

# Degradation of 2.4 mm-HDPE geomembrane with high residual HP-OIT

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**ABSTRACT:** The degradation of a 2.4 mm-HDPE geomembrane at 70 and 85°C immersed in synthetic leachate was investigated for ~5 years. This geomembrane met the GRI-GM13 (2011) specifications such that it had an initial high pressure (HP) oxidative induction time (OIT) > 400 min and more than 80% of its initial HP-OIT was retained in the GMB after ageing for more than three months in air exposure at 85°C. The tensile strains and strengths at break decreased at 85°C to less than 50% of their initial values after 30 to 45 months of ageing despite having a HP-OIT (670 min) more than 400 min specified by GRI-GM13 for a new GMB. This finding implies that an HP-OIT > 400 min does not necessarily mean that the geomembrane is protected from degradation when immersed in synthetic leachate at 85°C. At 70°C, the HP-OIT depleted to a residual value (760 min) higher than that reached at 85°C (i.e., 670 min); however, the tensile properties have not yet significantly degraded yet. The tensile strengths and strains at break in cross-machine direction at 85°C degraded faster than in the machine direction, nevertheless, at 70 and 85°C there were an initial decrease in the tensile strengths and strains at break measured in cross-machine direction of about 20% before Stage III started.

*Keywords: Geosynthetic, high pressure oxidative induction time, strain, strength.*

## 1 INTRODUCTION

The geomembrane (GMB) resin used to produce HDPE GMBs normally comprises (by weight): 96-97% medium density polyethylene resin; 2-3% carbon-black; and around 0.5% antioxidants and stabilizers (Hsuan et al. 2008; Scheirs 2009). Any change in one of these components may result in a change in the GMB's long-term performance (Ewais et al. 2014). The carbon-black, antioxidants and stabilizers are used to enhance the long-term performance of the GMB by protecting the polyethylene from degradation due to photo-oxidation and thermal oxidation. The depletion of antioxidants/stabilizers is usually monitored in terms of the depletions of standard oxidative induction time (Std-OIT, ASTM D3895) and high pressure OIT (HP-OIT, ASTM D5885) (Pauquet et al. 1993; Thomas and Ancelet 1993; Thilén and Shishoo 2000; Garcia et al. 2004; Li and Hsuan 2004; Ewais et al. 2014).

The classical chemical degradation model for HDPE GMBs conceptually involves three distinct stages (Hsuan and Koerner 1998, Figure 1a): Stage I: antioxidant depletion; Stage II: an induction period; and, Stage III: starts when there is a measurable change in the engineering properties of the GMB (e.g., tensile strength; melt index, stress crack resistance) due to degradation of the polymer until the GMB no longer meets the design requirements (Rowe 2004). Ewais et al. (2014) proposed a modification for the model as some GMBs may have complex formulation of antioxidants/stabilizers that leads to a difference in the depletion rates for Std-OIT and HP-OIT, nevertheless, GMBs may chemically degraded while having a significant HP-OIT values (Figure 1b). Thus, there may be two values for Stages I and II: one based on Std-OIT depletion and the other based on HP-OIT depletion.

In laboratory accelerated ageing tests, the end of Stage III is defined in terms of nominal failure which may correspond to the time at which a property of interest has decreased to 50% of either (a) its initial value (Hsuan and Koerner 1998; Figure 1), or (b) the value specified by GRI-GM13 (Rowe et al. 2009).

The times to nominal failure (i.e., time to the end of Stage III) in accelerated laboratory tests depends on the GMB, exposure conditions and temperature, and may range from months (at higher temperatures, e.g., 85°C) to hundreds of years at lower temperatures (e.g., 30°C) (Müller and Jacob 2003; Rowe et al. 2009; Rowe et al. 2009; Ewais and Rowe 2014b; Rowe et al. 2014). Similarly, for exposed GMBs, some GMBs were found to have reached the end of their service-life after 12 to 15 years (Peggs et al. 2010); others were found to have a good performance even after 30 years of exposures (Tarnowski and Baldauf 2006; Ramsey et al. 2013). The difference in exposure conditions may contribute to the difference of the GMB's performance discussed above; however, differences in performance is also greatly dependent on the differences in the GMB resins and/or different processing conditions (Hsuan et al. 1993a,b; Peggs et al. 2010; Ewais et al. 2014; Ewais and Rowe 2014a). Thus, to ensure longevity of HDPE GMBs, manufactures aim to produce HDPE GMBs having initial index properties (e.g., Std-OIT, HP-OIT, stress crack resistance) that meet the industrial specification GRI-GM13 (2011). Nevertheless, consultants may specify more stringent specifications for certain projects (Peggs et al. 2010).

The objective of this paper to investigate the ageing of a 2.4 mm HDPE GMB with high initial HP-OIT to provide greater insight regarding the degradation stages of the GMBs and to examine the effectiveness of inferring long-term performance from high initial HP-OIT values.

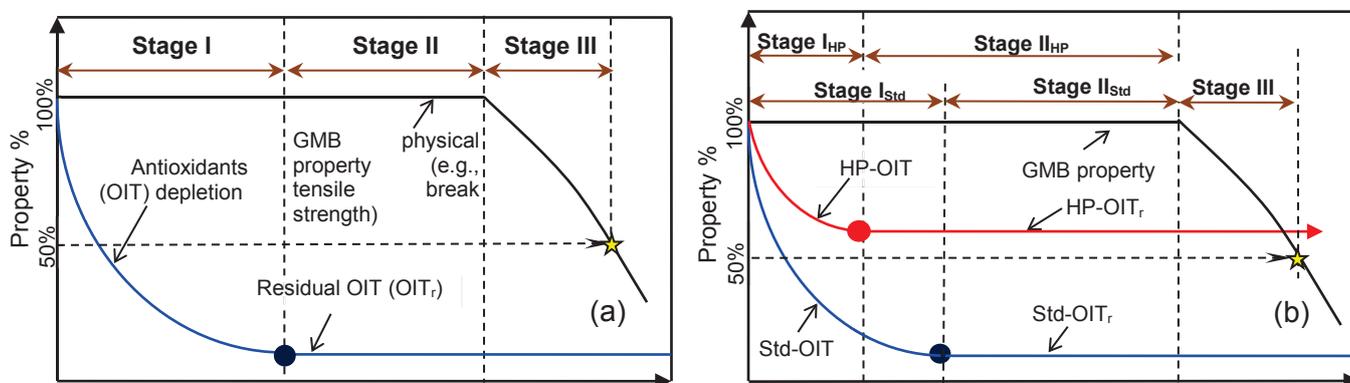


Figure 1. Classical three stage degradation model for GMBs (a) without HALS (Hsuan and Koerner 1998), (b) with HALS, such that Stage I<sub>Std</sub> may be greater or less than Stage I<sub>HP</sub> depending on the incubation conditions and temperatures (modified from Ewais et al. 2014).

## 2 MATERIALS AND EXPERIMENTS

A commercially available HDPE GMB (with initial properties shown in Table 1) was immersed in synthetic leachate at 85 and 70°C to investigate the changes in the antioxidants/stabilizers (OIT) and tensile properties of the GMB. Coupons (100×190 mm) were cut from the GMBs and were placed in four liter jars filled with synthetic leachate. The coupons were separated by 5mm diameter glass rods to ensure full contact with the leachate. The leachate used in this study is similar to that used in (Rowe et al. 2010) and was produced by mixing trace metals, and surfactant in water. A reducing agent was added to obtain  $E_h \approx -120$  mV. Specimens from the GMBs were retrieved at specified times and the changes in Std-OIT, HP-OIT, and tensile properties (strengths and strains) with ageing were monitored in accordance with ASTM D3895, ASTM D5885 and ASTM D638, respectively.

The measured initial properties (Table 1) of the tested GMB meet the requirements of the GRI-GM13 (2011) specification for the 2.5 mm GMB (when applicable). One requirement, inter alia, specified by GRI-GM13 (2011) is to meet one of two criteria: either (i) Std-OIT  $\geq 100$  min with 55% of the initial Std-OIT to be retained after 90 days of ageing in air at 85°C, or (ii) HP-OIT  $\geq 400$  min with 80% of the initial HP-OIT to be retained after 90 days of ageing in air at 85°C. Although the initial Std-OIT (175 min) was greater than 100 min, the GMB did not retain more than 55% of its initial value after 90 days of oven ageing in air at 85°C (Figure 2). Thus, the GMB did not meet criterion (i). However, the initial HP-OIT (960 min) was greater than 400 min and the GMB retained HP-OIT  $> 80\%$  of its initial value after 90 days of ageing in air at 85°C (Figure 2). Thus the GMB met criterion (ii) and therefore meets requirements of GRI-GM13.

Table 4.1. Initial Properties of the HDPE GMB examined.

Property	Method	Units	Value
Resin Density <sup>a</sup>	ASTM D1505	g/cm <sup>3</sup>	0.936
GMB Density <sup>b</sup>	ASTM D1505	g/cm <sup>3</sup>	0.946
Std-OIT	ASTM D3895	min	175 ± 3
HP-OIT <sup>c</sup>	ASTM D5885	min	960 ± 15
Degree of crystallinity (encapsulated)	ASTM E793	%	58.0 ± 1.4
LLMI (2.16 kg/190°C)	ASTM D1238	g/10min	0.115 ± 0.002
HLMI (21.6 kg/190°C)	ASTM D1238	g/10min	13.0 ± 0.3
FRR = (HLMI/MI)		ratio	112
Tensile properties for specimens type IV taken in machine direction. (ASTM D66693)			
Strength at yield		kN/m	45.0 ± 1.0
Strength at break		kN/m	78.7 ± 3.6
Elongation at yield		%	19.6 ± 0.5
Elongation at break		%	839 ± 38
Tensile properties for specimens type IV taken in cross-machine direction (ASTM D6693)			
Strength at yield		kN/m	46.0 ± 2.0
Strength at break		kN/m	78.6 ± 3.5
Elongation at yield		%	18.4 ± 0.5
Elongation at break		%	867 ± 39
Tensile properties for specimens type V taken in machine direction. (ASTM D638)			
Strength at yield		kN/m	45.8 ± 1.1
Strength at break		kN/m	83.34 ± 4.1
Elongation at yield		%	28.6 ± 0.9
Elongation at break		%	768 ± 44
Tensile properties for specimens type V taken in cross-machine direction (ASTM D638)			
Strength at yield		kN/m	46.9 ± 0.5
Strength at break		kN/m	86.7 ± 3.9
Elongation at yield		%	28.8 ± 0.6
Elongation at break		%	810 ± 39
SCR <sup>c</sup>	ASTM D5397	average (hrs)	1080 ± 38

<sup>a</sup> From the report provided by the manufacture.

<sup>b</sup> From the report provided by resin supplier.

<sup>c</sup> Mean ± standard deviation rounded to the nearest ten.

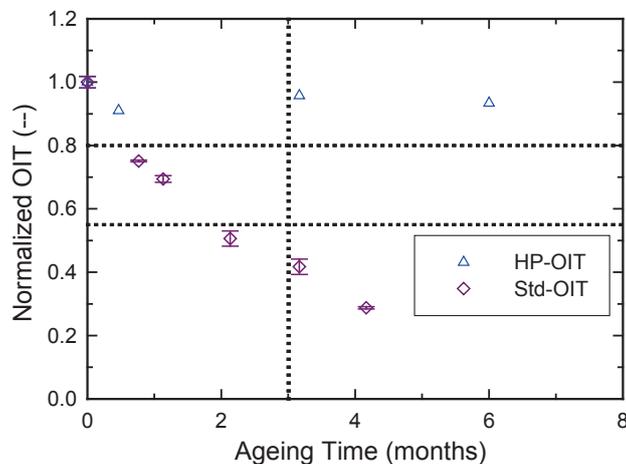


Figure 2. Variation in Std-OIT and HP-OIT (both normalized with respect to the initial values) with time at 85°C in air. Error bars represent ±1 standard deviation.

### 3 RESULTS

#### 3.1 Antioxidants Depletion

The Std-OIT depleted following first order decay model (Figure 3, Table 2) reaching a residual value of 3 min marking the end of Stage I<sub>Std</sub> after 3.3 and 10.1 months at 85 and 70°C, respectively. The HP-OIT also depleted following a first decay order model but reached a high residual value, HP-OIT<sub>r</sub>, of 670 and 760 at 85 and 70°C, respectively, marking the end of Stage I<sub>HP</sub> after around 13-17 and 15-23 months (Figure 3), respectively. The high residual HP-OIT<sub>r</sub> which is well in excess of 400 min required for a new GMB by GRI-GM13 as discussed above raises the question as to whether or not the high residual HP-OIT value would protect the GMB from degradation. The answer of this question will be explored in the following sections.

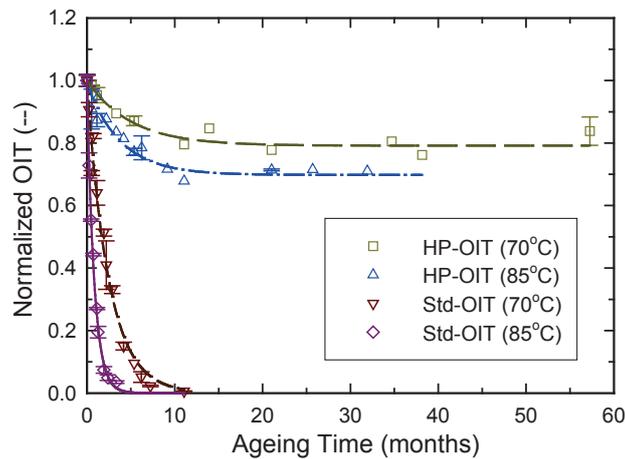


Figure 3. Variation in Std-OIT and HP-OIT (both normalized with respect to the initial values) with time at 85 and 70°C in synthetic leachate. Error bars represent  $\pm 1$  standard deviation.

#### 3.2 Tensile properties

The tensile strengths and strains at yield and break in both machine and cross-machine directions changed similarly with ageing (Figure 4 and Figure 5). The strengths and strains at yield increased up to 20% of the initial value and then remained stable at these values until the end of this study ( $\approx 58$  months of ageing). The increase in yield strength at 85°C was slightly higher than at 70°C (Figure 4a, Figure 5a); however, the increase in strains was similar (Figure 4b, Figure 5b). This may imply that the GMB samples aged in either 85 or 70°C will require the same deformation to reach the yield point; however, the GMB aged at 85°C may require greater force to reach yield point because of greater enhancement in the strength (perfection) of the crystal lamellae of the GMB at 85°C than that at 70°C (Ewais 2014).

The change in strengths and strains at break with ageing at 85°C measured in the machine direction followed the classical degradation model (Figure 4c,d). The strength and strains at break measured in the machine direction at 85°C remained un-changed for almost 25 to 28 months then degraded reaching the end of Stage III (nominal failure at 50% decrease as defined by Hsuan and Koerner 1998) after about 40 to 45 months (Figure 4c,d). The strength and strains at break measured in cross-machine direction at 85°C initially decreased by about 20% then remained un-changed for almost 18 to 20 months then degraded reaching the end of Stage III after about 30 to 36 months (Figure 5c,d).

At 70°C, there was a little to no change in tensile strength and strain observed in machine direction at break (until the end of this study  $\approx 58$  months) (Figure 4c,d), whereas, the tensile strength and strains at break measured in cross-machine direction (Figure 5c,d) initially decreased by about 20% (on average) with no further decrease in either the strains or strengths up to the end of this study (Figure 5c,d).

### 4 DISCUSSION

At both 70 and 85°C, the Std-OIT depleted differently to the HP-OIT (Figure 2 and Figure 3) implying that there are some antioxidants/stabilizers in the GMB that can be detected by HP-OIT but not detected by Std-OIT and vice versa. Although the Std-OIT depleted significantly reaching a low residual value, the HP-OIT continued to deplete after the depletion of the Std-OIT reaching a very high residual value of 670 and 760 min at 85 and 70°C, respectively, implying that there is a significant amount of antioxidants/stabilizers in the GMB even when residual was reached. However, the GMB started to

degrade at 85°C after HP-OIT depleted to the residual value with the degradation starting between 18 to 28 months (depending on the property monitored) and reaching the end of Stage III after around 30 to 45 months (depending on the property monitored) even though the GMB had greater than the minimum 400 min specified by GRI-GM13 for a new HDPE GMB. Thus, GMBs having a high HP-OIT (e.g., > 400 minutes) does not mean that a GMB will be protected against oxidative degradation at 85°C. Specifying a high HP-OIT for new GMB to ensure good long-term performance for GMBs may be misleading in that only a portion of this high value may be effective in protecting the GMB in leachate (less than 300 min or 30% on the initial HP-OIT was effective for the cases examined here at 85°C). This also implies that in long-term laboratory studies of the ageing of the GMBs, the physical properties of the GMB should be monitored during antioxidant/stabilizer depletion to confirm the effectiveness of antioxidants/stabilizers detected by HP-OIT in protecting the GMB from oxidative degradation; one cannot assume the presence of antioxidants/stabilizers (detected by HP-OIT) at a high values implies that the GMB is still in Stage I.

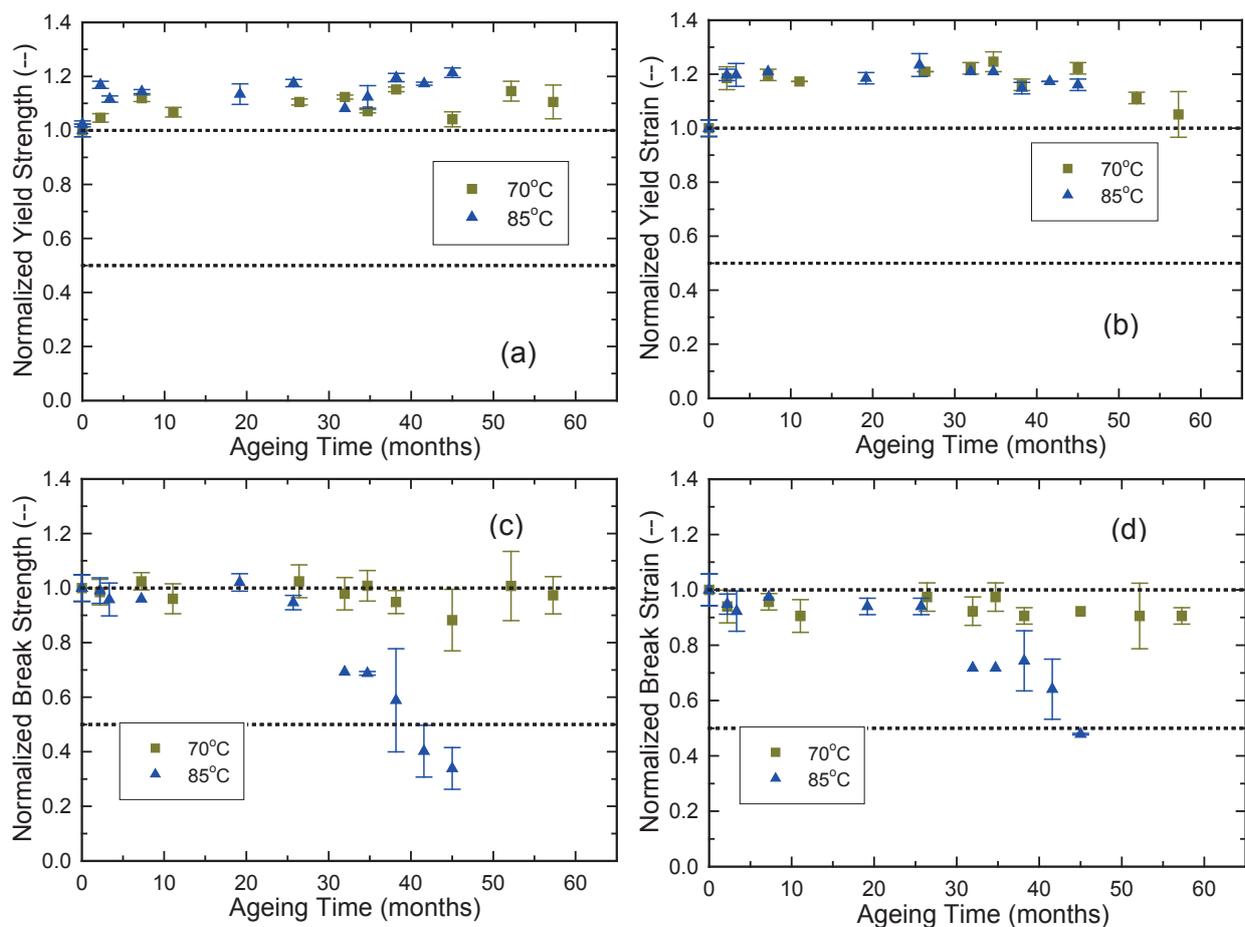


Figure 4. Variation in tensile properties (normalized with respect to their initial values) with time for specimens taken along the machine direction from the GMB samples aged at 85 and 70°C in leachate: (a) yield strength, (b) yield strain, (c) break strength, and (d) break strain. Error bars represent  $\pm 1$  standard deviation.

The degradation of the GMB while possessing high HP-OIT (implying significant amount of antioxidants/stabilizers present in the GMB) may be because these antioxidants/stabilizers underwent some antagonistic mechanisms in the GMB preventing them from protecting the GMB (Ewais et al. 2014)

Another possible explanation for the degradation of the GMB although it possess a high HP-OIT is that the GMB did not undergo oxidative degradation but, rather, it experienced thermal degradation in the absence of oxygen (Holström and Sörvik 1974; Kuroki et al. 1982), i.e., the antioxidants/stabilizers are protecting the GMB from oxidative degradation but they are not able to protect it from chain scission and/or chain cross-linking due to thermal ageing in absence of oxygen. This latter explanation is still under investigation.

The strengths and strains at break cross-machine direction initially decreased at both 85 and 70°C (Figure 6 and Figure 7) by 20%; whereas, no to little initial change was observed in the machine direction. The reason of this trend is still under investigation but it may be due to some change in the

morphological structure of the GMB that did not involve either chain scission nor cross-linking (e.g., chain disentanglement, Ewais and Rowe 2014b). Also, at 85°C, Stage III inferred from cross-machine direction strength and strains (Figure 6) started and ended earlier than that inferred from machine direction, implying that the properties in machine direction is less sensitive to ageing as compared to that in cross-machine direction.

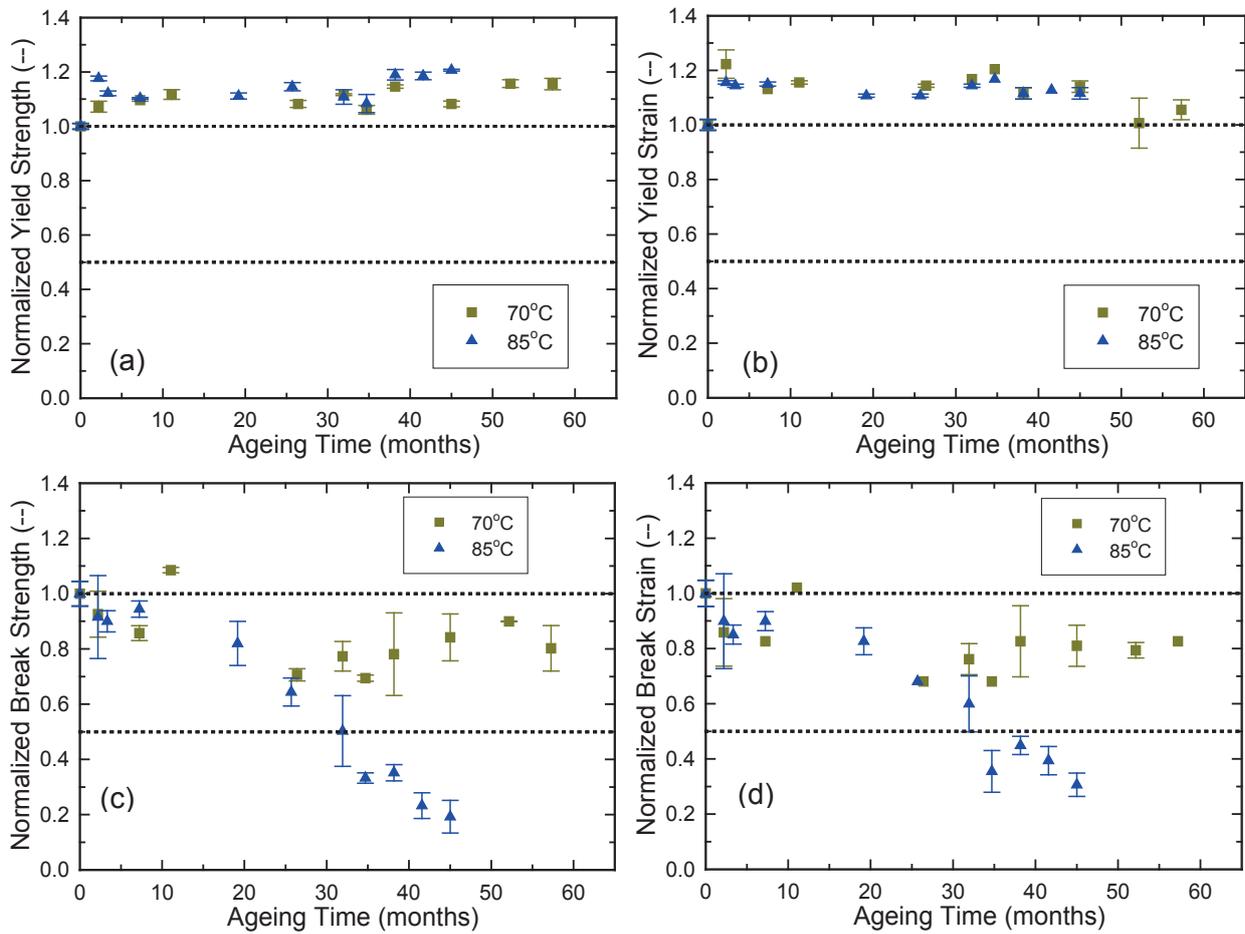


Figure 5. Variation in tensile properties (normalized with respect to their initial values) with time for specimens taken along the cross-machine direction from the GMB samples aged at 85 and 70°C in leachate: (a) yield strength, (b) yield strain, (c) break strength, and (d) break strain. Error bars represent  $\pm 1$  standard deviation.

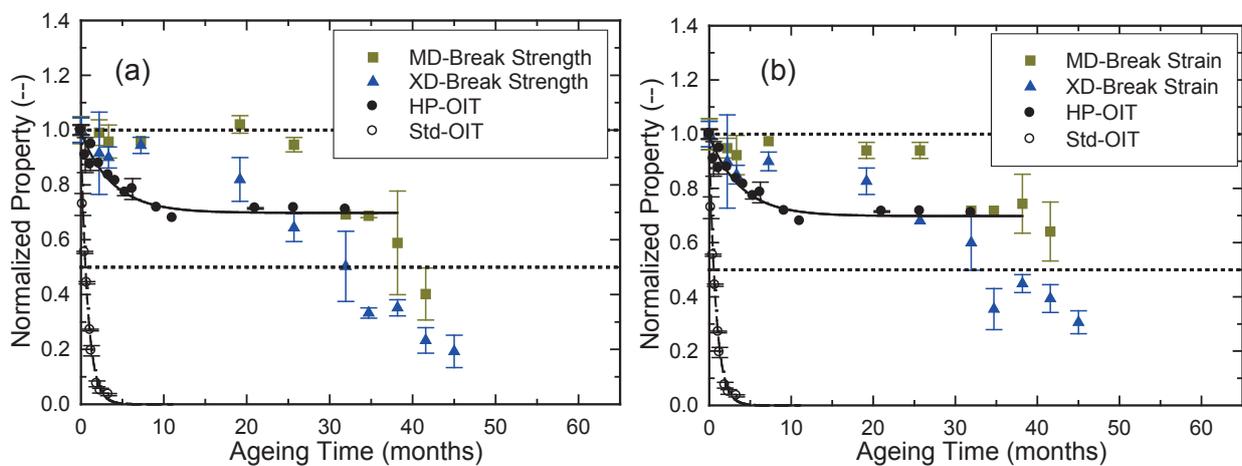


Figure 6. Comparison between: (a) the variation in tensile break strengths in machine and cross-machine directions and (b) the variation in tensile break strains in machine and cross-machine directions with time at 85°C in synthetic leachate. The depletion of Std-OIT and HP-OIT with time is also plotted with the changes in the tensile strains and strengths at 85°C. Error bars represent  $\pm 1$  standard deviation.

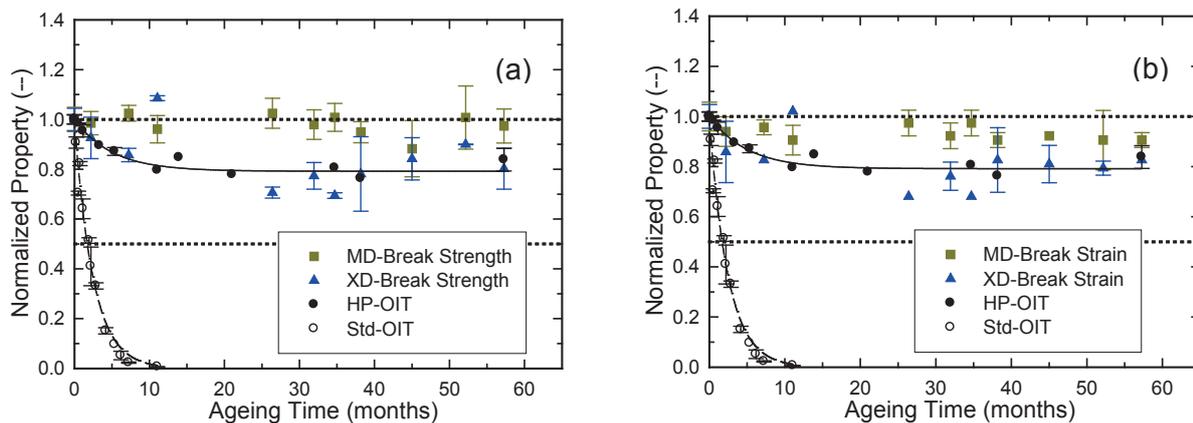


Figure 7. Comparison between: (a) the variation in tensile break strengths in machine and cross-machine directions and (b) the variation in tensile break strains in machine and cross-machine directions with time at 70°C in synthetic leachate. The depletion of Std-OIT and HP-OIT with time is also plotted with the changes in the tensile strains and strengths at 70°C. Error bars represent  $\pm 1$  standard deviation.

## 5 CONCLUSION

The current study shows that relying on HP-OIT to ensure the longevity of HDPE may be misleading as the investigated GMB has degraded at 85°C in leachate while still having more than 400 min HP-OIT (the minimum value specified by GRI-GM13 for a new GMB). This might be because the antioxidants in the GMB had undergone antagonistic mechanism in the GMB hindering their functionality. However, there is a possibility that these antioxidants/stabilizers detected by HP-OIT were effective in protecting the GMB from oxidative degradation but the GMB had undergone thermal degradation in the absence of oxygen. The latter hypothesis needs more investigation.

The study also shows that for this 2.4 mm-thick GMB, the properties in machine direction is less sensitive to ageing than in cross machine direction. Nevertheless, there was an initial decrease (about 20%) in the tensile strength/strains during Stages I and II before chemical degradation took place) in cross-machine direction. Thus, the no to little changes in the physical properties in Stages I and II proposed in the classical degradation model may be applicable to the tensile properties measured in machine direction but not in cross-machine direction.

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