

## DURABILITY OF GEOMEMBRANE EXPOSED TO JET FUEL A-1

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### ABSTRACT

A fluorine surface treated HDPE geomembrane was selected as a part of an experimental subsurface composite geosynthetic barrier including a Geosynthetic Clay Liner (GCL) to limit migration of hydrocarbons to nearby water prior to future site remediation. The durability and long term performance of the HDPE geomembrane exposed to jet fuel is of particular interest. Samples of 1.5 mm thick HDPE geomembrane (both untreated and fluorinated) were exposed to jet fuel in the laboratory. The change in oxidative induction time (OIT), tensile properties, crystallinity and mass were monitored at selected time intervals. The results indicated that immersion in jet fuel accelerates the antioxidant depletion rate relative to that observed in water or municipal solid waste (MSW) leachate. It was found that antioxidant depleted at much faster rate from untreated geomembrane than fluorinated geomembrane showing that fluorination had beneficial effect. Tensile test results indicated that immersion to jet fuel decreases the yield stress and the increases the strain at yield.

### RÉSUMÉ

Un geomembrane de HDPE traité par surface de fluor a été choisi comme partie d'une barrière geosynthetic composite au-dessous de la surface expérimentale comprenant un recouvrement d'argile de Geosynthetic (GCL) pour limiter la migration des hydrocarbures pour arroser tout près avant la future remédiation d'emplacement. La longévité et l'exécution à long terme du geomembrane de HDPE exposé au carburant pour réacteurs est d'intérêt particulier. Des échantillons de 1,5 millimètres de geomembrane épais de HDPE (non traité et fluoré) ont été exposés au carburant pour réacteurs dans le laboratoire. Le changement des temps d'induction d'oxydation (OIT) les propriétés de tension de la cristallinité et de la masse ont été surveillés à intervalles choisis de temps. Les résultats ont indiqué dans que l'immersion en carburant pour réacteurs accélère le taux antioxydant d'épuisement relatif qui a observé l'eau ou leachate municipal des déchets solides (MSW). On l'a constaté qu'antioxydant épuisé à une vitesse beaucoup plus rapide de geomembrane non traité que l'apparence fluorée de geomembrane que le fluorination a eu l'effet bénéfique. Les résultats d'essai de tension ont indiqué que l'immersion au carburant pour réacteurs diminue l'effort d'fléchissement et les augmentations la contrainte au rendement.

### 1. INTRODUCTION

High Density Polyethylene (HDPE) geomembranes are extensively used as a part of liner systems for modern landfills, heap leach pads, and leachate lagoons, as well as for spill protection around chemical storage tanks. In these applications the HDPE geomembrane is used to limit the migration of contaminants into surface or groundwater. In short-term tests HDPE geomembranes demonstrate a relatively high chemical resistance to leachate components and organic solvents (Koerner, 1998; Tisinger *et.al.*, 1991). However the geomembrane used as liner for contaminant barrier should not only have high chemical resistance and low permeability to the contaminants but they also must maintain their durability over the designed service life of the engineered facility. Both laboratory tests and field studies have shown that HDPE geomembranes change with time (Hsuan *et.al.*, 1991; Tisinger *et.al.*, 1991; Hsuan and Koerner, 1998; Sangam and Rowe, 2002; Rowe *et.al.*, 2003).

Use of HDPE geomembranes in hydrocarbon contaminated sites and their performance and durability under long term exposure condition is of growing interest. For example, at a former Canadian Distant Early Warning Line (DEW Line) site on Brevoort Island (east of Baffin

Island) in the Canadian Arctic, an area of hydrocarbon contaminated ground was identified by the Environmental Sciences Group (ESG) at Royal Military College of Canada (ESG, 2000; Li, *et.al.*, 2002). The contaminant distribution indicated that the hydrocarbon plume was moving from the site of decommissioned tanks towards the bay. Site remediation by excavation of contaminated soil followed by ex-situ treatment is planned however due to the difficulties of access and the limited construction season a short term strategy was needed to contain the contaminant plume until the remediation work could be undertaken. This strategy involved the installation of a trial geocomposite barrier system including geotextile, geomembrane and geosynthetic clay liner (GCL) to contain the plume (Li, *et.al.*, 2002).

A fluorinated HDPE geomembrane was selected as the primary liner for the subsurface barrier. The fluorinated polyethylene is more resistant to the diffusion of aromatic hydrocarbons than conventional untreated HDPE geomembrane liners (Sangam, *et.al.*, 2001). The primary questions relating to the use of this trial geocomposite liner concern the potential impact of interaction with the hydrocarbons and the impact of the extreme climatic conditions of the north on the durability and service life of the lining system. This barrier system has now been in

operation for three years since 2001 and appears to be working well. However there is a need to assess how long this temporary measure can be expected to work and this requires an assessment of how the HDPE geomembrane is likely to be affected by the interaction with jet fuel and the consequent likely effect on its long term performance.

The degradation of HDPE geomembranes proceeds in three distinct stages (Hsuan and Koerner, 1998), viz: Stage (I) depletion of antioxidants; Stage (II) onset of the degradation; Stage (III) degradation to failure changes of physical or mechanical property to an arbitrary level of 50% of the original value). Geomembrane are typically composed of 96-97.5% polyethylene resin, 2-3% carbon black, and 0.5-1% antioxidants and heat stabilizers. The antioxidants provide the protection against oxidative degradation during processing and prevent oxidative reactions during the first stage of degradation (Hsuan and Koerner, 1998). After the antioxidants are removed, the oxidation will begin to degrade the polyethylene.

In this study two different HDPE geomembranes (untreated and fluorinated) were exposed to jet fuel A-1 in laboratory tests. Geomembrane coupons were immersed in the jet fuel and located inside a fumehood at ~23°C. The immersed samples were periodically removed from the container and tested to assess the change in: oxidative induction time (OIT), crystallinity, tensile properties, and dimensions. The objective of this paper is to examine antioxidant depletion and the compatibility of both untreated and fluorinated HDPE geomembranes with jet fuel A-1.

## 2. ANTIOXIDANT DEPLETION FROM HDPE GEOMEMBRANE EXPOSED TO LIQUID CHEMICAL ENVIRONMENT

There are two simultaneously occurring primary mechanisms associated with the depletion of antioxidants from a geomembrane, viz: (1) chemical reactions, and (2) physical loss. The antioxidants depletion from a polymer can be attributed to various factors including chemical reaction with oxygen, oxidizing agents, free radicals and alkyl peroxides, and/or physical processes like extraction, diffusion or volatilization (Hsuan and Koerner, 1998; and Sangam and Rowe, 2002). For geomembrane samples exposed to air, depletion is mainly due to volatilization and chemical reactions (Sangam and Rowe, 2002). For geomembranes in a liquid environment, extraction is the dominant depletion mechanism. The extraction process involves the removal of antioxidants and other additives from the polymer material by interaction with the liquid chemical. Tisinger and Giroud (1993) noted that most modern antioxidants having high molecular weight are physically entangled among the polyethylene molecules which reduces the ability of chemicals to extract antioxidants. However the rate of extraction can be accelerated by increasing concentration or temperatures. After depletion of antioxidants the geomembrane will be susceptible to further degradation by oxidation (Grassie and Scott, 1985).

The principal factors affecting the loss of antioxidants due to the extraction at the geomembrane surface are: the diffusivity of the antioxidants in the HDPE, and swelling of the geomembrane which enhances the molecular mobility of the chemicals (Maisonneuve *et al.*, 1997). When geomembrane is in contact with liquid hydrocarbon for a long period of time, swelling can occur due to sorption. Prior studies have shown that many non-polar organic substances and particularly volatile organic compounds can readily migrate through the geomembrane liner (Park and Nibras, 1993; Rowe *et al.* 1995; Sangam, 2001; Sangam *et al.* 2001). The migration of organic compounds through the geomembrane occurs due to the three different processes of adsorption, diffusion and desorption which can cause swelling, stiffening, or softening of geomembrane (Tisinger *et al.*, 1991). But swelling does not necessarily mean chain scission or degradation of geomembrane to failure (Koerner, *et al.*, 1990). However the mobility of antioxidants and chemical molecules contained in the liquid media within the geomembrane will be enhanced due to swelling. HDPE is a semicrystalline polymer. Hsuan and Koerner (1998) observed that, due to the presence of crystalline and amorphous phases within the material, the distribution of antioxidants in HDPE is not uniform. The amorphous region of an HDPE contains the greatest concentration of antioxidants and the mobility of antioxidants in the amorphous phase controls their extractability. Amorphous regions can accommodate absorbed liquids without causing major changes in physical and mechanical properties but in some cases this can result in small loss of strength but the effect is more or less reversible when the liquid is removed by evaporation (Koerner, *et al.*, 1990; Tisinger and Giroud, 1993). The geomembrane ageing studies of Sangam and Rowe (2002) have shown that the synthetic leachate (chemical exposure) causes faster extraction of antioxidants from the geomembrane than exposure to distilled water or air. The OIT test results indicated that the depletion of antioxidants followed first-order decay kinetics. The extraction process plays a significant role when the geomembrane is in a liquid environment. The degree of crystallinity of polymer is a dominant factor in reducing swelling of polymer in organic solvents (Tisinger *et al.*, 1991). A semicrystalline polymer like HDPE reaches the finite sorption level whereas in a completely amorphous polymer will have infinite sorption of a liquid with identical solubility parameter (Stessel and Garcia-Rubio, 1994). This occurs in HDPE because crystalline region in HDPE does not adsorb liquids. Thus the crystalline zones in HDPE act as an impermeable barrier to the permeating liquid molecules.

## 3. EXPERIMENTAL INVESTIGATION

### 3.1 Materials

#### 3.1.1 Aluminum Container

An aluminium container (~46\*21\*21 cm) with a small outlet allowing the passage of air/jet fuel (as a vent) was used to immerse the geomembrane coupons in jet fuel.

### 3.1.2 Untreated Geomembrane

The untreated HDPE geomembrane was manufactured by GSE Linings Inc., Houston, Texas, USA. This 1.5 mm thick (60 mils) smooth black-surfaced HDPE geomembrane is produced from specially formulated virgin polyethylene resin. The average density of the geomembrane is 0.94 g/cm<sup>3</sup>. As per manufacturer's information it contains approximately 97.5% polyethylene and remaining 2.5% consists of carbon black, trace amounts of antioxidants and heat stabilizers. The standard OIT value for the geomembrane is 135min. This OIT value is greater than the minimum value of 100 min. recommended by the Ontario Regulation 232/98 - Environmental Protection Act, 1998. The relevant properties of the untreated geomembrane are summarized in Table 1.

Table 1: Properties of the geomembranes examined

Property	Method (ASTM)	Untreated		Fluorinated	
		Avg.	COV	Avg.	COV
Thickness (mm)	As received	1.5	--	1.5	--
OIT (min)	D3895	135	1.5	137	0.7
Crystallinity (%)	E794	52	2.4	51	2.2
Tensile-strength at yield (kN/m)	D6693	24	3.7	23	2.4
Tensile-strength at break (kN/m)	D6693	51	10.5	54	2.5
Tensile-strain at yield (%)	D6693	20	1.9	20	2.8
Tensile-strain at break (%)	D6693	869	11.9	979	2.8

Avg.=average; COV= coefficient of variation

### 3.1.3 Fluorinated Geomembrane

The fluorinated HDPE geomembrane was produced by GSE Linings Inc., Houston, Texas, USA and treated at Fluoroseal Inc., Texas, USA. This geomembrane was manufactured from the same 1.5mm thick (60 mils) smooth black-surfaced untreated HDPE geomembrane but had been treated by fluorination process. In this process elemental fluorine gas is applied to both sides of the untreated geomembrane. The fluorine atoms chemically substitute the hydrogen atoms in the carbon-hydrogen (C-H) polyethylene chain, creating carbon-fluorine (C-F) covalent bond on the outermost surface of the untreated geomembrane. This creates a thin carbon-fluorine layer (0.31-0.37 microns measured by Scanning Electron Microscope/Energy Dispersive X-Ray) on the two sides. The relevant properties of the fluorinated geomembrane are summarized in Table 1.

### 3.1.4 Jet Fuel A-1

Jet fuel (grade Jet A-1) is commonly a colourless to pale yellow liquid with kerosene like odour. According to the ASTM D1655 (ASTM 1998b) the specific gravity of Jet A-1 at 15°C can be between 0.775 to 0.840, freezing point is

-47°C and viscosity at -20°C is 8 mm<sup>2</sup>/s. Jet A-1 is almost insoluble in water with solubility of 5 mg/L (Cheminfo, 2001). Jet fuel is composed of a mixture of many different saturated and unsaturated hydrocarbons. It consists of blends of refined hydrocarbons derived from crude petroleum, naturally occurring gasoline, naturally occurring non-hydrocarbons and additives (ASTM 1998b).

## 3.2 Procedure

### 3.2.1 Installation, Experimental Setup and Exposure Condition

The jet fuel container was setup inside a fumehood (at laboratory temperature ~23°C. The coupons were placed upright and separated by 5 mm diameter glass rods so that no two coupons touched and both faces of each geomembrane were completely exposed to jet fuel. As some evaporative loss of jet fuel was observed the level was carefully maintained by topping up jet fuel at regular intervals. Jet fuel was completely replaced every few weeks (the rationale behind completely replacing the jet fuel is discussed in the results section).

### 3.2.2 Sampling Method

At each sampling period the coupons withdrawn from the bath were cut, rinsed, and wiped before all testing. Geomembrane mass monitoring tests were started immediately on a cut portion of both untreated and fluorinated geomembrane samples. All samples used for testing were taken at least 8mm from the edge of the coupons. This was done so that the tested specimens were free from any diffusion effect or depletion of antioxidants from the edge.

## 3.3 Analytical and Mechanical Testing Methods

### 3.3.1 Oxidation Induction Time Test

OIT is considered to be a good indicator of the amount of antioxidant present in the geomembrane. It is useful for comparing the relative oxidative resistance of geomembranes and extremely useful for monitoring the depletion of antioxidants in the geomembrane (Koerner *et.al.*, 1992; Hsuan and Koerner, 1995; Hsuan and Koerner, 1998; Sangam and Rowe, 2002; Müller and Jakob, 2003). Standard OIT tests were carried out (ASTM D3895, 1998c) using a differential scanning calorimeter (DSC).

### 3.3.2 Crystallinity Test

Crystallinity tests were performed according to ASTM E794 (ASTM, 1998d) also using a differential scanning calorimeter. The degree of crystallinity is one of the most important molecular properties of a polymer because it affects important physical and mechanical properties such as yield stress, modulus of elasticity, impact resistance, density, melting point and permeability (Kong and Hay, 2002; Sperling, 1992).

### 3.3.3 Diffusion, Sorption and Swelling

To assess the rate of diffusion/sorption into and out of the geomembrane, the density of each coupon was monitored. The mass per unit area was compared with the unexposed coupon, so that the percentage increase in mass of the coupon could be monitored with time. To measure diffusion out of the geomembrane coupons were removed after 75 days of exposure and cut into a 140x175mm rectangle. The coupons were then laid flat, out of direct sunlight, at 20°C. The fuel they contained was allowed to diffuse out into the atmosphere as the mass of the samples was periodically monitored.

### 3.3.4 Tensile Test

The tensile properties (load and elongation at yield and break) of geomembrane were obtained in accordance with ASTM D6693 (ASTM, 2003). Tensile tests were carried out using a universal testing machine (Instron Model 3396) consisting of 5KN load cell and self-aligning wedge grips appropriate to hold dumbbell shaped specimen (ASTM D638 for Type IV specimen (ASTM, 1998a); and ASTM D6693). The test was conducted at a speed of 50mm/min. Tensile properties at yield and break were evaluated using this data.

## 3.4 Results

### 3.4.1 Antioxidant Depletion

Figure 1 shows the change in OIT values with respect to time. At each sampling event at least 3 replicate samples were tested and the vertical bars in Figure 1 represent the standard deviation deduced from these tests. The OIT values decreased exponentially with immersion time. The

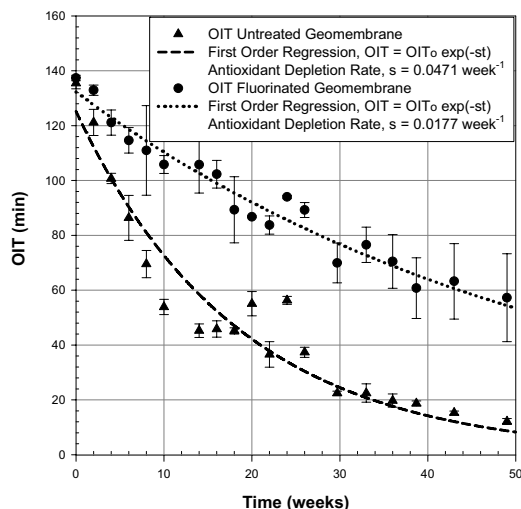


Figure 1: Variation of Standard OIT with immersion time for the untreated and fluorinated geomembranes antioxidant depletion process was much faster for the untreated geomembrane than the fluorinated

geomembrane. The antioxidant depletion rate for the jet fuel exposed samples was extremely fast compared to that observed for geomembrane samples exposed to municipal solid waste leachate at room temperature (Sangam and Rowe, 2002).

### 3.4.2 Antioxidant Depletion from Untreated Geomembrane

The initial OIT value of the untreated geomembrane was 135min. This reduced to 70min. after 8 weeks and 22min. after 30 weeks of immersion in jet fuel. The relationship between OIT and immersion time is exponential ( $R^2 = 0.98$ ) within the first 14 weeks of immersion. During weeks 16 to 26 some scatter is observed in the data and the OIT values did not decrease at the same rate as it was observed during the initial 14 weeks of immersion. It was hypothesised that the level of antioxidants had increased in the due to the migration of antioxidants from geomembrane into the jet fuel and that this slowed outward diffusion of antioxidants at later times due to the consequent decrease in concentration gradient. Having detected the trend and identified the potential concerns, on week 27 the Jet fuel in the bath was completely replaced. After this replacement, a more rapid decrease in OIT value was again noted providing some support for the hypothesis. The relationship between OIT and immersion time up to the testing period of 49 weeks is exponential having an  $R^2 = 0.94$  (Figure 1). The antioxidants depletion rate was calculated using regression of the  $\ln(\text{OIT})$  versus incubation time (Figure 2). At any given immersion period the OIT value representing the total antioxidant amount in the geomembrane can be expressed as:

$$\text{OIT} = \text{OIT}_0 \exp(-st) \quad [1]$$

or taking  $\ln$  both sides

$$\ln(\text{OIT}) = -st + \ln(\text{OIT}_0) \quad [2]$$

Where  $\text{OIT}_0$  = initial OIT (in minutes);  $s$  = antioxidant depletion rate (in weeks<sup>-1</sup>); and  $t$  = time (in weeks).

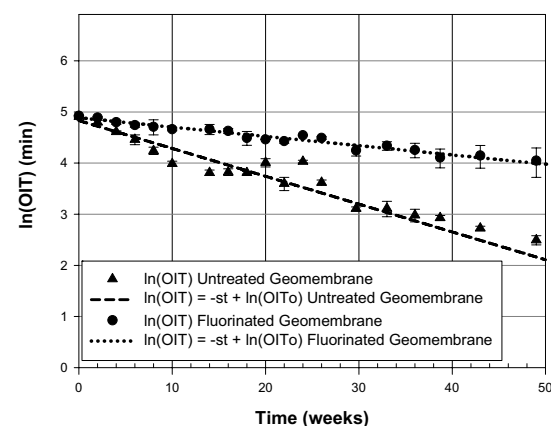


Figure 2: Plot of  $\ln(\text{OIT})$  with immersion time for the untreated and fluorinated geomembrane

The antioxidant depletion rate of Jet A-1 immersed untreated geomembrane was 0.0471 week<sup>-1</sup> or 0.2018 month<sup>-1</sup>. This is much faster (10.7 times) than the

depletion rate for untreated 2mm HDPE geomembrane in synthetic MSW leachate at 22°C which was measured by Sangam and Rowe (2002) i.e. 0.0188 month<sup>-1</sup>.

#### 3.4.3 Antioxidant Depletion from Fluorinated Geomembrane

The initial OIT value of the fluorinated geomembrane was 137min. and this reduced to 111min. after 8 weeks and 70min. after 30 weeks of immersion in jet fuel. The relationship between OIT and immersion time is exponential (with  $R^2 = 0.95$ ) as shown in Figure 1. The antioxidant depletion rate was calculated to be 0.0177 week<sup>-1</sup> or 0.0758 month<sup>-1</sup>. This is 2.6 times slower than the untreated geomembrane.

#### 3.4.4 Antioxidant Depletion Time at Laboratory Temperature

The antioxidant depletion time was calculated by estimating the time for the complete depletion of antioxidants from an initial value of 135min. (for untreated geomembrane) and 137min. (for fluorinated geomembrane) to a residual OIT = 0.5 minutes (unstabilized HDPE) using Equation 1. It was estimated that at laboratory temperature ~23°C the antioxidant depletion time for the untreated geomembrane was 119 weeks (2.3 years) and fluorinated geomembrane was 317 weeks (6.1 years). The difference between the antioxidant depletion time for the untreated and fluorinated geomembranes was 198 weeks (3.8 years). Thus the results indicated that the fluorination had beneficial effect and increases the antioxidant depletion time of the geomembrane exposed to jet fuel.

#### 3.4.5 Crystallinity

Crystallinity of the immersed geomembrane samples was examined at selected times. There was no significant change in crystallinity for either the untreated or fluorinated geomembrane immersed in jet fuel over the period examined.

#### 3.4.6 Effects on Diffusion, Sorption and Swelling

The decrease in mass with time resulting from diffusion of Jet A-1 out of the geomembrane after 75 days of immersion is shown Figure 3. Diffusion out of the untreated geomembrane proceeded more quickly initially than from the fluorinated geomembrane. The diffusion rates of both were large. After fourteen days of exposure to ambient air, both geomembranes had released over 70% of the fuel they had absorbed. At the start of the diffusion test, the mass of fuel in the fluorinated geomembrane was about 20% less than in the untreated geomembrane. This shows that diffusion into the fluorinated geomembrane was slower than diffusion into the untreated geomembrane.

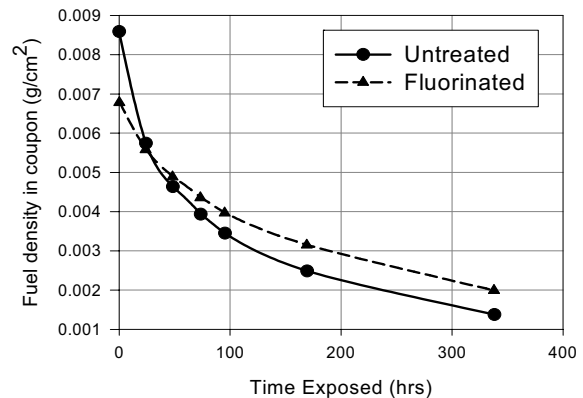


Figure 3: Density of retained fuel in HDPE coupon vs. hours exposed to air in the laboratory

#### 3.4.7 Effects on Tensile Properties

Two tensile stress-strain curves: one for a geomembrane that has not been exposed to jet fuel and one that has been exposed for ten weeks are presented in Figure 4.

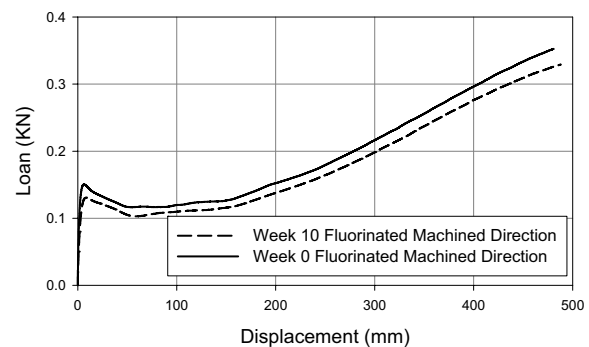


Figure 4: Load-displacement curves for fluorinated geomembrane, loaded in the machine direction

The two curves selected were for the specimens that had the median ultimate strain in their respective testing cohorts. It is noted that after 10 weeks immersion, less force is mobilized at a given displacement (strain). These two curves are fairly representative of the stress-strain curves of other specimens. The softening noted above raises the questions as to the effect the length of the period of exposure on yield stresses and strains. It was found that exposure to jet fuel reduces the yield stress, and increases the yield strain (see Figures 5 and 6). The effect of fluorination on the geomembrane is also of interest. Since fluorination is performed on geomembranes to reduce their permeability, one might expect that fluorinated geomembrane would take longer to be affected by external agents. However the effect does not appear to have been significant in this case and both the fluorinated and untreated geomembrane yield at a higher strain after exposure to Jet A-1 (the differences between the two being small).

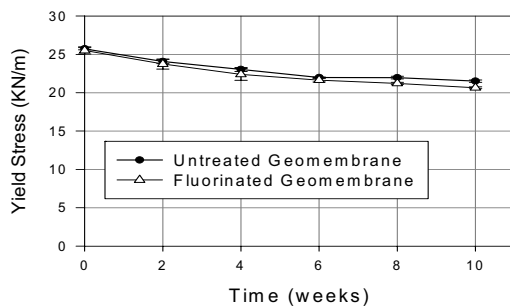


Figure 5: Yield stress vs. time of exposure curves for geomembrane loaded in the cross-direction

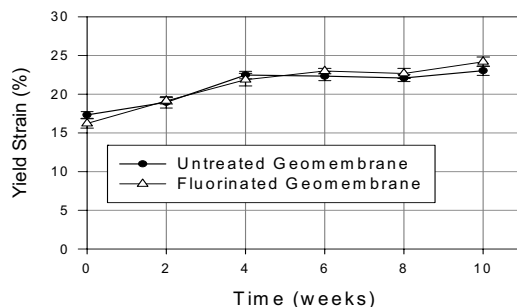


Figure 6: Yield strain vs. time of exposure curves for geomembrane loaded in the cross-direction

Similar results were obtained for the ultimate strength, but they were not as conclusive. One of the difficulties with analyzing ultimate behaviour is that not all specimens broke during testing (the test frame had a maximum extension of 550 mm).

#### 4. SUMMARY AND CONCLUSIONS

The effect of jet fuel on both 1.5mm thick untreated and fluorinated HDPE geomembrane has been examined. Geomembrane coupons were immersed in an aluminium container containing jet fuel A-1 at ~23°C. The change in oxidative induction time (OIT) (ASTM D3895), tensile properties (ASTM D6693) and geomembrane mass were monitored with time. The results indicated that immersion in jet fuel accelerates the antioxidant depletion rate relative to that observed in water or municipal solid waste (MSW) leachate by Sangam and Rowe (2002). It was found that fluorination of HDPE geomembrane provided significant beneficial effect and the antioxidants depleted at much higher rate for the untreated geomembrane than that for the fluorinated geomembrane. The depletion rate of antioxidants in untreated geomembrane is 2.6 times higher than the fluorinated geomembrane. The total antioxidant depletion time was estimated from these tests for both untreated and fluorinated geomembranes at laboratory temperature. The antioxidant depletion time for the fluorinated geomembrane is 198 weeks greater than for the untreated geomembrane at laboratory temperature. The results indicate that the fluorinated geomembrane performed better in jet fuel than the untreated geomembrane.

The results from crystallinity tests showed that there is no significant change in crystallinity in either the untreated or fluorinated geomembrane over the period examined. This provides indication that there are no observable changes in molecular arrangement of the polymer during the test period. The tensile tests results indicated that within the first ten weeks of immersion in jet fuel there is a statistically significant effect on the yield stress and strain of the HDPE geomembranes. The amount of stress required to cause yielding decreased, and of the yield strain increased corresponding to a general decrease in the axial stiffness of both geomembranes. Both samples lost about 20% of their strength during the ten weeks of exposure. Diffusion of fuel from the membrane into air was extremely rapid. Fluorination succeeded in roughly halving the rate of diffusion, but even the fluorinated geomembrane displayed a diffusive half-life of under a week.

Although these tests were performed at laboratory temperature and are subject to the number of limitations, the results provide an indication of the beneficial effect of fluorination of HDPE geomembrane used at the BAF-3 site. In particular the service life with respect to oxidation appears to be substantially longer for the fluorinated geomembrane with the time to full depletion of antioxidants at room temperature predicted to be 6.1 years compared to 2.3 years for the untreated geomembrane. The service life is expected to be even longer at the lower temperatures relevant to the arctic. This is an initial investigation and there is a need for additional testing to confirm these results.

#### 5. ACKNOWLEDGEMENTS

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