Rapid Screening Test for Assessing Durability of Geomembranes in Mining Liquors

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ABSTRACT
High Density Polyethylene (HDPE) geomembranes contain proprietary blends of antioxidants and stabilisers (known as the additive package) to improve the service life of the polymer. The service lifetime of HDPE liners is mainly dependent on the level and permanence of the additive package. The problem is manufacturers do not disclose the mix of additives to the end-user, and they regularly engage in resin substitution, masterbatch substitution and additive substitution based on price and local availability of the raw materials. This has led to a lot of uncertainty around the consistency and quality of HDPE geomembrane products available today. The way to overcome these issues is by reverse engineering the additive package through deformulation, and performing the newly developed rapid screening procedures outlined in this paper. Although conventional long-term immersion testing is now recognized as the preferred method to assess the durability of HDPE geomembranes in specific end-use media, the protracted amount of time required to develop meaningful ageing of the geomembrane is a disadvantage. Conventional accelerated ageing tests typically require a minimum of 3 to 6 months and sometimes years to complete, while the new rapid screening procedures can be completed within 2 to 4 weeks. The additive de-formulation combined with the knowledge of the acid and alkali tolerance of each additive (known as pK_a values) allows one to predict the performance of the geomembrane for a given service environment, while the rapid screening procedure can ameliorate the risk level associated with using HDPE geomembranes and provide a greater degree of comfort to designers and facility owners, particularly when project timelines are tight.

1. INTRODUCTION

An important question for a designer or owner of a structure which uses HDPE geomembrane as part of a liner system is 'how long will my geomembrane last?'. The service life of HDPE geomembranes is mainly controlled by the slow loss of antioxidants and stabilisers. The disadvantage of conventional immersion is that it takes a minimum of 3 to 6 months (and sometimes years) to deplete the additives of high-performance geomembranes to values which are meaningful for the purpose of estimating the service life via the Arrhenius model. Conventional immersion testing or "pot" testing typically involves the immersion of geomembrane samples in a specific chemical environment, and incubation at different elevated temperatures (commonly 55, 75 and 95°C) to accelerate the depletion of additives. However, these tests use the full cross-sectional thickness of the geomembrane sheet, meaning the diffusion path from the geomembrane core to the surface is relatively large.

The diffusive flux of permeant thought a geomembrane is provided by Fick's first law of diffusion:

$$J = -D\frac{\partial c}{\partial x}$$

Where, J = flux or rate of transfer of diffusing substances through a unit area of the geomembrane (mol/m^{2}sec)
D = diffusion coefficient in the geomembrane (m^2/s)
C = diffusing substance in the geomembrane (mol/m^3)
X = the distance parallel to the direction of diffusion (m)

In response to the need to obtain faster results in commercially relevant timeframes a rapid thin-film depletion test has been developed. By first compression moulding the geomembrane samples to uniformly thin-films (nominally 250 - 300 µm) the diffusion path for the additives is minimized thus increasing the rate of additive loss. The advantages of thin film testing compared to conventional immersion testing is the ability to rapidly undertake assessment of the geomembrane's suitability or resistance to a particular chemical environment without having to conduct long-term immersion studies. The availability of results from the laboratory testing can be considerably reduced to as little as 2 to 4 weeks, without increasing the risk level associated with identifying a geomembrane which is suitable for a specific service environment.

2. GEOMEMBRANE ADDITIVE PACKAGES

HDPE geomembranes contain proprietary blends of antioxidants and stabilisers to retard oxidative degradation of the polymer. Different HDPE geomembrane manufacturers use different blends, ratios and levels of antioxidants and stabilisers. The antioxidants are based on different hindered phenolic antioxidants and thioesters while the stabilisers are based on hindered amines. These additives are depleted by 5 main mechanisms namely: leaching (by liquids), migration (to interface), consumption (by free radicals), deactivation (by chemicals) and neutralization (by acid/base interactions). The problem is the HDPE geomembrane manufacturers do not disclose the 'mix' of proprietary blends of antioxidants and stabilisers they add to their products. An additional problem is that for each category of additive there exists 4 to 5 options each varying in functionality, performance, acid-tolerance, alkali tolerance, extractability and cost. What one ends up with in terms of durability is a lottery. Manufacturers regularly engage in resin substitution, masterbatch substitution and additive substitution based on price and local availability of the raw materials. This has led to a lot of uncertainty around the consistency and quality of HDPE geomembrane products available today. The way to overcome these issues is by reverse engineering the stabilizer package and de-formulating the additives mixture. The additive deformulation combined with knowledge of the acid and alkali tolerance of each additive allows one to predict the performance/lifetime of that additive for a given service environment.

The type of carbon black also plays a role in the durability of the geomembranes with the smaller particle size (18-22 µm) being more effective UV screens but on the other hand also irreversibly binding/adsorbing the antioxidants and stabilisers onto their high surface area. The coarse grades of carbon black (40-60 µm) intended for the rubber industry are less effective UV screens but also adsorb and immobilize less antioxidants/stabilisers.

3. DETAILS OF GEOMEMBRANE SAMPLES

Four common commercial HDPE geomembranes were used in this study, and have been designated "S1 to S4". Three of the geomembranes are multicomponent geomembranes (MCG), which refers to those that are heterogeneous with respect the component layers such as white-on-black HDPE liners which have titanium dioxide white pigment in the top layer instead of carbon black or conductive liners that have a bottom layer of HDPE that is heavily-filled with carbon black. Since Standard-Oxidation Induction Time (Std-OIT) and High Pressure-Oxidation Induction (HP-OIT) measurements on MCGs give only an averaged OIT result it is good practice to run OIT testing on the individual layer to obtain a "complete picture" of the stabilization profile of the geomembrane.

Details of the geomembrane samples used in this study are shown in Table 1.

Table 1. Details of geomembrane samples

Parameter	S1	S2	S3	S4
Material Type	HDPE	HDPE	HDPE	HDPE
Nominal Thickness (mm)	1.5	1.5	2.0	2.0
MCG Layers	Black/Black	White/Black	Black/Black	White/Black
Conductivity of Layers	None	None	Bottom	None
Surface Finish	All double-sided smooth			

The Std-OIT and HP-OIT values of each layer of the MCGs were measured in accordance with ASTM D3895 and ASTM D5885 prior to compression moulding to thin-films. The results of this testing are summarized in Table 2 and Table 3.

Table 2. Results of Std-OIT for samples prior to thin-film compression moulding.

MCG Layer	S1	S2	S3	S4
Full Profile (min)	186	176	146 | 166[1]	234
White only (min)	-	1267	-	293
Black only (min)	-	191	187	226
Conductive only (min)	-	-	138	-

[1] Due to presence of two different layers (conductive and non-conductive) of S3, the sample showed multiple exothermic peaks during the oxidative induction time measurement. The higher amount of carbon black in the conductive layer adsorbs a large proportion of the antioxidant/stabilisers which can reduce the STD-OIT values of the conductive layer.

Table 3. Results of HP-OIT for samples prior to thin-film compression moulding.

MGC Layer	S1	S2	S3	S4
Full Profile (min)	1630	2537	284	2521
White only (min)	-	1420	-	>5500
Black only (min)	-	>3935	1297	937
Conductive only (min)	-	-	260	-

The initial HP-OIT values reflect the level of hindered amine stabilizer additives in the samples. Table 3 shows that the white surface geomembranes (S2 and S4) have the highest HP-OIT values which is consistent with the deformulation results (refer Section 4) which also showed the white surfaced samples to have the highest levels of hindered amine stabilisers.

From examining Table 3 it is apparent that the HP-OIT results for the conductive geomembrane sample (S3) are apparently cause for concern given HP-OIT values of only 260 and 284 mins for the conductive layer and entire conductive geomembrane, respectively.

It is well known that activated carbon is a good filter and sorbent material for absorbing impurities and odors. In the same way carbon black's very high surface area means it can adsorb and immobilize antioxidants and reduce their effectiveness (Scheirs 2008). The apparent very low HP-OIT for the conductive layer can be explained by this adsorption and immobilization of the hindered amine stabilisers on the carbon black and hence the stabilizer molecules are not able to have mobility to trap free radicals and give a normal HP-OIT response.

4.   REVERSE ENGINEERING ADDITIVE PACKAGE

The additive analysis of 4 common commercial HDPE geomembranes was undertaken using quantitative Nuclear Magnetic Resonance (NMR) and High-Performance Liquid Chromatography (HPLC). A flowchart which outline the process is presented in Figure 1. The results of the additive analyses are summarized in HPA = Hindered phenolic antioxidant, HPS = Hindered Phosphite Stabilizer, TEA = thioester antioxidant, HAS = Hindered amine stabilizer, GMB = Geomembrane

Table *4*, while the correlation of the mix of additives to the service performance as predicted by Thin-Film Accelerated Immersion (TFAI) testing is discussed in Section 5.

| Extraction and NMR/HPCL analysis | Reverse engineering of the additive package of the GMB using quantitative **NMR/HPLC** to determine all additives and their levels |

| Determine total active concentration of HPA, HPS, TEA and HAS | Best practice GMB should contain greater than **5000 ppm** total active antioxidant and stabilizer concentration |

| Determine total active concentration of HAS | Best practice GMB should contain greater than **3000** ppm total active high molecular weight hindered amine stabilizer concentration |

| Determine total active concentration of HPA | Best practice GMB should contain greater than **2000 ppm** total active hindered phenolic antioxidant concentration |

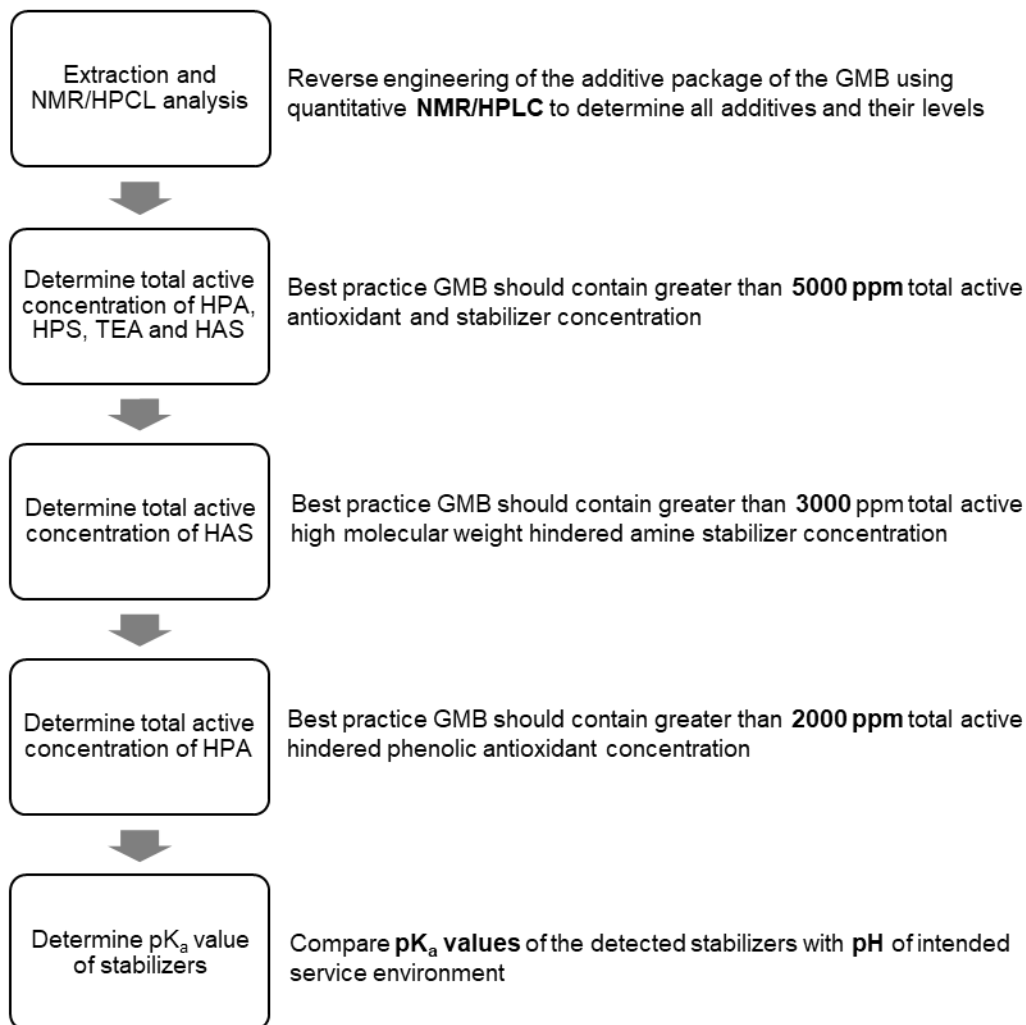| Determine pK$_a$ value of stabilizers | Compare **pK$_a$ values** of the detected stabilizers with **pH** of intended service environment |

Figure 1. Deformulation process of GMB to determine fitness for purpose for specific service environments.

HPA = Hindered phenolic antioxidant, HPS = Hindered Phosphite Stabilizer, TEA = thioester antioxidant, HAS = Hindered amine stabilizer, GMB = Geomembrane

Table 4. Additives present in analysed geomembrane samples.

Additives	Type	S1	S2	S3	S4
Chimassorb 944 (wt%)	Hindered amine stabilizer	0.183	0.427	0.130	0.272
Cyanox STDP (wt%)	Thioester antioxidant	0.063	0.044	0.041	0.028
Irganox MD 1024 (wt%)	Metal deactivator	-	0.116	-	0.089
Irganox 1010 (wt%)	Hindered phenolic antioxidant	0.132	0.114	0.194	0.189
Irganox 1330 (wt%)	Hindered phenolic antioxidant	0.081	0.072	0.081	Not detected
Irgafos 168-Phosphite (wt%)	Hindered phosphite stabilizer	0.252	0.231	0.343	0.263
Irgafos 168-Phosphate (wt%)	Not active	0.05	0.031	0.067	0.047
Irganox 1076 (wt%)	Hindered phenolic antioxidant	Not present in all samples			
total (wt%)		0.761	1.035	0.856	0.888
total (ppm)		7610	10350	8560	8880
Total active stabiliser package (ppm)		7110	10040	7890	8410
Active hindered amine stabilisers (ppm)		1830	4270	1300	2720

The above table shows that the white-surfaced geomembranes (S2 and S4) are more difficult to stabilize and hence require higher levels of stabilization (than all-black HDPE geomembranes) as evidenced by the higher active hindered amine stabilizer levels of 4270 ppm for S2, which is more than double the HAS loading of the all-black GMB (S1). The titanium dioxide pigment in the white layer is also potentially photocatalytic and hence the white formulation also contains a metal deactivator (Irganox MD 1024) to suppress metal-catalyzed oxidation of the polyethylene. Furthermore, since the white surface layer does not contain carbon black which is a highly effective UV screen, the titanium dioxide particles allows light penetration into the outermost surface to a greater degree than with carbon black filled liners.

5.   THIN-FILM ACCELERATED IMMERSION TESTING (TFAI)

In the TFAI testing the geomembrane samples are first compression moulded at 220°C to uniformly thin films (nominally 250 - 300 μm) with controlled cooling to provide a high and consistent surface area to volume ratio. Different layers of the MCG samples were separated into their constituent layers before thin-film preparation.

The thin-films were immersed in concentrated caustic soda solution (50% sodium hydroxide solution) with continuous liquid circulation at 85°C to increase the rate of additive leaching. The continuous fluid flow (~0.1 m/s) prevents formation of stagnant boundary layers that otherwise reduce additive migration. The thin film increases the surface area to volume ratio so that the diffusion path for the additives from the geomembrane core to the film surface is minimized thus increasing the rate of additive loss by Fickian diffusion. It also exposes the polymer to a higher surface area of liquor contact so acid-catalyzed reactions are accelerated hence increasing additive consumption and additive neutralization/deactivation. Furthermore, the continuous sweeping flow of liquor over the polymer sample increases the concentration gradient creating an enhanced driving force for additive migration, additive leaching and additive depletion. The above accelerating factors allow geomembrane liner samples to be assessed for long-term durability in as little as 2 to 4 weeks as compared with conventional immersion testing which typically takes a minimum of 3 to 6 months (typically longer) to create significant levels of additive depletion.

The use of thin-films can give a 10-fold acceleration of additive depletion rates compared with 2.0-2.5 mm liners based on Fick's Law. Furthermore, a continuous fluid flow is expected to give an approximately 2-fold increase in additive depletion rates. Therefore, the cumulative advantages of the thin-film test are about 20-fold acceleration compared with static immersion of full thickness samples at 85°C.

A flowchart which outlines the TFAI testing process is presented in Figure 2. The results of the thin-film Std-OIT and HP-OIT testing are summarized in Table 5 and Table 6, respectively.
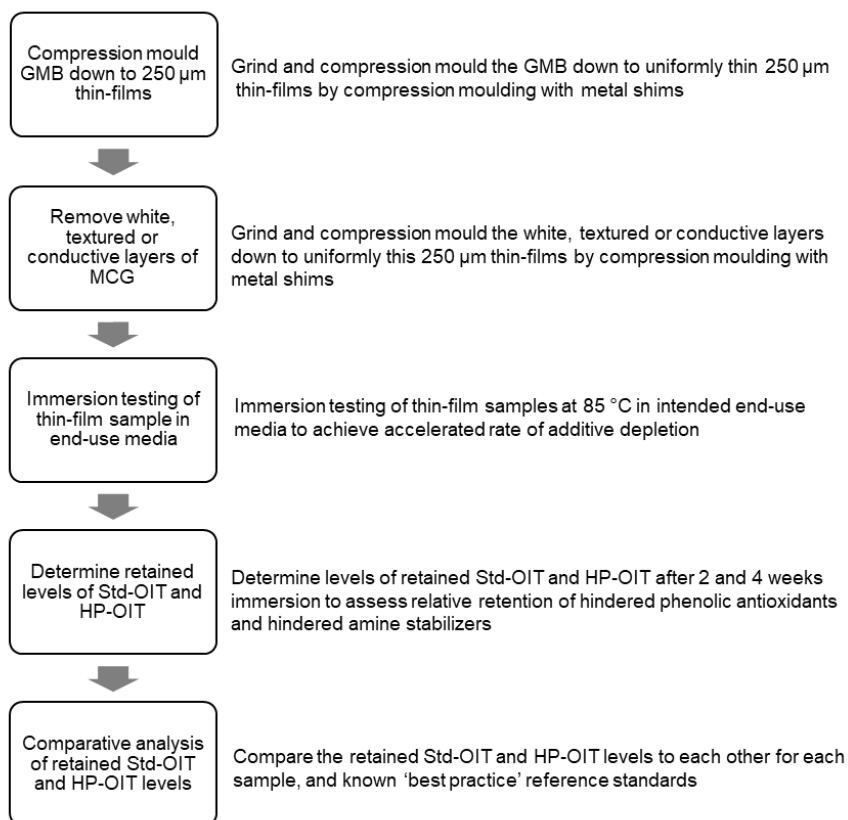
| Compression mould GMB down to 250 µm thin-films | Grind and compression mould the GMB down to uniformly thin 250 µm thin-films by compression moulding with metal shims |

| Remove white, textured or conductive layers of MCG | Grind and compression mould the white, textured or conductive layers down to uniformly this 250 µm thin-films by compression moulding with metal shims |

| Immersion testing of thin-film sample in end-use media | Immersion testing of thin-film samples at 85 °C in intended end-use media to achieve accelerated rate of additive depletion |

| Determine retained levels of Std-OIT and HP-OIT | Determine levels of retained Std-OIT and HP-OIT after 2 and 4 weeks immersion to assess relative retention of hindered phenolic antioxidants and hindered amine stabilizers |

| Comparative analysis of retained Std-OIT and HP-OIT levels | Compare the retained Std-OIT and HP-OIT levels to each other for each sample, and known 'best practice' reference standards |

Figure 2. TFAI testing to determine fitness for purpose for specific service environments.

Table 5. Results of St-OIT for TFAI testing.

Sample	MGC Layer	Thin Film Thickness (µm)	Room temperature	2 weeks at 85°C	4 weeks at 85°C
S1	Black only (min)	280	126	3.16	4.17
S2	White and Black (min)	300	124	4.34	2.39
	White only (min)	210	119	2.66	1.74
	Black only (min)	250	114	4.05	3.07
S3	Black and Conductive (min)	310	121	3.63	10.50
	Black only (min)	260	96	2.00	2.65
	Conductive only (min)	270	140	14.00	26.4
S4	White and Black (min)	320	128	2.66	2.46
	White only (min)	210	107	1.77	1.61
	Black only (min)	250	168	1.58	1.85

Table 5 shows all of the thin-film samples have substantially reduced STD-OIT after the 2-week accelerated testing. The large Std-OIT reduction is not unexpected in aggressive media since the phenolic antioxidants that contribute to the STD-OIT response are relatively small molecules of relatively low molecular weight that have high migration potential and therefore can be extracted and leached relatively quickly. Since the Std-OITs are severely depleted after 2 weeks, there are no significant differences observed between 2 weeks and 4 weeks' time period.

The STD-OIT results are dominated by the behavior of the lower-molecular weight hindered phenolic antioxidants which are lost relatively quickly by leaching and extraction in aqueous media, particularly at very high pH conditions where hydrolysis of ester bonds can occur (a particular issue with Irganox 1010).

Table 6. Results of HP-OIT for TFAI testing.

Sample	MGC Layer	Thin Film Thickness (µm)	Room temperature	2 weeks at 85°C	4 weeks at 85°C
S1	Black only (min)	280	958	557	487
S2	White and Black (min)	300	1080	906	100 | 1039[1]
	White only (min)	210	664	>670	>700
	Black only (min)	250	711	680	161 | >715[1]
S3	Black and Conductive (min)	310	237 | 481[1]	157 | 389[1]	95 | 407[1]
	Black only (min)	260	494	473	72 | 502[1]
	Conductive only (min)	270	470	342	156 | 368[1]
S4	White and Black (min)	320	865	716	158 | 405[1]
	White only (min)	210	821	560	103 | 544[1]
	Black only (min)	250	637	392	379

[1]The most likely reason for the observed multiple peaks in the HP-OIT curves is the occurrence of multiple exothermic oxidative transitions that arise from sample inhomogeneity. This has been reported previously by Scheirs (2000). The root cause of the multiple oxidative transitions is sample inhomogeneity that occurs when polyethylene is quickly oxidized on the surface where there is full contact with oxygen and there is negligible oxidation under the surface of the sample.

The retained HP-OIT results from Table 6 show much better retention of the hindered amine stabilisers after the accelerated leaching testing of 2 weeks and 4 weeks with all thin-films from each geomembrane showing 50 to 100 % retained HP-OIT. Greater than 80% retention of HP-OIT after 4 weeks of the accelerated thin-film immersion testing is considered very good in terms of long-term containment of concentrated caustic soda.

Plots of the % retained Std-OIT and HP-OIT versus time are presented in
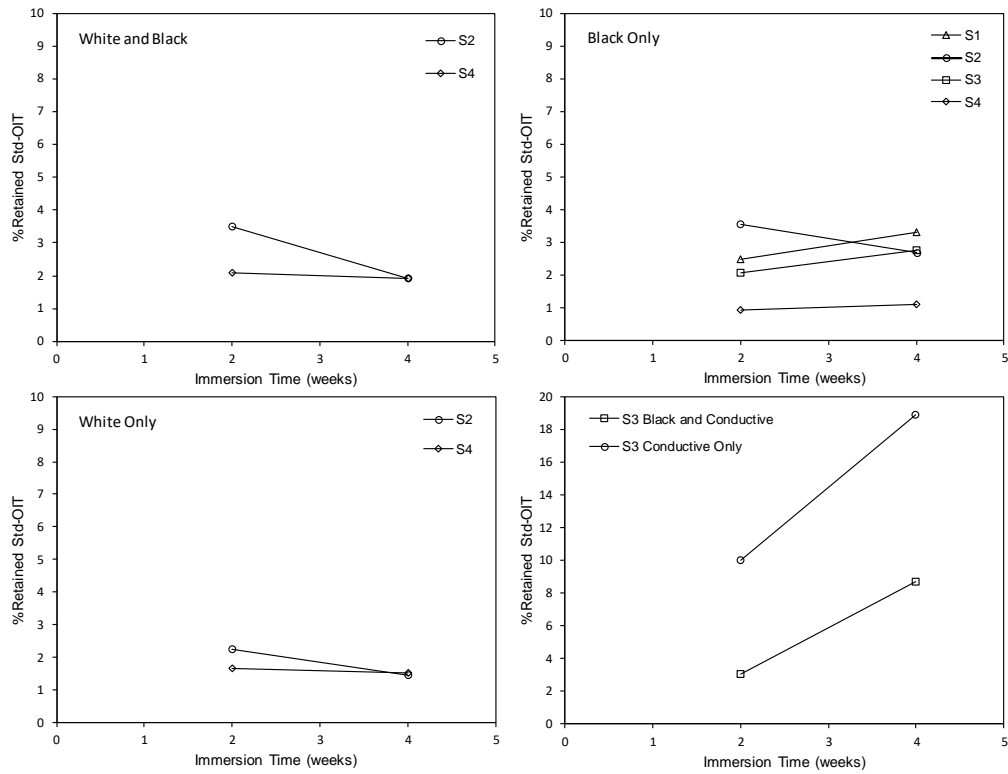Figure *3* and Figure *4*, respectively.

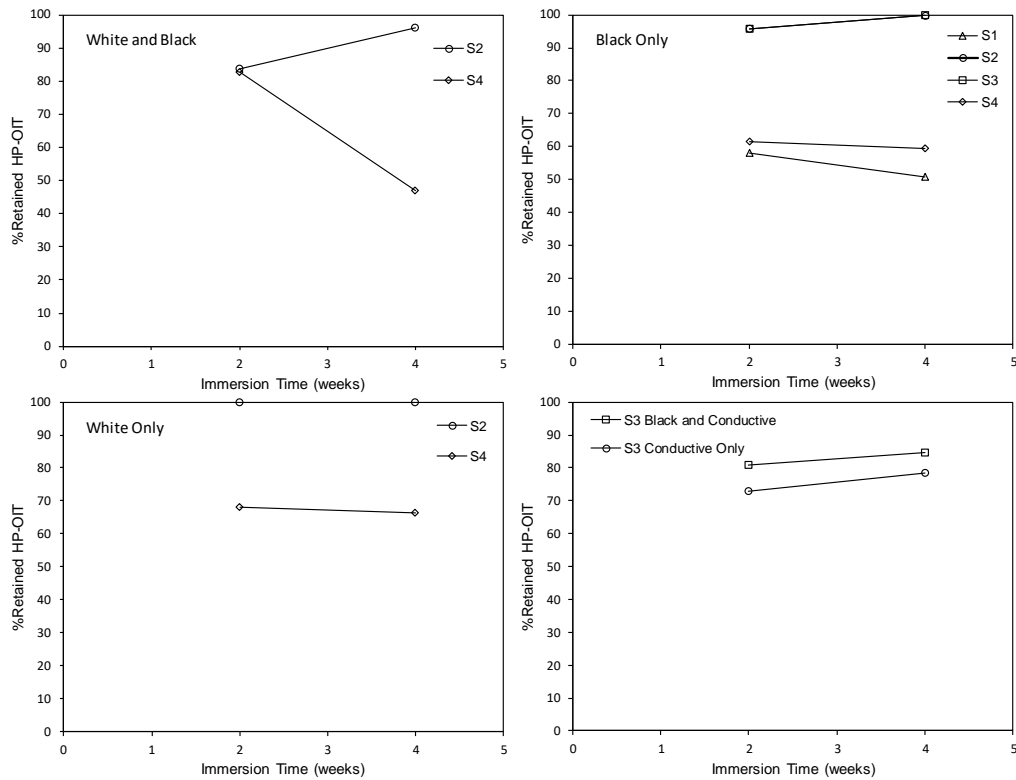Figure 3. Plots of % retained Std-OIT from TFAI testing.

Figure 4. Plots of % retained HP-OIT from TFAI testing.

6. DISCUSSION

The ageing resistance of HDPE geomembranes under extreme conditions is a function of the specific geomembrane resin and the composition and loading of their antioxidant/stabiliser package. The geomembrane samples used in this study were subjected to TFAI testing at 85°C in 50% sodium hydroxide solution to compare the resistance of their different antioxidant/stabilizer packages to extraction, leaching and depletion. Table 7 provides a summary of the deformulation analysis and TFAI testing, and ranks the samples in order of their performance, based on the total active stabilizer packages and the % retained HP-OIT.

Table 7. Summary of testing results and ranking of each geomembrane sample

Total Active Stabiliser Package	Retained HP-OIT at 2 weeks	Retained HP-OIT at 4 weeks
S2 (10040 ppm)	S2 (84%)	S2 (96%)
S4 (8410 ppm)	S4 (83%)	S3 (85%)
S3 (7890 ppm)	S3 (81%)	S1 (51%)
S1 (7110 ppm)	S1 (58%)	S4 (47%)

From Table 7 it can be seen that there a direct correlation between the total active stabilizer package and the % retained HP-OIT at 2 weeks immersion at 85°C. Sample S2 had the highest concentration of additives for both the total active stabilizer package and alkaline resistant hindered amine light stabilisers (Chimassorb 944), and also had the best % retained HP-OIT at 2 and 4 weeks immersion. At 4 weeks immersion, the correlation between the total active stabilizer package and the % retained HP-OIT becomes less clear due to hydrolysis of the hindered phenolic antioxidants which act as synergists for the hindered amine stabilizer response. Since Irganox-1330 is resistant to hydrolysis it has much better efficiency and synergy with the hindered amine stabilisers. However, since sample S4 lacks Irganox-1330 and only has Irganox-1010 as the hindered phenolic antioxidant, its synergy potential with the hindered amine stabilizer is reduced (Parrondo, 2004).

It is known that the phenolic antioxidants contribute exclusively to the Std-OIT results. Since they are relatively small molecules with molecular weights in the range from 600-1200 Dalton, they are relatively easily extracted from the polymer which explains the rapid decreases observed in the Std-OIT values with incubation time.

The hindered amine stabilisers which contribute significantly to the HP-OIT values on the other hand are much larger, more bulky molecules which remain entrapped in the polymer matrix for much longer and depending on their particular structure are relatively resistant to migration, leaching and extraction. The molecular weight of the most common hindered amine stabilisers used in modern geomembranes are in the range of 2200-4000 Dalton. Hence the HP-OIT curves asymptote at higher residual levels than those for the Std-OIT curves.

The Std-OIT values reflect the shorter-term thermal stability of the polymer and the Std-OIT result is dominated by the behavior of the lower-molecular weight antioxidants which are lost relatively quickly by leaching and extraction in aqueous media particularly at very high pH conditions where hydrolysis of ester bonds can occur (a particular issue with Irganox 1010 due to the ester linkages in the structure of the molecule of this antioxidant). For Irganox 1010, hydrolysis is the main degradation mechanism, leading to products with an increased solubility in water. Therefore, this stabiliser is less suitable for materials intended for water applications. Thus, in applications with aqueous alkali contact the stabilizing efficiency of Irganox 1010 is worse than for Irganox 1330 (Beissmann, 2013).

The Irganox 1010 molecules which contains ester groups are hydrolysed and that the new species migrate out into the surrounding aqueous solution. Hence, in applications with water contact the stabilizing efficiency of Irganox 1010 is worse than for Irganox 1330 (Thörnblom, 2011).

7. CONCLUSIONS

The use of thin-films and turbulent flowing liquor provides a high diffusion gradient for leaching and blooming of additives from the interior of the geomembrane to its surface, and thus accelerating the extraction of antioxidants and stabilisers from the geomembrane. The flowing (turbulent) liquor at elevated test temperatures prevent the formation of stagnant boundary layers and creates a high diffusion-gradient differential to extract antioxidants and stabilisers to the surface of the geomembrane.

The hindered amine stabilisers are large and bulky molecules that have much lower migration ability and are far more difficult to extract. Instead the hindered amine stabilisers are more affected by consumption (by free radicals), deactivation (by chemicals) and neutralization (by acid/base interactions).

This paper has shown the additive analysis of four common commercial HDPE geomembranes and has shown there is a positive correlation between the mix of additives present and the service life as predicted by TFAI testing. Based on the thin-film testing conducted in this study the preferred geomembrane for maximum durability in caustic service is the white/black geomembrane (S2) with 84-96% retention of stabilization based on retained HP-OIT values. The combination of the deformulation analysis and accelerated thin-film testing used in this study shows that the white/black geomembrane (S2) is the preferred liner of choice for caustic applications. The conductive HDPE geomembrane (S3) is next in performance and closely follows S2 with up to 81-85% retention of stabilization based on retained HP-OIT values. These conclusions are able to be drawn with confidence based on only 4 weeks of immersion testing, which is advantageous over the protracted time required to undertake convention immersion tests, particularly when project timelines are tight. The advantages of TFAI testing includes the following:

1. Enables an assessment of the ability of the liner to resist a specific service environment without having to conduct long-term immersion studies
2. Gives a prediction of the suitability of the liner for a particular chemical environment
3. Allows the time for 3 to 6 months of conventional immersion testing to be considerably reduced to 2 to 4 weeks without increasing risk
4. Thin-film testing in combination with deformulation analysis provides an in-depth understanding of the chemical compatibility of the additive package with the intended service environment.

Based on experience with HDPE liners in long-term caustic immersion the white/black geomembrane (S2) and the conductive HDPE geomembrane (S3) will exceed a service life of 25 years in the caustic containment applications if manufactured and installed correctly. It has been assumed that the distribution of various stabilizing additives will be uniform throughout the liner samples as manufactured.

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