HDPE geomembrane properties in mine reclamation covers after 13 and 20 years

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ABSTRACT: Geomembranes (GMs), especially high-density polyethylene (HDPE) ones, are used to provide a barrier to water and oxygen in mine site reclamation cover systems. The physical stability of these GM remains a major concern as it affects their performance to control fluid flows. Considering that HDPE GMs can crack even while being in the elastic domain, a maximum allowable strain (MAStrain) was fixed at 4% to avoid stress cracking. The MAStrain corresponds to a maximum allowable stress that must not be exceeded. This paper assessed the chemical and mechanical properties of exhumed GMs from two cover systems installed 13 and 20 years, and the impact of the tensile properties on the MAStrain. The results show that the GM remains in the first degradation stage and no negative impact was found on the mechanical properties. The tensile behavior of the exhumed GM indicates a gain of stiffness that reduces the MAStrain.

1 INTRODUCTION

Geomembranes (GMs) are widely used in the mining industry. They are used in the mining operations as a liner in heap leach pads or for water ponds, as in the mine waste management as a liner for tailings storage facilities or in the reclamation process as a fluid-tight component in cover systems (Lupo & Morrison 2007; Touze-Foltz & Lupo 2009). For this last example of use, the role of the GM is to prevent water and oxygen ingress into the sulphide tailings to limit the generation of acid mine drainage that could be harmful to the environment (Magsoud et al. 2021; Rarison et al. 2022). Almost all of the GMs used in covers are high-density polyethylene (HDPE) (Maqsoud et al. 2021). Nevertheless, HDPE GMs are prone to stress cracking because of their high crystallinity (Müller 2007; Scheirs 2009). Stress cracking is defined as "an external or internal crack in a plastic caused by tensile stresses less than its short-time mechanical strength" (ASTM D883 2005). Those cracks would then compromise the performance of the cover to control fluid flows. The physical stability of GMs then remains a major concern, especially in covers as the tailings can settle over time. Settlement can generate tensile stresses inside the GM during the service life of the cover. The maximum allowable strain was then introduced as a design criteria in order to ensure the long-term durability of the cover system (Dixon & von Maubeuge 1992; Eldesouky & Brachman 2018; Jones et al. 2000; Peggs et al., 2005; Rowe & Yu 2018; Rowe & Yu 2019; Rowe et al. 2020).

Nevertheless, little is known about the in situ long-term behaviour of GMs in service. One way to learn more about this behaviour is to exhume GMs installed in cover systems. GM exhumation is commonly used for in-service performance assessment (e.g., McWatters et al. 2020; Rowe et al. 2010). The main objective of this study is to assess the actual properties of GMs after years of service. To this purpose, GMs were exhumed from two sites. The exhumed

geomembranes were characterized in the laboratory to assess their antioxidant and stabilizer levels to know the degradation stage of the GMs, and their tensile properties. The aim is not to compare the properties of the two GMs. As the initial properties of these GMs are unknown, the obtained results would be compared to typical virgin HDPE GM properties. The results will be then used to assess the impact on the design criteria of maximum allowable strain.

2 GEOMEMBRANE EXHUMATION

To reach the study objective, two sites were selected, S1 and S2, where cover systems with smooth HDPE GMs were installed 13 and 20 years ago to reclaim these sites. S1 is located in the western part of Québec, Canada. S1 was reclaimed in 2005-2006 with a cover system composed by a 1.5 mm-thick smooth black HDPE GM installed directly above oxidized tailings and a 0.6 m-thick silt protection layer. S2 is located in the mid-north of Québec, Canada. This site was reclaimed in 1999-2000 with a cover system made up of 1.5 mm-thick smooth black HDPE GM installed directly above oxidized tailings and covered by a 1.4 m-thick till protection layer. The cross sections of the two cover systems are presented in Figure 1. On both sites, the surface of the protection layer was vegetated.



Figure 1. Cross sections of the cover at the sites 1 and 2.

To access the GMs, S1 was exhumed in August 2019 and S2 in August 2022. The first step was to form tiles of surface vegetation of the cover systems. The second step was to excavate the protection layer through to the GM level with a mechanical shovel, with particular care to the proximity of the GM so as not to damage it during excavation efforts. The third step was to sample the GM with dimensions of 2 m by 2 m and 1 m by 1 m for S1 and S2, respectively. The fourth step was to substitute the exhumed GM with new HDPE GM patch that was seamed by extrusion to the GM on the site to ensure the sealing of the cover. Finally, the excavated soil was returned and recovered with the surface vegetation tiles.

3 GEOMEMBRANE CHARACTERIZATION

Details on the performed physical, chemical and mechanical characterizations of the two HDPE exhumed GMs are presented below. The initial properties of these GMs are unknown as the different characterizations were not performed when the membranes were put in place. The results obtained with the exhumed GMs will be compared to those obtained from a typical virgin HDPE GM that was characterized in the laboratory and is assumed to be representative of the initial GM installed in the two sites. To assess the significance of the differences of the GM mechanical properties (typical virgin GM compared to S1 GM, and typical virgin GM compared to S2 GM), two-sample t-tests (Student 1908) were performed. Prior to two-sample t-tests, the normality of the distributions was verified with Kolmogorov-Smirnov tests (Lilliefors 1967). The significance was set at 0.05 (95% of confidence) for these two tests; which means that when the p-value obtained with the two-sample t-test is higher than the 0.05, the difference between the compared data is not significantly different; and when the p-value obtained with the normality test is higher than 0.05, the data are significantly drawn from a normal distribution.

3.1 Physical characterization

The physical characterization of the GMs (virgin, S1 and S2) consisted of measuring the GM thickness. The thickness measurement was performed according to ASTM D5199 (2019b) using MTG-DX2 thickness gauge which has the accuracy of $\pm 4 \mu m$ (Checkline, USA). The GM thickness T_{GM} is defined as the mean of 10 thickness measurements on 10 disks of 80 mm diameter.

3.2 Chemical characterization

To assess the antioxidant and stabilizer level, which would give information on the GM degradation state, low-pressure and high-pressure differential scanning calorimetry (DSC) were performed. The low-pressure DSC (140 kPa) was operated at high temperature (200°C) to measure the standard oxidative-induction time (Std-OIT) according to ASTM D3895 (2019). The high-pressure DSC (3.4 MPa) was conducted at lower temperature (150 °C) to measure the high-pressure oxidative-induction time (HP-OIT) according to ASTM D5885 (2017). The two tests give complementary information as antioxidant and stabilizer effective temperature ranges are different. For example, hindered phenols and phosphites whose effective temperature range is above 150° C can be detected with std-OIT while hindered amines and thiosynergists whose effective temperature range is up to 150° C can be detected with HP-OIT (Hsuan & Koerner 1998). The Std-OIT was performed in duplicate (for the virgin, S1 and S2 GMs). Only single measurements of the HP-OIT were performed for the S1 and S2 GMs. The HP-OIT of the virgin GM was given by the manufacturer.

3.3 Mechanical characterization

The mechanical properties of the GM from S1 and S2 were assessed with tensile tests performed on dog-bone shaped specimens (Figure 2.a) according to ASTM D6693 (2015). Raw data are presented in terms of force-displacement curves as illustrated in Figure 2.b. The force-displacement curve gives four parameters: the yield force (*YF*), the yield displacement (*YD*), the break force (*BF*), and the break displacement (*BD*). These parameters are used to define four tensile properties following equations (1) to (4): the tensile yield strength (*TYS*, N/mm) and the percent yield elongation (*PYE*; %); the tensile break strength (*TBS*; N/mm) and the percent break elongation (*PBE*; %).

$$TYS = \frac{YF}{W} \tag{1}$$

$$PYE = \frac{YD}{GL_Y} \cdot 100 \tag{2}$$

$$TBS = \frac{BF}{W}$$
(3)

$$PBE = \frac{BD}{GL_B} \cdot 100 \tag{4}$$



Figure 2. Dog-bone shaped specimen for tensile test (a) and typical raw result from a tensile test (b).

where W (specimen width), GL_Y (gauge length for yield) and GL_B (gauge length for break) are defined on Figure 2.a.

The dog-bone shaped specimens were prepared in the machine/roll direction (MD) and in transverse or crossmachine direction (CD) to assess the anisotropy. Five tensile tests were performed in each direction for each GM (virgin, S1 and S2).

4 RESULTS

4.1 Geomembrane thickness

The thicknesses of the three GMs assessed in this study are presented in the Figure 3. The T_{GM} of the virgin, S1, and S2 GMs are 1.51 mm, 1.55 mm, and 1.57 mm, respectively. The three values are above the nominal thickness of 1.50 mm and show the variability of the GM thickness.



Figure 3. Box plots of the thicknesses of virgin and exhumed geomembranes.

4.2 Antioxidant and stabilizer level

The results of the antioxidants and stabilizers level are presented in terms of Std-OIT and HP-OITs in Table 1 below for the virgin, and the S1 and S2 GMs who are 13 and 20 years old, respectively. The Std and the HP-OITs of the GM from the site S1 (211 min and 496 min, respectively) are close to those of the typical virgin GM (195 and >400 min, respectively), which means that the S1 GM degradation is in its earlier part. The Std and the HP-OITs of the GM from the site S2 (111 min and 314 min, respectively) are lower than those of the typical virgin GM. The lower OITs (Std and HP) of S2 GM would then indicate that this GM has an advanced degradation state.

The Std and HP-OITs of the S1 and S2 GMs are higher than the residual values (when the antioxidants and stabilizers are completely depleted) that can be as low as 1.5 min and

Table 1. Standard and High-Pressure OITs of a typical HDPE geomembrane and of the exhumed geomembranes.

Properties	Units	Virgin	S1	S2
Std-OIT	min	195	211	111
HP-OIT	min	>400*	496	314

*Provided by the geomembrane manufacturer.

80 min, respectively (Ewais et al. 2014). These results then show that there are still antioxidants and stabilizers inside the GMs from S1 and S2, which means that the GM remains in the antioxidant depletion stage according to Hsuan and Koerner (1998). During this stage, the GM's properties (e.g., tensile strength) are expected to remain unaffected.

4.3 Mechanical properties

The box plots of the yield and break properties of the virgin, and the exhumed HDPE GMs (S1 and S2) are presented in Figures 4 and 5, respectively. The results from the two principal directions (MD and CD) are also included. All the data are significantly drawn from a normal distribution as all the p-values obtained with the Kolmogorov-Smirnov tests are above the significance level of 0.05 (presented in Table 2). Two-sample t-tests could be then performed to assess the significance of the differences (e.g., MD vs. CD, virgin GM vs. S1 GM, virgin GM vs. S2 GM).



Figure 4. Box plots of tensile yield strength (a) and percent yield elongation (b) obtained with the virgin and the exhumed HDPE GMs.



Figure 5. Box plots of tensile break strength (a) and percent break elongation (b) obtained with the virgin and the exhumed HDPE GMs.

	Table 2.	P-values obtained	with the Kolmogorov-Smirnov	normality tests.
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GM	Direction	TYS	PYE	TBS	PBE
Virgin	MD	0.89	1.00	1.00	1.00
e	CD	1.00	0.51	0.63	0.84
S1	MD	1.00	1.00	1.00	1.00
	CD	1.00	0.64	0.90	0.82
S2	MD	1.00	0.50	1.00	0.53
	CD	1.00	0.35	1.00	1.00

Regarding the anisotropy of the tensile properties, it can be seen in Figures 4 and 5 that the results obtained in MD can be different from those obtained in CD. The two-sample t-

tests show that the anisotropy is verified for the yield properties of the virgin and S1, for the TBS of the S2 GM and for the PBE of the S1 GM as it can be seen in Table 3. The anisotropy would then be more observable for recent GMs.

GM	Compared direction	TYS	PYE	TBS	PBE
Virgin	MD and CD	0.00^{*}	$0.00^{*} \\ 0.00^{*} \\ 0.88$	0.93	0.13
S1	MD and CD	0.00^{*}		0.11	0.02 [*]
S2	MD and CD	0.27		0.04*	0.35

Table 3. P-values obtained with the two-sample t-tests for the anisotropy assessment.

*Anisotropy is verified.

Table 4. P-values obtained with the two-sample t-tests for the assessment of the difference significance.

Direction	Compared GM	TYS	PYE	TBS	PBE
MD	Virgin and S1	0.02^{*}	0.17	0.55	0.68
	Virgin and S2	0.02^{*}	0.73	0.00^{*}	0.00^*
CD	Virgin and S1	0.00^{*}	0.01^{*}	0.08	0.01^{*}
	Virgin and S2	0.30	0.00^{*}	0.00^{*}	0.00^{*}

*Difference is significative.

Regarding the yield properties, TYS of both exhumed GM are significantly higher than TYS of virgin GM (except for S2 in CD) while there is no statistically significant difference for the PYE (except in CD). That means the exhumed GMs are stiffer than the typical virgin HDPE GM. These observations can be observed in Figure 4 that presents box plots of TYS and PYE, verified with the statistical analyses in Table 4.

Regarding the break properties, there is no significant statistical difference between the TBS and PBE results for the GM exhumed from the site S1 and the virgin GM (except the PBE in CD); while TBS and PBE are significantly higher for the GM exhumed from the site S2 than the virgin GM. The GM exhumed after 20 years (site S2) is more resistant than the typical virgin one. The GM used at that time could be more resistant than those manufactured more recently.

All the tensile properties of the exhumed GMs are at least similar to those of the typical virgin GM.

5 DISCUSSIONS

The above data indicate that the exhumed GMs are stiffer than the typical virgin HDPE GM. It would be then interesting to know how this gain of stiffness could impact the design criteria concerning the maximum allowable strain (MAStrain). Indeed, the GM could also break under a constant load, even in the elastic zone, due to stress cracking, particularly for HDPE GMs owing to their high crystallinity (Hsuan et al. 1993; Müller 2007; Scheirs 2009). Hence, some authors use the MAStrain as the strain that should not be exceeded to ensure the durability of the GM (Dixon & von Maubeuge 1992; Eldesouky and Brachman 2018; Jones et al. 2000; Peggs et al. 2005; Rowe & Yu 2018; Rowe & Yu 2019; Rowe et al. 2020). Initially, a MAStrain of 6% was proposed for HDPE GMs (Dixon & von Maubeuge 1992; Jones et al. 2000). Different MAStrain values were subsequently proposed, depending on the GM use, for example, from 4 to 5% for GMs used as a cover which consider a safety factor of 1.5 to 1.2, respectively, compared to the initially proposed 6% (Rowe & Yu 2019), and 3% for GMs used as a liner which means a safety factor of 2 (Jones et al. 2000; Rowe & Yu 2019; Rowe et al. 2020).

In this study, the GMs were exhumed from cover so the MAStrain considered is set at 4% with a safety factor of 1.5. To assess the impact of the gain of stiffness on the MAStrain, consider first the maximum allowable stress (MAStress₀) that corresponds to the initial MAStrain of 4% of the virgin GM. MAStress₀ is determined graphically with the linear stress-strain curve obtained with the tensile test of a virgin GM (Figure 6.a) as 17.7 N/mm and 18.1 N/mm in MD and CD respectively. These values are then used to plot the corresponding strain on the stress-strain curve of the GM exhumed from the sites S1 and S2 (Figure 6.b). These values will be the strains that would be needed to mobilize the MAStress₀ and would be then the MAStrain that should not be exceeded.



Figure 6. Determination of the MAStress₀ (a) and the MAStrain_{S1} and MAStrain_{S2} (b).

Figure 7 presents the obtained MAStrain of 3.55 % and 3.04 % for S1, in MD and CD, respectively; and 3.71 % and 3.73 % for S2, in MD and CD, respectively. All the data are significantly drawn from a normal distribution as all the p-values are higher than the significance level of 0.05. The two-sample t-tests can then be performed to compare the results of the virgin GM to the exhumed ones (see Table 5). The obtained MAStrains are significantly lower than the initial MAStrain of 4% as the p-values are lower than the significance level of 0.05, except for S2 in CD where the p-value of 0.06 is above the significance level (see Table 5). The strain corresponding to the MAStress0 would be lower than it was initially, which means that the maximum allowable stress is mobilized earlier. The critical stress could be reached before the critical strain considered in the design. It is important to note that the GM behaviour could change over time. In this case study, there is a gain of stiffness that could negatively impact the design criteria.



Figure 7. Box plots of the maximum allowable strains determined for the typical HDPE GM and the exhumed GMs.

Direction	GM	p-value of the normality test	p-value of the two-sample t-test
MD	Virgin	1.00	0.01
	S1	1.00	
	Virgin	1.00	0.03
	S2	0.52	
CD	Virgin	1.00	0.00
	S1	0.94	
	Virgin	1.00	0.06
	S2	0.92	

Table 5. P-values of the normality tests and the two-sample t-tests on the obtained MAStrains.

6 CONCLUSIONS

This paper presents actual physical, chemical, and mechanical properties of 1.5 mm-thick HDPE geomembranes exhumed from cover systems used for the reclamation of two mine sites (S1 and S2) that have 13 and 20 years of service, respectively. Results are compared to those of a typical virgin HDPE geomembrane with the same thickness. It can be concluded that:

- The antioxidant and stabilizer level of the S1 geomembrane is close to that of the virgin one, and the S2 geomembrane degradation is in an advanced state;
- The exhumed HDPE geomembranes still contain antioxidants and stabilizers, and remain in the antioxidant depletion stage during which no negative impact is expected on the mechanical properties;
- The exhumed HDPE geomembranes are stiffer than the virgin one and may have better resistance.

The effect of the tensile behaviour on the design criteria of the maximum allowable strain is that the gain of stiffness reduces the MAStrain, which could impact negatively the design criteria.

However, it should be noted that the comparisons were made with a typical virgin HDPE geomembrane whose properties could be different of the initial properties of the exhumed geomembranes. This was done for comparison purpose only. A large and complete characterization of the geomembranes should be done before its installation to complete the data provided by the manufacturer. These data should be then available for a better understanding of the in-service durability of geomembranes. In the absence of the initial properties of the GM as installed, the results presented in this study constitute reference values for future in-service performance assessments.

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