Leachate composition and antioxidant depletion from HDPE geomembranes.

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ABSTRACT

The results of an accelerated ageing laboratory test program examining the influence of leachate chemical composition on the depletion of antioxidant from 1.5mm thick high density polyethylene (HDPE) geomembranes are reported. Results are presented for geomembrane immersed in four different synthetic leachates involving different combinations of volatile fatty acids, inorganic nutrients, trace metal solution, and surfactant. The depletion of OIT followed a first-order degradation pattern. The activation energy of the antioxidant depletion mechanism in all four leachates was calculated using Arrhenius model and found to be between 61 and 64 kJ/mol. This indicates that the four leachates are very similar in term of OIT depletion. The lowest antioxidant depletion time was observed for the simplest leachate (#2 which consisted of trace metal solution and surfactant). Given the uncertainty regarding actual leachate compositions in the field, it would appear that this leachate could reasonable and conservatively used for assessing potential depletion rates of different geomembranes and exposure conditions.

1. INTRODUCTION

High density polyethylene (HDPE) geomembranes have been extensively used as barrier materials in waste containment facilities (e.g., landfills and leachate lagoons) because of their excellent resistance to a wide range of chemicals. However, geomembranes are expected to experience ageing during their service life as a landfill barrier due to the adverse chemical effects of leachate, high physical stresses from waste loads, and elevated operating temperatures (Hsuan and Koerner, 1998). Ageing is defined as a slow and irreversible evolution of material properties under environmental stresses (Verdu, 1984). Evaluation of ageing characteristics under field condition is extremely time consuming and therefore accelerated ageing tests are conducted in the laboratory at elevated temperatures.

Sangam & Rowe (2002) performed accelerated ageing test at temperatures between 22 and 850C to investigate the depletion of antioxidant in synthetic municipal solid waste (MSW) leachate, water, and air. Their results revealed that antioxidant depletion rate was much faster in synthetic leachate than in water and air. The slowest depletion rate was observed in air aged samples.

Hsuan and Koerner (1998) conducted antioxidant depletion studies on geomembranes fully immersed in water without stress and water saturated on one-side (saturated sand above and dry sand below) with 260 kPa applied normal stress at four elevated temperatures (55 - 850C). Testing results showed that the antioxidant depletion rate was faster for the water immersed samples than for samples with one-sided exposure to water.

Müller and Jacob (2003) studied accelerated ageing test of nine commercially available 2.5mm thick HDPE geomembranes at 800C by immersing in hot water and hanging up in a forced air oven. Results showed that OIT decreased at a faster rate in water than in air as had been previously observed by Sangam and Rowe (2002) for 2mm thick geomembrane. They noted that the oxidation of the geomembrane starts only after complete depletion of the antioxidant. They also concluded that the service life of a HDPE geomembrane is controlled by the slow loss of stabilizers.

Gulec et al. (2004) conducted degradation tests on 1.5mm thick HDPE geomembrane in three immersion mediums: synthetic acid mine drainage (AMD), acidic water having pH of 2, and deionized water at 20, 40, and 600C. OIT test results showed that antioxidant depleted at a faster rate in AMD followed by acidic water and deionized water.

From the investigations described above, it is clear that antioxidants deplete at a faster rate in liquid immersion tests compared to forced-air oven. It also appears the depletion rate is faster for synthetic municipal solid waste (MSW) leachate than for AMD, acidic water, or water alone (deionized or distilled). MSW leachate typical contains volatile fatty acids, inorganic ions, trace metals, and surfactant. However it is not clear as to whether one needs to full leachate chemistry or if a simplified leachate can be used in geomembrane ageing experiments to simulate MSW landfill leachate. Thus the objective of this paper is to examine the hypothesis that a leachate comprised of the trace metals and surfactant would provided results sufficiently close to that for full leachate that it could be used in ageing experiments instead of the full leachate.

2. BACKGROUND

2.1. Ageing Mechanisms

Various ageing and degradation mechanisms can take place in geosynthetics depending on the type of polymer and environmental exposure. The degradation mechanisms include: chemical degradation, ultraviolet degradation, biological degradation, degradation by swelling, degradation by extraction, thermal degradation and oxidative degradation (Haxo and Nelson, 1984 and Koerner et al., 1990). Among these degradation processes, oxidative degradation is considered to be the most harmful for high density polyethylene geomembranes (Hawkins,1984). However, more than one degradation mechanisms occurs in the HDPE GM that creates synergistic effects and eventually the overall HDPE geomembrane degradation process accelerates. Due to the degradation of the polymer, the following changes take place in the geomembranes: gain or reduction of molecular weight, formation of free radicals, brittleness, loss of additives and plasticizers (Kulshershtha, 1992).

2.2. Oxidation of Polymers

Oxidation in polymeric materials (e.g. HDPE geomembrane) is associated with the formation of free radicals or auto-oxidation chain mechanism (Kelen, 1983). The auto oxidation chain mechanism has two interacting cyclical processes: cycle A and cycle B as shown in Figure 1 (Grassie and Scott, 1985). In cycle A, hydroperoxide (ROOH) and free radicals are formed due to alkyl/alkylperoxyl chain reactions whereas in cycle B, homolysis of hydroperoxide occurred and more free radicals are formed. Geomembranes which experience a decreased molecular weight due to the chain scission eventually become more susceptible to environmental stress cracking (Tisinger and Giroud, 1993). As described by Grassie and Scott (1985), Equations (1) to (6) describe the oxidative degradation process in polymers. The oxidative reaction initiates by forming a free radicals (R•) from the polymer chain (RH) under the influence of energy or catalyst residues in the polymer:

$$RH \rightarrow R\bullet + H\bullet \qquad (1)$$

Once a free radical is formed it reacts with oxygen and to produce a peroxy radical (ROO•) (Cycle A, Figure 1).

$$R\bullet + O_2 \rightarrow ROO\bullet \qquad (2)$$

A hydrogen atom from the surrounding polymer reacts with the peroxy radical (ROO•) and forms hydroperoxide (ROOH) and another free radical (Fay and King, 1994).

$$ROO\bullet + RH \rightarrow ROOH + R\bullet \qquad (3)$$

The breakdown of hydroperoxide initiates when a significant concentration of ROOH accumulates (Cycle B, Figure 1).

$$ROOH \rightarrow RO\bullet + OH\bullet \qquad (4)$$

More free radicals form due to the decomposition of ROOH which eventually attacks the polymer chain to form more free radicals and alcohol (ROH).

$$RO\bullet + RH \rightarrow ROH + R\bullet \qquad (5)$$

Simultaneously, there are reactions between the OH• radical and the polymer chain which yield free radicals and water.

$$OH\bullet + RH \rightarrow H_2O + R\bullet \qquad (6)$$

Oxidative degradation is known to be accelerated by the presence of transition metals (Osawa and Ishizuka, 1973). More free radicals are formed when metal ions react with hydroperoxide through 'redox' reactions according to the following equations (Osawa, 1992):

$$ROOH + M^{n+} \rightarrow RO\bullet + M^{(n+1)} + OH^{-} \qquad (7)$$

$$ROOH + M^{(n+1)+} \rightarrow ROO\bullet + M^{n} + H^{+} \qquad (8)$$

2.3. Antioxidants

Antioxidants are normally added to polymeric materials to retard the oxidative reactions and to extend the induction period (Grassie and Scott, 1985 and Al-Malaika, 1998). Antioxidants break the links in the auto-oxidation chains as shown by letters (a) – (d) in Figure 1. Various factors affect the effectiveness of antioxidants: temperature, type of antioxidant, amount of antioxidant, and the combination of antioxidants used. There are four different antioxidants that are commonly used in the formulation of polyethylene geomembranes: hindered phenols, hindered amines, phosphites, and thiosynergists (Fay and King, 1994). Antioxidants can also be divided into two categories: primary (hindered amines and hindered phenol) and secondary antioxidants (phosphates, thiosynergysts, and hindered amines). Primary antioxidants react with free radical either by accepting or donating an electron and form a stable polymer chain. Secondary antioxidants interfere the formation of free radical by decomposing hydroperoxides into stable alcohols. The selection of antioxidant should be such that they can withstand the manufacturing temperatures (2000C and 2800C) as well as service temperatures (Gulec et al., 2004). More than one antioxidant is generally used by the manufacturers to provide greater stability to the geomembrane.

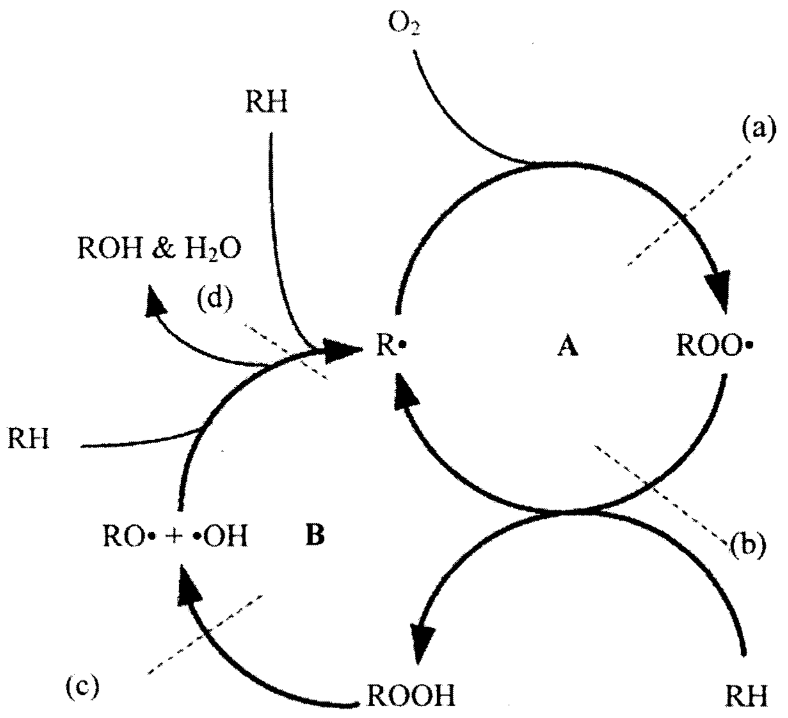

Figure 1. Auto-oxidation chain reaction cycles in polyethylene (adapted from Grassie and Scott, 1985)

Antioxidants are depleted from polymeric materials due to the chemical reactions with oxygen, free radicals, and hydroperoxide; and/or to their physical loss by diffusion, extraction or volatilization (Gedde et al., 1994; Hsuan and Koerner, 1998; and Sangam and Rowe 2002). Since the typical operating temperature for HDPE geomembrane is below 600C, volatilization is not a major concern for HDPE geomembranes (Hsuan and Koerner, 1998). Loss due to extraction is significant in controlling the life time prediction of antioxidant. Tests conducted by Smith et al. (1992) on medium density polyethylene pipes exposed to water inside and air outside have shown a 75% reduction in antioxidants due to extraction.

3. EXPERIMENTAL PROCEDURES

3.1. Materials

In this study a 1.5 mm thick high density polyethylene geomembrane was used. The geomembrane was manufactured by GSE Lining Technology Inc., Houston, TX and was supplied by Terrafix Geosynthetics Inc., Toronto. The initial OIT (ASTM D3895) and the density (ASTM D792) of the geomembrane were 174 min and 0.946 gm/cm3 respectively.

3.2. Exposure Conditions:

Geomembrane samples were cut into small coupons (190mm by 100mm) and immersed in four different synthetic leachates at 22, 40, 55, 70, and 850C. To avoid the effects of leachate interaction with the container material, 4 liter glass jars with Teflon lined lids were used. As many as 16 geomembrane coupons were put in a glass jar for each specific temperature and leachate type. The coupons were separated by 2mm glass rods to ensure that there was leachate in contact with all surfaces. Synthetic leachates were produced by mixing different combinations of volatile fatty acids, inorganic nutrients, trace metals, and surfactant with distilled water (Tables 1 and 2) and were changed every two weeks. Surfactants arise in landfill leachate due, inter alia, to the disposal of detergents in the waste stream. Several researchers observed surfactants in different MSW landfills (e.g., Maisonneuve et al., 97; Hrapovic, 2001; Kjeldsen et al., 2002; Borghi et al., 2003 etc.). A representative surfactant, Igepal® CA 720 was chosen in this study to simulate the landfill leachate condition. The chemistry of synthetic leachate -1 was based on average data for leachate from the Keele Valley Landfill, Ontario.

Table 1. Composition of synthetic leachates

COMPONENT	Concentration, mg/L (except where noted)			
	Leachate–1[a]	Leachate–2	Leachate–3	Leachate–4
Volatile Fatty Acids				
Acetic Acid (mL/L)	7	---	---	7
Propionic Acid (mL/L)	5	---	---	5
Butyric Acid (mL/L)	1	---	---	1
Inorganic Nutrients				
NaHCO3	3012	---	3012	---
CaCl2	2882	---	2882	---
MgCl2•6H2O	3114	---	3114	---
MgSO4•7H2O	319	---	319	---
NH4HCO3	2439	---	2439	---
CO(NH2)2	695	---	695	---
NaNO3	50	---	50	---
K2CO3	324	---	324	---
KHCO3	312	---	312	---
K2HPO4	30	---	30	---
Trace Metal Solution (mL/L)	1	1	1	1
Others				
Surfactant, Igepal® CA720 (mL/L)	5	5	5	5
pH (adjusted by adding either NaOH or H2SO4) (-)	6	6	6	6
Eh (adjusted by adding 3% w/v Na2S•9H2O) (mV)	-120	---	-120	-120

[a] based on Hrapovic (2001)

Table 2. Composition of trace metal solution (modified from Hrapovic, 2001)

COMPONENT	Formula	mg/L(except where noted)
Ferrous Sulfate	FeSO4•7H2O	2000
Boric Acid	H3BO3	50
Zinc Sulfate Heptahydrate	ZnSO4•7H2O	50
Cupric Sulfate Pentahydrate	CuSO4•5H2O	40
Manganous Sulfate Monohydrate	MnSO4•H2O	500
Ammonium Molybdate Tetrahydrate	(NH4)6Mo7O24•4H2	50
Aluminum Sulphate 16-Hydrate	Al2(SO4)3•16H2O	30
Cobaltous Sulphate Heptahydrate	CoSO4•7H2O	150
Nickel (II) Sulfate	NiSO4•6H2O	500
Sulfuric Acid (mL)	H2SO4	1

3.3. Oxidative Induction Time (OIT) by Differential Scanning Calorimeter (DSC)

The amount of antioxidant present in the geomembrane was assessed according to ASTM D3895 in terms of the oxidative induction time (OIT) obtained using a Q-100 series differential scanning calorimeter (DSC). The OIT deduced using this technique is useful in monitoring the relative oxidative resistance of the GM (Tisinger, 1989; Dudzik and Tisinger, 1990; Surman et al., 1995; Hsuan and Koerner, 1995, 1998; Maisonneuve et al., 1997; Sangam and Rowe, 2002; Gulec et al., 2004; and Rimal et al., 2004). For each specimen, at least five replicate samples were tested and the average used for each data point.

4. RESULTS AND DISCUSSIONS

4.1. Antioxidant Depletion

Figure 2 shows the variation in logarithm of OIT (ln OIT) with time for geomembranes immersed in leachate-1 at different temperatures. The standard deviation of each data point is represented by the vertical bars. The linear relationship between ln(OIT) and time represents the first-order decay law for antioxidant depletion. The slope of the line, s, represents antioxidant depletion rate. The depletion rate increases with increasing temperature indicating that OIT depleted at a faster rate with higher temperature, as has been observed by other researchers (Hsuan and Koerner, 1995 and 1998; Hsuan and Guan, 1998; Sangam and Rowe, 2002; and Gulec et al., 2004). At 850C and 700C, OIT depleted completely after around 4.5 and 20 months respectively. For all other temperatures OIT has not been depleted completely within the testing period (20 months). Similar trends were observed for all other leachates. Since, OIT was depleted after 4.5 months at 850C, the data points after that time are not included in establishing the regression line.

The depletion of OIT in four different leachates at all testing temperatures are shown in Figures 3-4. There was no statistically significant difference between the OIT data points for samples immersed in leachate-1 and leachate-4 which both contained fatty acids. There were some (albeit small) differences between leachate-1 and leachate-3; between leachate-1 and leachate-2; between leachate-2 and leachate-3; and between leachate -2 and leachate-4. The highest depletion rate was observed for samples immersed in leachate-2, which only contained trace metals and surfactant. Although the same amount of trace metals and surfactant was added to all four leachates, it appears that the presence of either fatty acids or inorganic nutrients, or both, may have reduced the antioxidant depletion rate although the effect is small enough that it may not be real. The significance of this is discussed below.

Although there is some variability in OIT data, there appeared to be a trend with the OIT depletion rates following the order (highest to lowest): leachate-2 (trace metals and surfactant only) > leachate-3 (same as 2 plus inorganic ions)> leachate-4 (same as 2 plus VFAs) > leachate-1 (full leachate). The reason for the highest depletion rate being for the simplest leachate (leachate-2) is not yet known and further research is needed to understand this phenomenon.

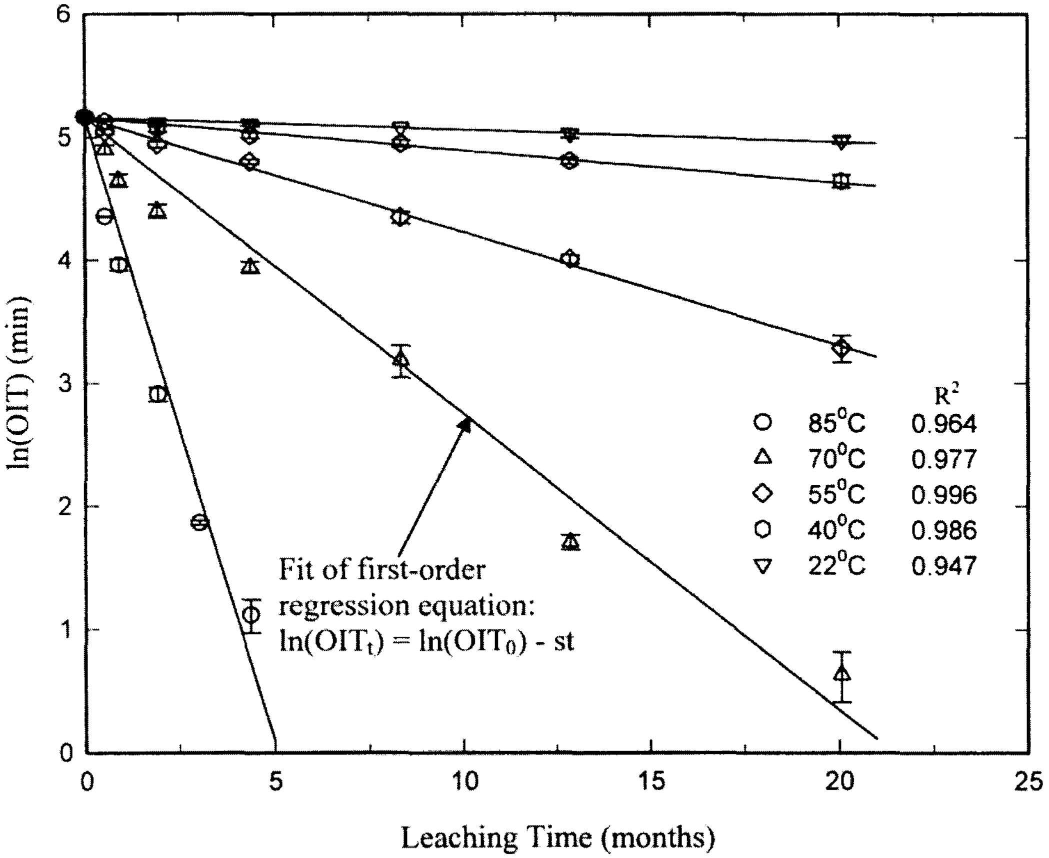

Figure 2. Variation in ln(OIT) with time at different temperatures in Leachate -1. OIT_0 is the initial OIT and OIT_t is the OIT at time t (month), s is the antioxidant depletion rate ($month^{-1}$).

4.2. Prediction of Antioxidant Depletion Time

The antioxidant depletion time at a temperature of interest (e.g. at expected field temperature) is usually predicted using the Arrhenius equation which describes the relationship between reaction rate and temperature (Hsuan and Koerner, 1998). The Arrhenius equation can be expressed as:

$$s = Ae^{-(E_a/(RT))} \qquad (9)$$

where s is the antioxidant depletion rate, A is a constant often called as collision factor, R is the universal gas constant (8.314 J/(mol K)), Ea is the activation energy of the depletion process

(J/mol), T is the absolute temperature (K). Equation (9) can also be written as follows by taking natural logarithm on both sides:

$$\ln(s) = \ln(A) - \left(\frac{E_a}{R}\right)\left(\frac{1}{T}\right) \qquad (10)$$

Plotting the variation in ln(s) with the inverse of absolute temperature (1/T) one can then get the best fit for equation (10) as shown in Figure 5 and tabulated in Table 3. The activation energy, Ea, varied between 61 and 64 kJ/mol for the four leachate immersed conditions. The lowest activation was energy observed for leachate-2 (trace metal and surfactant only) and the highest for leachate 1 (full leachate) immersed samples. However the relatively small difference in activation energies suggests that all leachates have a similar effect on antioxidant depletion.

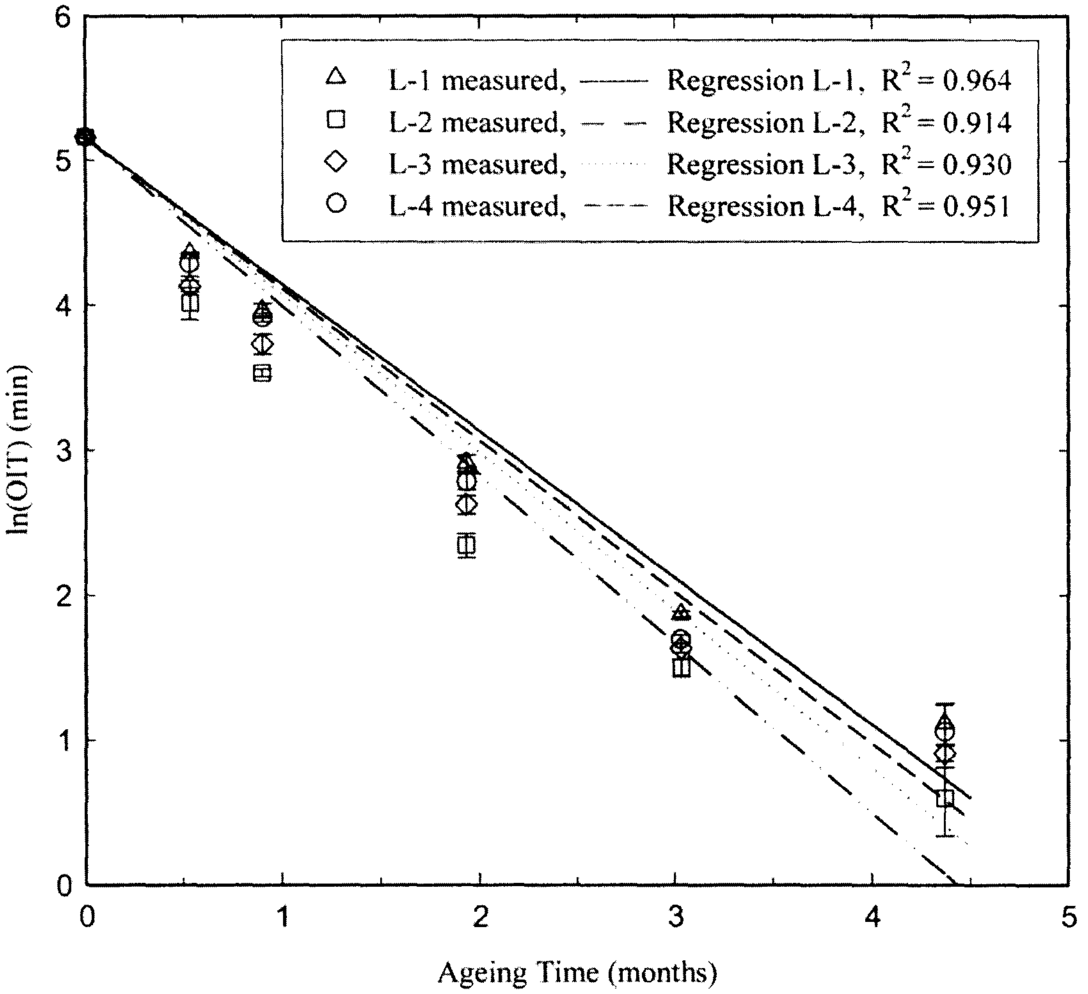

Figure 3. Variation in ln(OIT) with time at 85^0C in four different leachates.

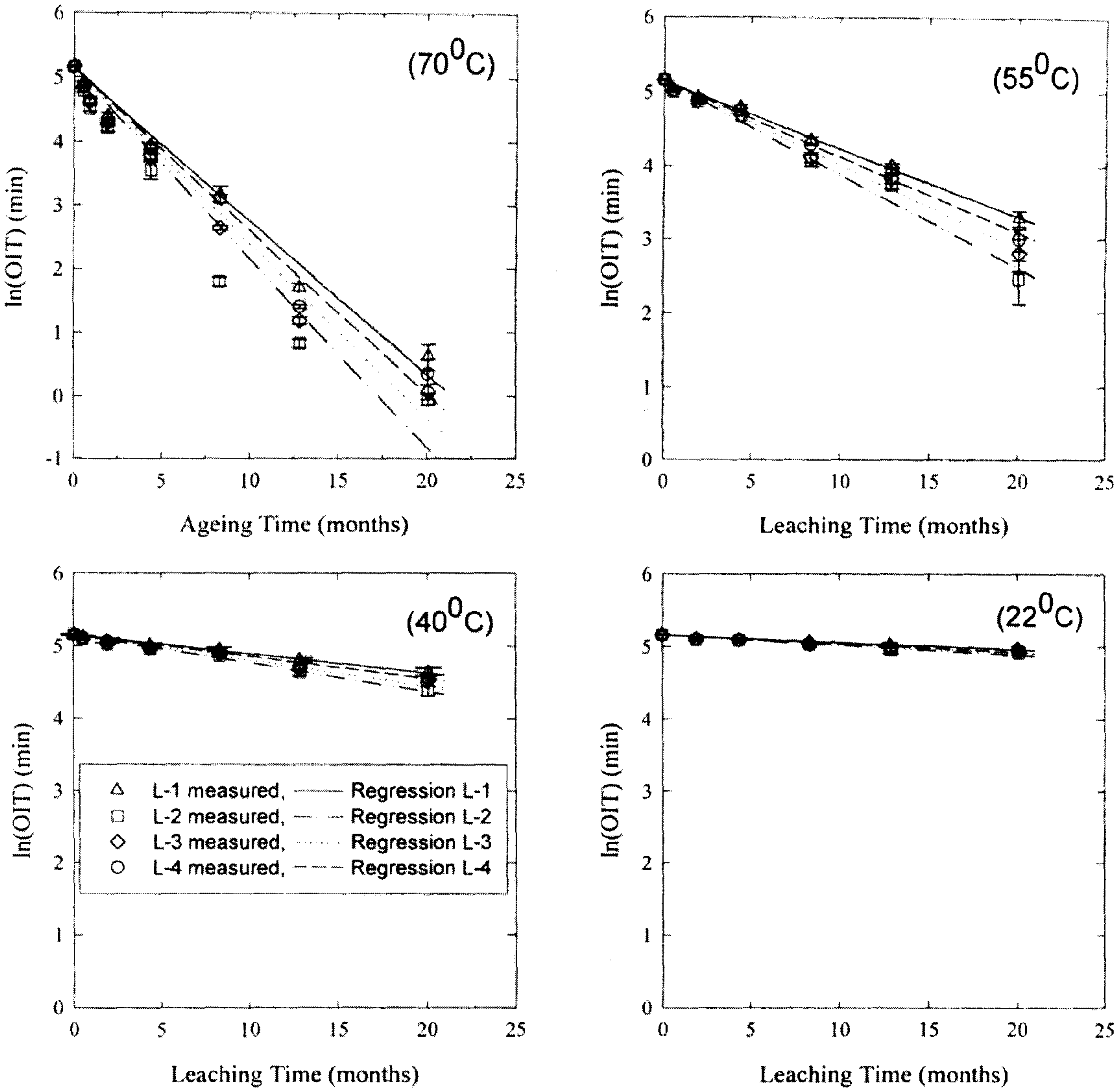

Figure 4. Variation in ln(OIT) with time at 22, 40, 55, and 70^{0}C in four different leachates.

Table 3. Activation energy and Arrhenius equation for four different leachates. (TM & S = trace metals and surfactant; II = inorganics; VFA = volatile fatty acids; Full = TM & S + II+VFA)

Leachate Type		Activation Energy,	Arrhenius Equation	R2
Leachate - 1	Full	64.02	ln(s) = 21.216 – 7700/T	0.9793
Leachate - 2	TM & S	60.81	ln(s) = 20.319 – 7315/T	0.9839
Leachate – 3	TM&S + II	61.91	ln(s) = 20.617 – 7447/T	0.9836
Leachate - 4	TM&S + VFA	61.84	ln(s) = 20.519 – 7438/T	0.9784

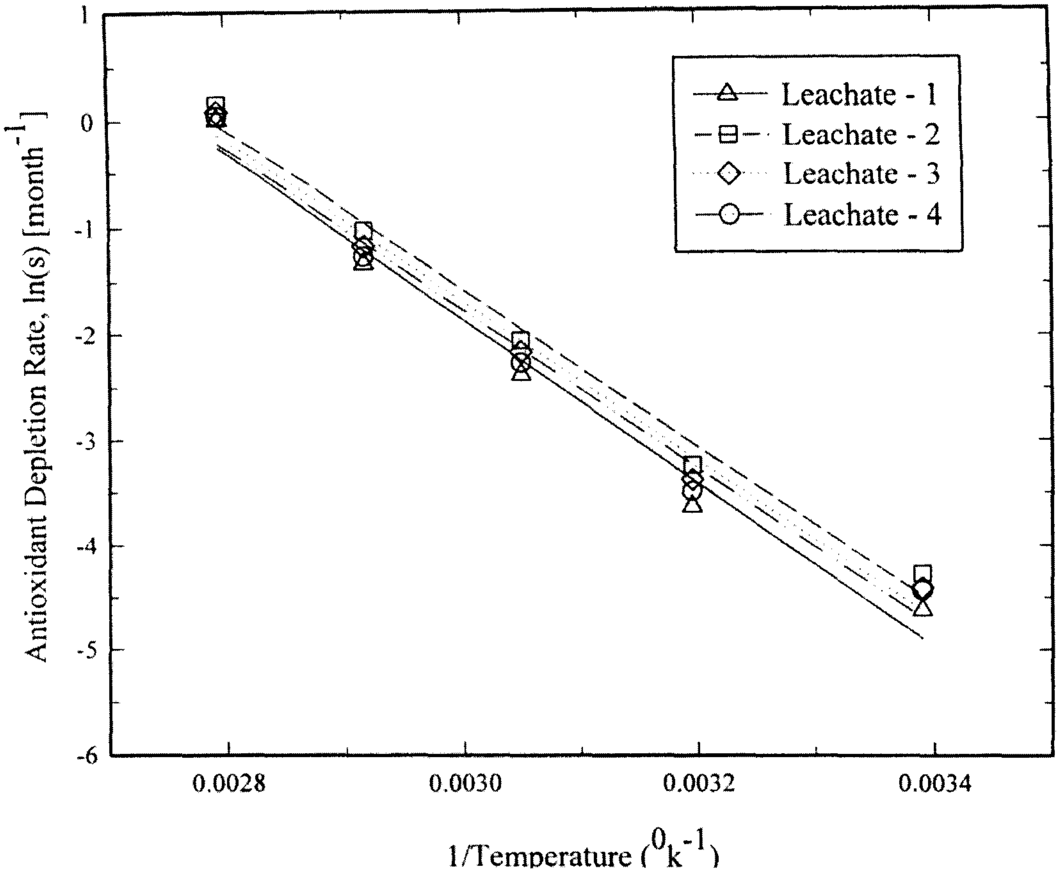

Figure 5. Arrhenius plot of antioxidant depletion in four different leachates.

The temperature at the base of the landfill depends on various factors such as waste types, age of the landfill, geologic conditions, performance of leachate collection systems etc. (Rowe, 2005). A wide range of temperature at the base of the landfill has been reported in the literature. Koerner and Koerner (2006) monitored the temperature of the geomembrane at two different cells (e.g., dry and wet cells) in the same MSW landfill in Philadelphia, USA. In the dry cell, where liquid was reduced intentionally, the temperature was 200C for the first 5.5 years and after that it rapidly increased to an average value of 320C. For the wet cell, where liquid was added at a constant rate, the average initial temperature was 250C and this then increased to between 41 and 460C over the next 3.7 years of monitoring. Rowe (2005) reported a temperature of 30-400C at the base of the Keel Valley Landfill in Ontario, Canada. Tokyo Fort Landfill in Japan had a temperature of 500C for the first 7-10 years of Landfilling (Yoshida and Rowe, 2003). The probable upper end of primary liner temperature may be anticipated to 600C where there is a significant leachate mound (Rowe, 2005). Considering the wide variations in landfill base temperature, antioxidant depletion time is predicted for three hypothetical base temperatures (e.g., 20, 40, and 600 C) in Table 4. It can be seen that the difference between antioxidant depletion times is not significant at 600C, however at lower temperatures the difference is as much as 25 years. As mentioned earlier, there is no statistically significant difference between OIT data values for Leachate 1 and 4 immersed samples. However, the difference in antioxidant depletion time based on the best fits to the data at 200C is 10 years (Table 4). This shows that when extrapolating over a wide range of temperatures the predictions based on the Arrhenius plot are sensitive to some natural variability in the data and thus while they indicate the relative magnitude of the depletion time, based on this data, any resolution beyond ± 5 years can not be justified at 20oC. Clearly the sensitivity decrease as the temperature increases. Therefore, reasonable assumption and care should be taken in predicting antioxidant depletion time using an Arrhenius relationship. Given the uncertainties regarding actual leachate composition in the field it can be seen the simplest leachate (#2) provides conservative, but reasonable predictions. It should be noted that a similar Arrhenius modelling based on tests with leachate #1 reported by Rowe (2005, Table 5) for a different geomembrane gave a depletion time of 25 years at 200C. Rowe's paper also makes the point that while ageing in leachate is good for assessing the relative performance of different geomembranes and effects of different leachates, it is a very severe test and the in-service life is likely to be much longer than predicted based on the immersion test and the interested reader is referred to Rowe (2005) for more details.

Table 4. Predicted antioxidant depletion times for different leachates at different temperatures.

Temp (0C)	Antioxidant depletion time (yrs)			
	Leachate-1	Leachate-2	Leachate-3	Leachate-4
20	75	50	60	65
40	15	10	10	15
60	3.3	2.5	2.8	3.0

5. CONCLUSIONS

An experimental investigation on the depletion of antioxidant from HDPE geomembrane immersed in four synthetic leachates at five different temperatures (22-850C) is described in this paper. Results of immersion test showed that the difference of activation energies in four leachates were quite small (61 and 64 kJ/mol). This indicates that the four leachates were very similar in term of OIT depletion. The lowest antioxidant depletion time was observed for the simplest leachate (#2 which consisted of trace metal solution and surfactant). Given the uncertainty regarding actual leachate compositions in the field, it would appear that this leachate could reasonable and conservatively used for assessing potential depletion rates of different geomembranes and exposure conditions.

Acknowledgements

This study was financed by the Natural Sciences and Engineering Research Council of Canada (NSERC). The authors are grateful to Terrafix Geosynthetics Inc., Toronto for donating the geomembrane used in this study.

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