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Article

PERFORMANCE OF AN ENVIRONMENTAL PROTECTION LINER AFTER LABORATORY UV EXPOSURE

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High-density polyethylene (HDPE) geomembranes are commonly used as an environmental protection liner due to their good chemical and mechanical resistances and low cost. Ultraviolet (UV) radiation is an essential issue in durability studies for pond applications. This study evaluated a 1.5-mm thick HDPE geomembrane exposed to ultraviolet fluorescent radiation for 8760 h in a laboratory and thermoanalytical and physical analyses were conducted towards the understanding of its performance after exposure. According to the results, although the geomembrane maintained the ductile behavior, it showed a 52.48% final decrease in stress crack resistance (SCR) compared to virgin SCR. Moreover, a considerable antioxidant depletion occurred after 8760 h exposure shown by the Std. OIT (standard oxidative-induction time) results, demonstrating a Std. OIT value decrease of 89.19% compared to the virgin Std. OIT. Such a behavior contributed to the susceptibility of thermal effects in the DSC (differential scanning calorimetry) curves and the losses observed in the SCR values, attesting the geomembrane's oxidative degradation mechanism occurred and changed the polymer's structure.

Keywords: geomembrane; high-density polyethylene; ultraviolet radiation; durability; physical and thermal analysis.

INTRODUCTION

Geosynthetics used as environmental protection barriers are polymeric sheets with low permeability coefficients; they are sometimes combined with natural materials, industrially manufactured, and installed in the field. Geomembranes are a type of geosynthetics commonly employed for landfill liners since the 1970s. However, they are currently applied as a liner in water ponds, industrial waste ponds, and waste liquid ponds. High-density polyethylene (HDPE) geomembranes show high chemical and mechanical resistance, associated with a low manufacturing cost and a very low permeability coefficient, i.e., typically 10^{-11} to 10^{-13} cm s^{-1}.[1-6]

Geomembranes are exposed to climate conditions during the construction time for landfill applications. However, regarding pond applications, exposure to climate conditions on slopes above the water table continues for a lifetime and an exposed geomembrane can initiate UV, thermal, and oxidative degradations. Such aging mechanisms can influence the properties of the materials, decreasing durability[7,8] and the synergy between UV radiation and thermal exposure can degrade the material.[9,10] When UV radiation reaches the geomembrane surface, photo-oxidation starts to act, generating several free radical reactions, hence, degradation and polymer chain scission and polymer property degradation.[11,12]

The oxidation process of HDPE degradation starts with the free radical chain mechanism. The oxidation mechanism involves two cycle processes, namely, a chain reaction of alkyl/alkylperoxyl and formation of new radicals by chain reaction (homolysis of hydroperoxides). Oxidation can be stopped if all links are blocked.[13,14] The HDPE geomembrane formulation includes 2-3% of UV protection (usually carbon black) and 0.5-1.0% of two different types of antioxidants (primary and secondary) are used to prevent the oxidation of the polymer during extrusion and guarantee the longevity of the material.[15,16]

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Several studies have analyzed the influence of UV radiation and weather effects on HDPE materials. Sahu *et al.*[17] studied 1 to 3% carbon black concentrations in an HDPE using a UV fluorescent weatherometer for 192 h and both differential scanning calorimetry (DSC) and Fourier transform infrared spectroscopy (FTIR) analyses revealed the total carbon black concentrations protected the resin adequately with no HDPE degradation. Reis *et al.*[18] chose eight different regions in Portugal for analyses of five 2.0 mm-thick HDPE geomembranes after 12 years of field exposure. Locations in the country with higher UV indexes suggest an impact on tensile properties and antioxidant depletion. The study also demonstrated that HDPE geomembranes covered with nonwoven geotextiles and uncovered displayed the same behavior. Lavoie *et al.*[19] evaluated the final condition of a 0.8 mm-thick HDPE geomembrane that had been over 15 years in contact with waste and environmental conditions in a biodegradable waste pond. The exhumed sample exhibited brittle tensile behavior, low stress crack resistance, and almost an entire antioxidant depletion, leading to the conclusion its final condition would cause a rupture, hence, an environmental impact on the site. Lavoie *et al.*[20] analyzed an HDPE geomembrane exhumed from a liner in an industrial water pond after 2.25 years of operation and the results showed a mechanical brittle behavior, indicating changes in the polymer morphology.

Safari *et al.*[21] studied the antioxidant depletion of an exhumed HDPE geomembrane installed 25 years ago in a hazardous waste landfill in Canada. The samples were exhumed from the bottom and the cover liners. The authors concluded the Std. OIT (standard oxidative-induction time) test values and some HP OIT (high-pressure oxidative-induction time) test levels were significantly lower than those of a modern virgin geomembrane and the exposure conditions significantly influenced the antioxidant depletion of the geomembrane. Mendes *et al.*[22] analyzed the behavior of an HDPE using a phenol-type antioxidant and two HALS-type light stabilisers (hindered amine light stabilizer) as additives in the HDPE resin after 4000 h weathering exposure in Rio de Janeiro, Brazil. The samples

were compared with and without additives. The former displayed a good behavior after exposure, whereas those with no additives showed losses in ductility, an increase in crystallinity, and a reduction in the molecular weight.

This study evaluated the behavior of a virgin 1.5-mm thick HDPE geomembrane after a UV fluorescent exposure for 8760 h. Thermoanalytical and physical analyses were conducted towards the understanding of the final conditions of the sample.

EXPERIMENTAL

HDPE geomembrane

A 1.5 mm-thick HDPE smooth geomembrane provided by a Brazilian manufacturer, formulated with 96-97.5% medium-density polyethylene (density ≥ 0.940 g cm^{-3}), 2-3% anti-UV additive (carbon black), 0.5-1.0% thermostabilizers and antioxidants,[23] and produced by the extrusion blown film process was used. According to Ewais *et al.*,[24] antioxidants, stabilisers, and carbon black retard polymer degradation due to photo-oxidation and thermal oxidation.

Accelerated weathering exposure

A UV-weathering chamber, model EQUV Philips, from Equilam (Diadema, Brazil) with fluorescent UVA-340 lamps was used and programmed to work in cycles of 20 ± 0.01 h of UV light at 75 ± 1 °C followed by 4 ± 0.01 h of condensation at 50 ± 0.01 °C[25] for 960, 4380, and 8760 ± 0.01 h.

Melt flow index (MFI) test

A plastometer, model CEAST MF20, manufactured by Instron (Norwood, USA) ran the MFI test.[26] The material was extruded at 190 ± 0.08 °C with a 5.0 ± 0.01 kg of deadweight in a smooth bore of 2.095 ± 0.005 mm (diameter) and 8000 ± 0.025 mm (length) and then measured by an analytical balance with 0.0001 g precision in 10 ± 0.01 min.

Tensile test

The tensile test[27] was conducted in a universal machine with a 2-kN load cell, pneumatic grips, IV dog bone specimen, and at 50 ± 0.05 mm min^{-1} test speed. The material was analyzed regarding tensile at break in the machine direction, model DL 3000 (EMIC, São José dos Pinhais, Brazil).

SCR test

The stress cracking was evaluated in equipment manufactured by WT Indústria (São Carlos, Brazil) with capacity to test 20 specimens simultaneously. The NCTL-SP (notched constant tensile load test - single point)[28] prescribes the specimen's immersion in a solution with 10 ± 1% Igepal CO 630 and 90 ± 1% water at 50 ± 1 °C and application of 30% of the sample's yield strength (10 g precision) in five specimens notched with 20% of their thicknesses (0.001 mm precision). The result was recorded in rupture time with 1 second precision.

OIT tests

OIT tests were conducted in two phases, i.e., an endothermic reaction with nitrogen gas purge followed by specimen oxidation, both in a DSC equipment model Q20 manufactured by TA Instruments (New Castle, USA). The Std. OIT[29] was conducted at 200 ± 2 °C with 140 ± 5 kPa constant oxygen pressure, 20 ± 1 °C min^{-1} heating rate, and 50 ± 5 mL min^{-1} flow rate, whereas the HP OIT[30] was conducted at 150 ± 0.5 °C with 20 ± 1 °C min^{-1} heating rate and 3.4 ± 0.06 MPa constant oxygen pressure.

DSC analysis

The analysis was performed in a DSC equipment model Q20 manufactured by TA Instruments (New Castle, USA) using nitrogen gas purge with 50 ± 5 mL min^{-1} flow, an aluminum crucible with 10 ± 0.5 mg sample mass, 10 ± 1 °C min^{-1} heating rate, and 25 to 200 ± 2 °C temperature range.

RESULTS AND DISCUSSION

Sample characterisation results

Table 1 shows the initial and minimum property values of the samples required by American standard GRI-GM13.[31]

The characterisation results of the sample showed non-compliance with the American standard for the HP OIT test results, since the

Table 1. Initial property values of HDPE geomembrane samples and minimum property values required by the American standard

Property	Method	Mean value ± SD	GRI-GM13
Thickness / mm	ASTM D5199[32]	1.652 ± 0.039	≥ 1.50
Density / (g cm^{-3})	ASTM D792[33]	0.945 ± 0.001	≥ 0.940
MFI (5 kg/190 °C) / (g 10 min^{-1})	ASTM D1238[26]	0.5054 ± 0.0102	-
Carbon black content / %	ASTM D4218[34]	2.92 ± 0.12	2.0-3.0
Carbon black dispersion (category)	ASTM D5593[35]	10 different views in category I	10 different views: 9 in categories I or II and 1 in category III
Tensile break resistance / (kN m^{-1})	ASTM D6693[27]	46.93 ± 7.04	≥ 40
Tensile break elongation / %	ASTM D6693[27]	704.67 ± 101.30	≥ 700
Tear resistance / N	ASTM D1004[36]	242.53 ± 0.40	≥ 187
Puncture resistance / N	ASTM D4833[37]	677.30 ± 16.47	≥ 480
SCR / h	ASTM D5397[28]	629.84 ± 67.55	≥ 500
Std. OIT / min	ASTM D3895[29]	199.78 ± 4.03	≥ 100
HP OIT / min	ASTM D5885[30]	287.25 ± 1.06	≥ 400

SD: standard deviation; MFI: melt flow index; SCR: stress crack resistance; Std. OIT standard oxidative-induction time: HP OIT: high-pressure oxidative-induction time.

minimum HP OIT value required is 400 min. However, the HP OIT test result was approximately 70% of the minimum value required, showing the additive package of the sample probably has no HALS. Moreover, the tensile elongation test result showed a high standard deviation.

MFI test results

Table 2 shows the MFI test results for both virgin and UV fluorescent exposure samples after 960, 4380, and 8760 h and Figure 1 displays the behavior of the samples after exposure, exhibited in retained MFI results.

Table 2. MFI test results after UV fluorescent exposure and retained MFI test values compared with the virgin sample values

Exposure time / h	MFI mean value ± SD / (g 10 min^{-1})	MFI / %
0	0.5054 ± 0.0102	100.0
960	0.5088 ± 0.0132	100.68
4380	0.5045 ± 0.0225	99.82
8760	0.4978 ± 0.0109	98.49

SD: standard deviation. MFI: melt flow index.

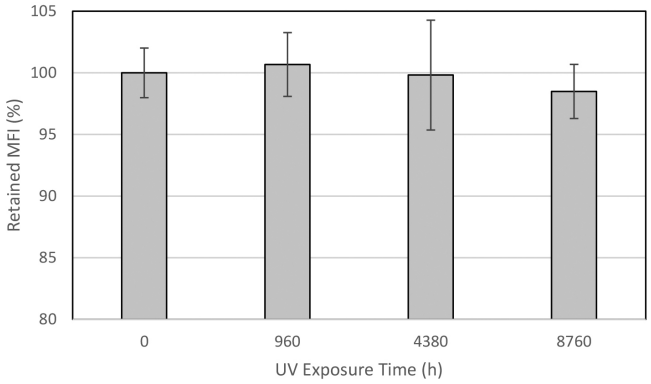

Figure 1. *Retained MFI test results compared with virgin sample test result after UV fluorescent exposure*

According to Gulec *et al.*,[38] the MFI test is commonly used as a molecular weight index for chemical compatibility studies; it is also a simple method for assessments of a molecular weight of the polymer. Minor variations in MFI test results were observed among the exposure samples. After 960 h of exposure, the result increased 0.68%; however, after 4380 and 8760 h of exposure, they decreased 0.18 and 1.51%. The MFI result after 8760 h demonstrates an influence of UV exposure on the polymer.

Tensile test results

The tensile properties evaluated were resistance and elongation at break. Table 3 shows the results for both virgin and UV fluorescent exposure samples after 960, 4380, and 8760 h and Figure 2 displays the behavior of the samples after exposure, exhibited in retained tensile properties results.

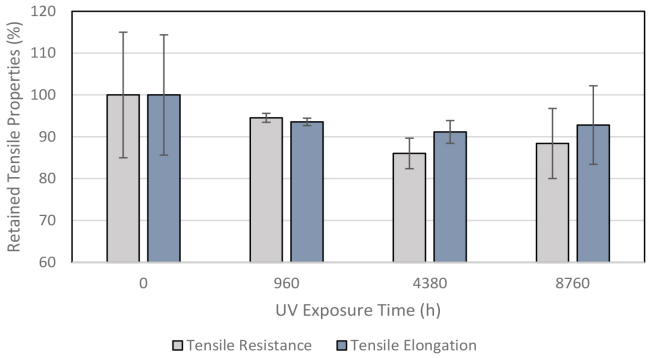

Figure 2. *Retained tensile test results (resistance and elongation at break) compared with virgin sample test result after UV fluorescent exposure*

Firstly, the virgin sample showed a high standard deviation due to a 39.25 kN m^{-1} tensile resistance at break and a 596.50% tensile elongation at break of the specimen.

The tensile resistance at break results decreased 5.48, 13.97, and 11.60%, respectively, for 960, 4380, and 8760 h UV exposure, compared to the virgin sample. On the other hand, the tensile elongation at break decreased 6.44, 8.85, and 7.20%, respectively, for 960, 4380, and 8760 h UV exposure compared to the virgin sample. The slight increase in the tensile properties' values between 4380 and 8760 h can be attributed to a variation in the manufacturing process. Koerner *et al.*[39] exposed a 1.5 mm-thick HDPE geomembrane for 28,000 h using a UV fluorescent weatherometer. The sample showed an approximately 20% higher decrease in the tensile properties (resistance and elongation) in comparison to the present study.

Lavoie *et al.*[40] analyzed a 1.0 mm-thick HDPE geomembrane after 8760 h of UV fluorescent exposure and reported an approximately 30% decrease in both resistance and elongation tensile values during the exposure times. The sample analyzed in the present study showed lower decreases in the tensile properties values after UV exposure, hence, a better behavior.

SCR test results

Table 4 shows the SCR test results for both virgin and UV fluorescent exposure samples after 960, 4380, and 8760 h and Figure 3 displays the behavior of the sample after exposure, exhibited in retained SCR.

The SCR results after 960, 4380, and 8760 h UV exposure decreased, respectively, 6.75, 20.36, and 52.48%, compared to the initial value. The SCR retained value after 1 year (8760 h) of laboratory UV exposure obtained in this study (47.52%) is similar to the SCR mean retained value reported by Rowe *et al.*[41] for eleven

Table 3. Tensile test results (resistance and elongation at break) after UV fluorescent exposure and retained tensile test values compared with the virgin sample values

Exposure time / h	Tensile resistance mean value ± SD / (kN m^{-1})	Tensile resistance / %	Tensile elongation mean value ± SD / %	Tensile elongation / %
0	46.93 ± 7.04	100.0	704.67 ± 101.30	100.0
960	44.36 ± 0.48	94.52	659.30 ± 5.83	93.56
4380	40.37 ± 1.47	86.03	642.30 ± 17.46	91.15
8760	41.48 ± 3.47	88.40	653.90 ± 61.35	92.80

SD: standard deviation.

Table 4. SCR test results after UV fluorescent exposure and retained SCR test values compared with the virgin sample values

Exposure time / h	SCR mean value ± SD / h	SCR / %
0	629.84 ± 67.55	100.0
960	587.32 ± 24.13	93.25
4380	501.62 ± 29.72	79.64
8760	299.29 ± 18.39	47.52

SD: standard deviation. SCR: stress crack resistance.

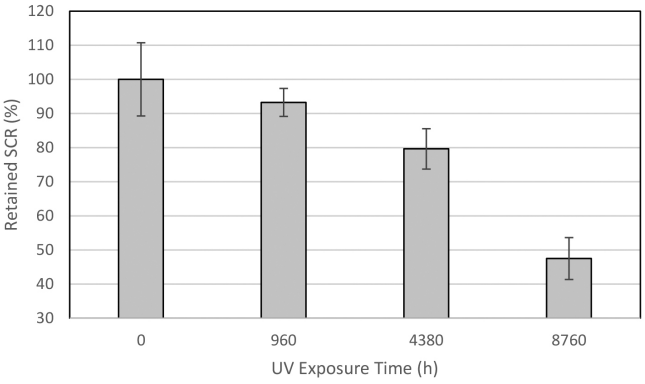

Figure 3. *Retained SCR test results compared with virgin sample test result after UV fluorescent exposure*

HDPE geomembranes immersed in leachate (37%), who suggest the SCR value can stabilize after 3 months of leachate incubation.

OIT test results

Table 5 shows the Std. OIT and HP OIT test results for both virgin and UV fluorescent exposure samples after 960, 4380, and 8760 h and Figure 4 displays the behavior of the samples after exposure, exhibited in retained OIT values.

The Std. OIT test results decreased 16.48, 27.99, and 89.19%, respectively, for 960, 4380, and 8760 h of UV exposure in comparison to the virgin sample result. A considerable decrease in the Std. OIT results was observed between 4380 and 8760 h of UV exposure. After 1-year exposure, the depletion of the antioxidants was almost complete and chain reaction and molecular composition started. Lavoie *et al.*[42] evaluated two 1.0 mm-thick exhumed HDPE geomembranes in mining facility constructions in Brazil and both showed low Std. OIT values, brittle tensile behavior, and low SCR values.

Nonetheless, the HP OIT test results decreased 6.52, 29.74, and 53.15%, respectively, for 960, 4380, and 8760 h of UV exposure compared to the initial value and decreased less than the Std. OIT test results. According to Abdelaal and Rowe,[43] the HP OIT test results after HDPE geomembrane samples heat exposure conducted to a high residual value. However, Lavoie *et al.*[44,45] obtained HP OIT value equal zero for one of the analyzed samples in the research, demonstrating that is possible to completed deplete the antioxidants for this test.

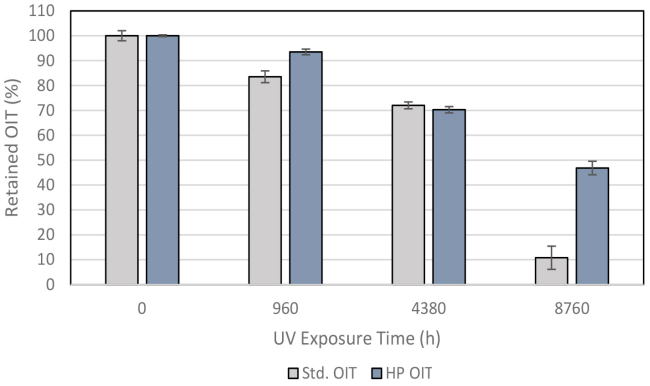

Figure 4. *Retained Std. OIT and HP OIT tests results compared with virgin sample test result after UV fluorescent exposure*

DSC analysis

Figure 5 shows the DSC curves for the first and second heatings (Figure 5a) to verify the behavior of melting point and cooling (Figure 5b) and understand the behavior of the crystallisation point. The first heating (except for the virgin sample) led to a deviation in the DSC curve before the melting point in the following temperature ranges: 96-105 °C for the 8760 h-sample, 82-93 °C for the 4380 h-sample, and 80-89 °C for the 960 h-sample. The deviation is attributed to the effect of exposure of the material to UV radiation, since no changes were observed in the DSC curve of the virgin sample. In the second heating, the samples exhibited a unique curve behavior due to the homogenization effect of the material after the first heating. Figure 5b shows that crystallisation is similar among the samples in function of the homogenization after melting. The homogenization caused by the material's melting displayed a new polymeric configuration, since the crystallisation points are coincident and the second evaluation of the melting point is also coincident. The essential DSC evaluation was the first heating analysis, which revealed a deviation from the samples' baseline.

Correlation between properties

The sample's tensile properties increase after 8760 h of UV exposure in comparison to the sample values after 4380 h exposure can be attributed to the polymer's crystallinity changes due to the UV radiation degradation mechanism. A UV radiation aging influence is observed in the MFI test, since the value decreased for the sample after 8760 h of UV exposure compared to the sample value after 4380 h of exposure, correlating with the tensile test results. Moreover, the high loss in the SCR value after 8760 h of UV laboratory exposure corroborates the DSC curve deviation before the melting point, which increased after the three exposure times evaluated. Despite the maintenance of the ductility of the geomembrane after the UV exposure, the SCR test results revealed a brittle failure behavior. After 8760 h of UV exposure, the antioxidants' depletion (Std. OIT) was almost complete, showing the level of

Table 5. Std. OIT and HP OIT tests results after UV fluorescent exposure and retained OIT test values compared with the virgin sample values

Exposure time / h	Std. OIT mean value ± SD / min	Std. OIT / %	HP OIT mean value ± SD / min	HP OIT / %
0	199.78 ± 4.03	100.0	287.25 ± 1.06	100.0
960	166.85 ± 3.94	83.52	268.52 ± 3.04	93.48
4380	143.87 ± 1.98	72.01	201.81 ± 2.53	70.26
8760	21.59 ± 1.01	10.81	134.57 ± 3.67	46.85

SD: standard deviation. Std. OIT standard oxidative-induction time: HP OIT: high-pressure oxidative-induction time.

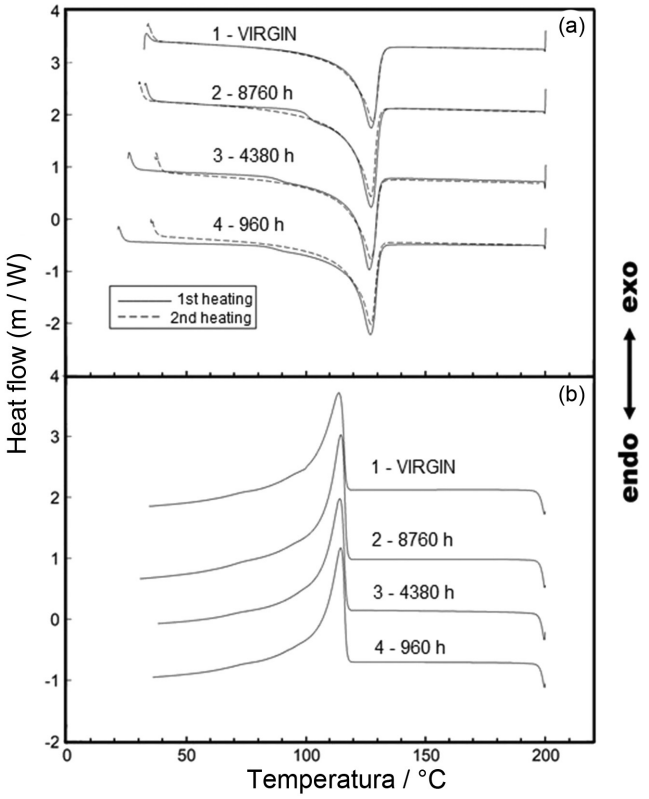

Figure 5. *DSC curves: (a) melting point (heating) stage and (b) crystallisation (cooling) stage. (1) Virgin sample; (2) 8760 h UV fluorescent sample; (3) 4380 h UV fluorescent sample; and (4) 960 h UV fluorescent sample*

degradation resulting from the exposure is directly associated with the depletion of the antioxidant. Such a behavior contributes to the susceptibility of thermal effects in the DSC curves, attesting the mechanism of oxidative degradation of the geomembrane occurs and changes the structure of the polymer.

CONCLUSIONS

This study analyzed a 1.5 mm-thick HDPE geomembrane exposed to UV radiation for 8760 h in the laboratory.

Although the geomembrane maintained the ductile behavior after exposure, it showed a 52.48% SCR final decrease in comparison to the virgin SCR result, demonstrating its susceptibility for the stress cracking phenomenon.

A considerable antioxidant depletion was observed after 8760 h exposure according to the Std. OIT results, reaching a 89.19% decrease in comparison to the virgin Std. OIT result. Regarding thermal behavior, the DSC curves showed a deviation before the melting point, which is attributed to the effect of exposure of the material to UV radiation, since the DSC curve of the virgin sample showed no changes.

Finally, such an antioxidant depletion behavior contributed to the susceptibility of thermal effects in the DSC curves and the losses in the SCR values, attesting the geomembrane's oxidative degradation mechanism occurs and changes the polymer's structure.

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