Antioxidants in an HDPE geomembrane used in a bottom liner and cover in a PCB containment landfill for 25 years

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ABSTRACT
HDPE geomembranes exhumed from the cover and bottom liner of a hazardous waste landfill containing PCB contaminated wastes and soil after 25 years are examined. This paper focuses attention on the antioxidants added to a geomembrane to delay polymer oxidation and extend its service life. The extent of antioxidant depletion is monitored in terms of the standard oxidative induction time (Std-OIT) and high pressure oxidative induction time (HP-OIT). The geomembrane in the cover was overlain by about one meter of soil and was exposed to moisture and air in the overlying soil. The geomembrane in the bottom liner was exposed to leachate at a relatively constant temperature. The differences between OIT values of the cover geomembrane and the base liner geomembranes are compared and the differences relative to each other and the initial OIT values of modern geomembranes are discussed.

RÉSUMÉ
On examine des géomembranes en PEHD prélevées dans la couverture et l'étanchéité de fond d'une installation de stockage de déchets dangereux contenant des déchets contaminés par le PCB et du sol, après 25 ans. Cet article se concentre sur les antioxydants ajouté à la géomembrane afin de retarder l'oxydation du polymère et de prolonger sa durée de vie. La consommation de l'antioxydant est quantifiée en termes de temps d'induction d'oxydation standard (Std-OIT) et temps d'induction de l'oxydation sous haute pression (HP-OIT). La géomembrane de la couverture était recouverte d'environ un mètre de sol et exposée à l'humidité et à l'air présents dans le sol la recouvrant. La géomembrane de l'étanchéité de fond était exposée au lixiviat à une température relativement constante. Les différences entre les valeurs d'OIT pour la géomembrane de la couverture et celle de l'étanchéité de fond sont comparées et les différences de chacune par rapport à des valeurs initiale d'OIT pour des géomembranes modernes sont discutées.

1 INTRODUCTION

Composite liners containing a high density polyethylene (HDPE) geomembrane have proved to be highly effective in minimizing migration of various contaminants from waste containment facilities (Hsuan et al. 2004). Although landfills containing municipal solid waste or various industrial and hazardous wastes are designed for a predetermined capacity, their contaminating lifespan will invariably extend well beyond the facility closure. Accordingly the service life of geomembranes is a matter of considerable interest to designers and regulators.

The polymer in HDPE geomembrane liners will be subject to oxidative degradation with time and eventually the liner will fail (Hsuan and Koerner, 1998, Hsuan et al. 2004, Rollin, 2004, Peggs et al. 2002, Rowe et al. 2010). To extend the service life of the geomembrane by delaying the induction of polymer oxidation/degradation, about 0.5% of antioxidants are typically added to the HDPE resin. The role of antioxidants has been well described in Hsuan et al. (2004). Geomembrane degradation is often considered to follow a three stage process (Hsuan and Koerner, 1998). During Stage I, the antioxidants are depleted. Antioxidants are prone to extraction from geomembrane through oxidation, diffusion/extraction and other physical and chemical reactions with leachate constituents. Stage II is the induction time to the onset of polymer degradation after the antioxidants cease to provide protection from free-radicals. Stage III involves a readily measurable degradation of physical characteristics (such as stress crack resistance) of the geomembrane to a point where it is considered to have ceased to be able to adequately perform its design function as a barrier (Hsuan et al. 2004 and Rowe and Sangam, 2002). The sum of the three stages is defined as the service life of a geomembrane.

The service life of geomembranes can be evaluated based on laboratory scale experiments (Rowe et al. 2010). Accelerated ageing experiments at elevated temperatures are often conducted by immersion of a given geomembrane in a fluid similar to that is expected in field. The rate of ageing is then evaluated based on monitoring the geomembrane properties including but not limited to Oxidative Induction Time (OIT), Stress Crack Resistance (SCR), Melt Index (MI) and Crystallinity. Antioxidant depletion is mainly characterized by the level of OIT retained in a given geomembrane sample (Mueller and Jakob, 2003). Considering the various environmental and operational factors influencing geomembrane ageing under field conditions, analysis of geomembranes having been in service as landfill barriers can provide further insight regarding the rate of geomembrane ageing and their service life (Rowe et al. 2003). A unique opportunity was provided to examine the state of antioxidants in the geomembranes used as part of the composite barrier system in a PCB waste landfill after 25 years of operation. Upon exhumation of the landfill in 2009, samples of geomembranes used in final cover, bottom liner and sidewalls were analyzed for OIT in order to characterize the state of the geomembranes.

2 PCB WASTE LANDFILL

A landfill-style PCB contaminated soil storage site was constructed to dispose of PCB contaminated soil, sediment and debris cleaned up from the Pottersburg Creek area in London Ontario, Canada during the 1980's. The storage cells were constructed, filled and capped over a four year period (i.e., 1984, 1985, 1986 and 1987). Cells 1 and 2 constructed in 1984 and 1985 were lined with a clayey liner whereas a 1.5 mm HDPE geomembrane was used as part of the composite liner system in Cells 3 and 4. All cells were capped with a composite liner system which included a 1.5 mm HDPE geomembrane. The configuration of cover, bottom liner and sidewalls of the cells is schematically shown in Figure 1. Cells were exhumed in 2009 to allow final destruction (by incineration) of the waste material.

The geomembranes in the cover were subject to exposure to oxygen due to the unsaturated and partially desiccated nature of the overlying soil and were also subject to (dampened) seasonal temperature fluctuations as well as precipitation/ evaporation.

Layer	Thickness (cm)
Topsoil	15
Common Fill Layer	45
Woven Geotextile*	
Sand – Drainage Layer	30
HDPE Geomembrane	1.5 mm
Sand - Drainage Layer	15
Clay Cap	60
Waste	

* Terratrack 2415 Filter Fabric

(a)

Layer	Thickness (cm)
Waste	
Woven Geotextile*	
Upper Sand LCS**	15
HDPE Geomembrane	1.5 mm
Lower Sand LCS**	10
Compacted Clay	60
Woven Geotextile*	
Sand	30
Compacted Clay	30

* Terratrack 400W Filter Fabric
** Leachate Collection System

(b)

Waste
Nonwoven Geotextile*
HDPE Geomembrane**
Nonwoven Geotextile*
Clay Sidewall

* AMOCO 4554 Filter Fabric
** 1.5 mm thick

(c)

Figure 1. Schematic configuration of (a) final cover, (b) bottom liners and (c) sidewalls of exhumed PCB waste landfill

The geomembranes in the bottom liner were well insulated from seasonal temperature fluctuations and the overlying soil would have limited oxygen migration to the geomembranes, however they were exposed to levels of leachate containing low concentrations of PCB. A relatively constant temperature could be expected for the bottom geomembrane since no exothermic chemical or biological reactions were expected in the waste soil. The sidewall geomembranes were exposed to waste material containing PCBs on top and to unsaturated soil below. However it can be assumed that sidewall geomembranes were subject to a relatively constant temperature.

Two large (i.e., approximately 1 by 1 m) geomembrane samples were obtained from the cover, bottom liner and sidewall, as appropriate, from each cell. The cover geomembranes were found to be scratched. Given the care taken during exhumation, the scratching is attributed to the initial construction. The bottom liner geomembranes also were scratched although to a much lesser extent than the cover.

Basic properties of the exhumed samples are shown in Table 1 along with a comparison to some modern day geomembranes. Due to lack of historical information on the initial geomembrane properties at the time of installation (including MI, crystallinity and OIT) it is not possible to infer the level of change compared to the initial properties.

All the geomembrane samples from the field had statistically similar MI values (the average of 0.11 g/10 min). Crystallinity of the exhumed geomembranes was higher than that of modern day geomembranes. Due to lack of information on the initial crystallinity value of the geomembranes, it is not clear whether the higher value is a result of physical ageing or because the crystallinity of geomembranes manufactured at that time were generally higher than today. However while some increase in crystallinity due to physical aging is certainly possible, it is suspected that most of the difference compared to modern geomembranes is due to a higher initial crystallinity of the manufactured geomembrane in the early 1980's.

Table 1. Basic properties of geomembranes exhumed from the PCB waste disposal site compared to modern day geomembranes

Property	Unit	Geomembrane from PCB waste disposal site	Selected typical modern day geomembranes		
			GM1	GM2	GM3
Thickness (ASTM D5199)	mm	1.58 ± 0.04	1.5	1.5	1.5
Density (ASTM 792-08)	kg/m^3	959 ± 8	947	946	944
Crystallinity (ASTM E794)	%	56.6 ± 1.6	47.6 ± 1.4	37.7 ± 1.9	52
Melt Index (ASTM D1238)	g/10 min	0.11 ± 0.01	-	0.43 ± 0.03	0.47

3 OIT OF THE GEOMEMBRANE AFTER 25 YEARS

The main function of antioxidants is to prevent initiation of oxidation chain reactions. Antioxidants are most effective over a certain range of temperature. As an instance, phosphites are most effective at higher temperatures whereas hindered amine light stabilizers (HALS) are effective at ambient temperature. Hindered phenols however are used as long term stabilizers since they are effective over a wide range of temperature (Mueller and Jakob, 2003).

Depletion of OIT has been shown to be a function of antioxidant concentration (Mueller and Jakob, 2003). Thus the level of OIT in a geomembrane sample could be employed as an indicator of remaining antioxidant concentration. Since the exact composition of antioxidants is generally unknown to users, OIT values are measured to evaluate the antioxidant depletion rate (Hsuan et al. 2004). There are two methods of OIT measurement; standard OIT (ST-OIT) and high pressure OIT (HP-OIT) measured as per ASTM D3895 and ASTM D5885 respectively. These values are measured using a Differential Scanning Calorimeter (DSC). The ST-OIT test is performed by heating a small piece of geomembrane specimen from room temperature to 200 °C at a rate of 20 °C/min in a nitrogen environment with a gas flow rate of 50 ml/min. The gas flow is then changed from nitrogen to oxygen under isothermal conditions at 35 kPa pressure. The HP-OIT test follows a similar procedure; however it is conducted at temperature and pressure of 150 °C and 3500 kPa, respectively. The HP-OIT test is used to quantify antioxidants, such as thiosynergists or hindered amines, that volatilize at the higher temperatures used in the ST-OIT test (Hsuan and Koerner, 1998).

Typical values of ST-OIT and HP-OIT for some commercially available modern geomembranes are shown in Table 2. Depending on the preferences of the geomembrane manufacturer or user the values of HP-OIT can be significantly higher. The last column in Table 2 (HP-OIT/ST-OIT) shows that ratio of the HP-OIT to ST-OIT of modern virgin geomembranes can vary substantially signalling very different antioxidant packages. Since the publication of the first release of GRI-GM13 in 1997, ST-OIT values have normally been over 100 min for a virgin geomembrane.

Table 2. Examples of OIT values of modern virgin geomembranes

Geomembrane	ST-OIT (min)	HP-OIT (min)	HP-OIT / ST-OIT
GM-1	135 ± 2.2	244 ± 13	1.8
GM-2	174 ± 1.9	903 ± 24	5.2
GM-3	135	227	1.7

Unfortunately the construction documentation at the time was not sufficient to know whether the same geomembrane was used in each year's construction or even if the same geomembrane was used throughout for a given year although one would expect that to be the case. This needs to be kept in mind when comparing the data discussed below. Table 3 shows the average ST-OIT and HP-OIT values and the ratio of HP-OIT/ST-OIT for the cover geomembranes retrieved from the PCB waste landfill. The ST-OIT was found to be considerably lower than that of typical modern day geomembranes. The HP-OIT of the cover geomembranes was also lower than typical of today's virgin geomembranes. The ratio of HP-OIT/ST-OIT was similar for four of the five locations. Cell 1 being the oldest cell has a relatively lower values of OIT compared to Cells 2, 3 and the western part of Cell 4 (i.e. Cell 4-1). Geomembrane sampled from eastern part of Cell 4 (i.e. Cell 4-2) had the lowest OIT even compared to the oldest geomembrane (i.e. Cell 1). Analysis of variance (ANOVA) indicated that statistically there was no significant difference between the average ST-OIT values of cover geomembrane exhumed from Cells 2, 3 and 4-1 however the same statement only applies to the HP-OIT of the geomembrane exhumed from Cells 2 and 3. The data for Cell 4-1 (Table 3) and Cell 3-2 sidewall (Table 4) suggest that the initial HP-OIT of geomembranes in the early 1980's could be about 200 min (or more).

Table 3. OIT of the exhumed cover geomembranes from PCB waste disposal landfill

Geomembrane	Age (year)	ST-OIT (min)	HP-OIT (min)	HP-IT / ST-OIT
Cover-Cell 1	25	15 ± 1.6	114 ± 10	7.6
Cover-Cell 2	24	24 ± 1.5	155 ± 7	6.5
Cover-Cell 3	23	27 ± 4.6	163 ± 15	6.0
Cover-Cell 4-1	22	25 ± 1.9	193 ± 5	7.5
Cover-Cell 4-2	22	9 ± 1.2	121 ± 13	14

The average OIT values and the ratio of HP-OIT/ST-OIT for the bottom liner and sidewall geomembranes are presented in Table 4. The ST-OIT and HP-OIT of the exhumed geomembranes and those of modern day virgin geomembranes are shown in Figures 2 and 3 respectively. As illustrated by the very high HP-OIT of GM-2 in Table 2, GM-2 has a very different antioxidant package than the other two modern geomembranes listed and was not considered as part of the comparison basis in Figure 2 and 3.

Hypothesizing that a very similar geomembrane was used for all cells, it would appear that the details of the exposure conditions can significantly influence the OIT depletion. Of the samples examined, the least antioxidant depletion occurred for the side walls of Cell 3 where a ST-OIT of about 60 minutes and HP-OIT of about 190 minutes remained after 23 years.

The bottom of Cell 4 experienced a little more depletion than the side walls of Cell 3 (presumably due to contact with leachate) but otherwise appear fairly similar. Both the ST- and HP-OIT values for Cell 3 bottom liner (ST-OIT about 7 minutes and HP-OIT about 66 minutes) are considerably lower than those on the Cell 3 side walls or Cell 4 bottom and the ratios of HP-OIT/ST-OIT are very different.

Table 4. OIT of the exhumed bottom liner and sidewall geomembranes from PCB waste disposal landfill

Geomembrane	Age (year)	ST-OIT (min)	HP-OIT (min)	HP-OIT / ST-OIT
Bottom Liner-Cell 3-1	23	7± 0.8	68 ± 3.4	9.2
Bottom Liner-Cell 3-2	23	8 ± 1	64 ± 3.7	8.0
Bottom Liner-Cell 4-1	22	53± 1	143 ± 26	2.7
Bottom Liner-Cell 4-2	22	52 ± 0.5	162 ± 16	3.1
Sidewall-Cell 3-1	23	62 ± 0.7	188 ± 21	3.0
Sidewall-Cell 3-2	23	60 ± 0.9	197 ± 9.7	3.3

Although it was expected that the Cell 3 and 4 bottom geomembranes were exposed to relatively similar conditions, if the geomembrane used in the base was the same as that used in the side walls and cover for Cell 3, then there was probably something significantly different in the leachate exposure conditions at the bottom of Cells 3 and 4 to cause the significant depletion of antioxidants implied by both ST-OIT and HP-OIT values for Cell 3. The most important factors influencing OIT depletion are temperature and presence of chemicals such as surfactants. Based on limited historical data available in terms of leachate composition and temperature over the bottom liner, it is practically impossible to identify the causes of faster depletion of OIT in this liner. Another possible explanation is that a different geomembrane with a lower initial OIT value was used in Cell 3 or it has been left exposed to atmospheric conditions for an extended period of time prior to installation. These however cannot be verified due to lack of historical information. The HP-OIT/ST-OIT ratio would suggest that the antioxidants reflected by the ST-OIT test depleted faster that those only captured by the HP-OIT test.

If, based on GM1 and GM3 in Table 2, the initial ratio of HP-OIT/ST-OIT was about 2, then it would appear that the antioxidant represented by ST-OIT depleted faster than those represented by HP-OT since after 23 years the ratio was about 3 for the side wall of Cell 3 and bottom of Cell 4. In the cover of Cell 3 there was much more substantial depletion of ST-OIT to about half that in the side walls (about 30 min versus about 60 min). HP-OIT depleted more in the cover than in the side wall but slower than ST-OIT so that the ratio of HP-OIT/ST-OIT in the cover was about 6.

For the cover in Cell 4, there was a significant difference between the eastern and western locations. At Cell 4-1, The HP-OIT of the cover was similar to that in the sidewall of Cell 3 and the ST-OIT was similar to that in the cover of Cell 3 At Cell 4-2 there was substantially more depletion of both ST- and HP-OIT with the former depleting faster (ratio of HP-OIT/ST-OIT of 14).

Assuming a similar geomembrane was used in all locations, it would appear that exposure conditions dominated the change in both ST- and HP-OIT values and that while there may be a difference in depletion that can be attributed to the geomembranes in Cell 1 being older than those in Cell 4, it is hard to distinguish this effect given the substantial difference that are related to location (and hence exposure) for the two samples from the cover of Cell 4.

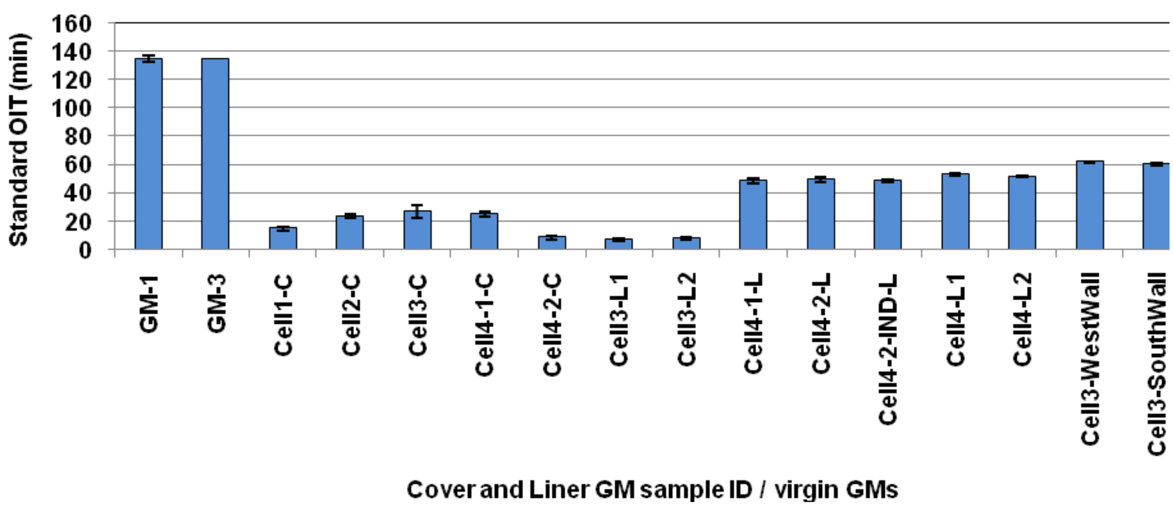

Figure 2. Standard OIT of modern and exhumed geomembranes

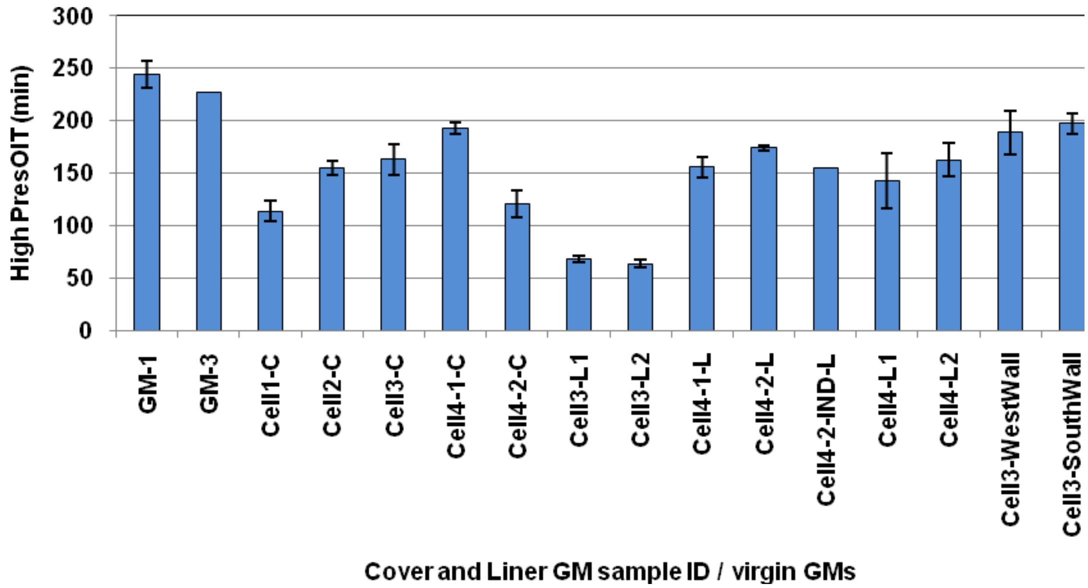

Figure 3. High pressure OIT of modern and exhumed geomembranes

The alternative hypothesis that the geomembrane used in the base of Cell 3 was different to that used anywhere else, including the side walls, can not be excluded but seems much less likely than the hypothesis that same geomembrane being used in Cell 3 (especially for bottom and side walls). Likewise it is possible that different geomembranes were used in the east and west of the cover of Cell 4, however this is considered less likely than that there was a substantial difference in exposure conditions. This matter is being further investigated.

4 ESTIMATED OIT DEPLETION RATES

OIT depletion is the first stage of geomembrane degradation and it follows a first order kinetic model as shown in Equation 1 (Hsuan and Koerner, 1998).

$$OIT_t = OIT_0 e^{-st} \quad [1]$$

where:
OIT_t : OIT (min) at time t (month)
OIT_0 : initial OIT (min) of the geomembrane
s: OIT depletion rate (month^{-1})

The rate of geomembrane degradation is known to be a function of temperature as well as exposure medium (Hsuan and Koerner, 1998 and Rowe et al. 2010). The rate of antioxidant depletion as well as polymer degradation increases with temperature (Hsuan and Koerner, 1998). Furthermore it has been shown through laboratory scale accelerating ageing experiments that antioxidants deplete faster in synthetic leachate resembling municipal solid waste landfill leachate, water and air respectively (Rowe et al. 2009).

Initial value of ST-OIT of the geomembrane used in this landfill are not known, however a modern day geomembrane is expected to have ST-OIT values of greater than 100 min. Adams and Wagner (2000) indicated that in the 1980s when the geomembrane being discussed in this paper was manufactured and installed, the ST-OIT of HDPE geomembranes was typically around 50 min. Nevertheless, taking into account the fact that in many cases, the ST-OIT of the retrieved geomembranes was higher than 50 min after 24 years, a typical value of 100 min was assumed.

Based on the above assumptions, antioxidant depletion rates, the total time to complete depletion of antioxidants as well as time to complete depletion of antioxidants after exhumation were estimated for the retrieved geomembranes (Table 6). The latter can be an indicator of approximately how long the antioxidants could protect the geomembrane from oxidative degradation if kept in service. In order to estimate the remaining time to complete depletion of antioxidants after exhumation a residual ST-OIT value of 0.5 min was considered based on Hsuan and Koerner (1998).

5 CONCLUSION

Presence of antioxidants in geomembranes retrieved from a PCB waste landfill was examined after 25 years through OIT analyses. The standard OIT levels and some HP-OIT levels were found to be significantly lower than those of modern day virgin geomembranes. If one assumes that the same or a very similar geomembrane was used for Cells 3 and 4, then the data suggest that the exposure conditions can significantly influence the OIT depletion. The least severe condition at this site appears to have been on the sidewall where (relatively) high values of both ST-OIT (about 60 minutes) and HP-OIT (about 190 minutes) remained after 23 years. In the cover of Cell 3 there was substantial depletion of ST-OIT to about half that in the side walls (about 30 min versus about 60 min.). HP-OIT depleted more in the cover than in the side wall but not at the same rate as ST-OIT so that the ratio of HP-OIT/ST-OIT in the cover was about 6 compared to 3 in the side slope. For the cover in Cell 4, there was a significant difference between the eastern and western locations. At Cell 4-1, the HP-OIT was similar to that in the side wall of Cell 3 and the ST-OIT was similar to that in the cover of Cell 4. At Cell 4-2 there was substantially more depletion of both ST and HP-OIT with the former depleting faster (ratio of HP-OIT/ST-OIT of 14). The greatest antioxidant depletion was in the base of Cell 3. Here the ST-OIT was down to 7-8 minutes and HP-OIT to 60-70 minutes. In contrast at Cell 4 at the bottom, the ST-OIT was still about 50 minutes and HP-OIT about 140-160 minutes and hence only a little lower than in the sidewall of Cell 3. Thus it appears that there was some interaction with the waste/leachate at the bottom of Cell 3 that led to much greater depletion of antioxidants than in the side slope or cover of the same cell or in the base or cover of Cell 4. At the time of writing the explanation for this is unknown.

Table 6. Approximated rates and times to complete depletion of OIT for geomembranes exhumed from the PCB waste disposal site

Geomembrane	Estimated OIT depletion rate (month^{-1})	Estimated Total time to complete depletion of OIT (years)	Estimated Time to complete depletion of OIT after exhumation (years)
Cover-Cell 1	0.0063	50	26
Cover-Cells 2, 3 & 4-1	0.0050	65	41
Cover-Cell 4-2	0.0093	35	11
Average - Cover	0.0068	50	26
Bottom liner-Cell 3	0.0093	30	6
Bottom liner-Cell 4	0.0024	100	76
Average - bottom liner	0.0059	65	41
Sidewall-Cell 3	0.0018	230	206
Overall average	0.0057	85	61

In this case, the average remaining time to complete depletion of ST-OIT was estimated to be about 60 years. However, the antioxidants in parts of the cover geomembrane and Cell 3 bottom liner would have been expected to be depleted in about a decade or less had the landfill remained in operation. It must be noted that this is only Stage I of the service life of the geomembrane and that the overall service life of the geomembranes (i.e. reaching to failure state) will be longer, as the second and third stage of geomembrane degradation should be added to antioxidant depletion time.

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