should be noted that the data used to establish this plot is preliminary and will give approximate extrapolations at field temperatures. A better estimate will be obtained after complete depletion of antioxidants. Table 4 shows the calculated antioxidant depletion times at some potential field temperatures based on the results presented in Figure 4. The calculated antioxidant depletion time is slightly longer for the GMB_5 than for GMB_1, however given the preliminary nature of the data and the uncertainties associated with extrapolations, the time to depletion is very similar and the differences are of no practical significance.

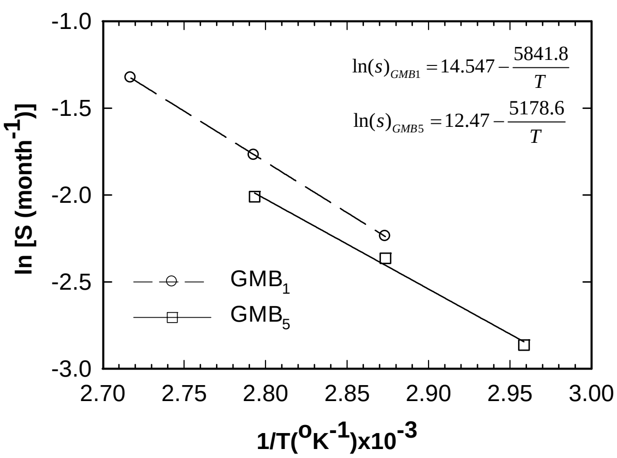


Figure 4. Arrhenius plot for GMB_1 and GMB_5

Table 4. Extrapolated antioxidant depletion time at some possible field temperatures

Temperature (^{o}C)	Antioxidant depletion time GMB_1* (years)	Antioxidant depletion time GMB_5 (years)
60	9	11
50	16	17
40	28	29

*Data from Abdelaal et al (2011)

3. CONCLUSIONS

The potential service life of GMBs in heap leach pad applications are being investigated for a range of HDPE and LLDPE GMBs and simulated pregnant liquor solutions (PLS). In this paper, preliminarily results were presented for antioxidant depletion from a 1.5mm LLDPE GMB in a PLS with a pH = 0.5 representing the chemistry of copper, uranium and nickel heap leaching. The results are compared with those for a 1.5mm HDPE GMB in the same solution. A comparison between the antioxidant depletion in solutions of pH=0.5, water and synthetic municipal solid waste landfill leachate was also presented. Based on the preliminary results presented in this paper, the following tentative conclusions have been reached:

- For GMB_5 (1.5mm LLDPE), the depletion of antioxidant was 7.5 and 1.4 times faster in MSW leachate and water (respectively) than in this PLS at a pH=0.5 (Solution1).
- Comparing antioxidant depletion of 1.5mm LLDPE to a 1.5mm HDPE, the antioxidant depletion of 1.5mm HDPE was slightly faster than for the 1.5mm LLDPE,
- The predicted antioxidant depletion times (Stage I of the service life) for the 1.5mm HDPE (GMB_1) and LLDPE (GMB_5) immersed in PLS with pH = 0.5 was about 9-11 years at 60^oC, 16-17 years at 50^oC and 28-29 years at 40^oC

These conclusions only apply to GMB_1 and GMB_5, the antioxidants detected by the Std-OIT test, and solutions examined over the time period examined. Since the tests are ongoing and (high pressure) HP-OIT tests are also in progress, these conclusions may be revised as more information becomes available in the future.

The ongoing testing will provide an indication of how rapidly antioxidants deplete for a number of different GMBs 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 firm conclusions.

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