

# Proposal of evaluation analysis and long-term performance of geomembrane with material composition through stress crack tests

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**ABSTRACT:** Stress cracking time was differed from each type of geomembrane(GM) resins and products. The failure times of ESCR(environmental stress crack resistance) test were too long compared to SP-NCTL(single point-notched constant tensile load) test. But there was no difference between each sample of the results of ESCR test within short term testing duration by resin. ESCR test was influenced by stress relaxation during the test. SP-NCTL test is reasonable for verifying stress cracking behavior and construction quality control. According to this study, ESCR test is not appropriate for determining the stress crack behavior, but SP-NCTL test is better test method for predicting the stress crack behaviors of HDPE GM.

*Keywords: geomembrane, stress crack, ESCR(environmental stress crack resistance), SP-NCTL(single point-notched constant tensile load)*

## 1 INTRODUCTION

To prevent the stress cracking of HDPE geomembranes for landfill construction, various additives are mixed with the polyethylene resins, and the stress cracking resistance of the material is improved by the additives. Typical tests to check the stress cracking resistance are ESCR method and SP-NCTL method, which sometimes show rather different results and take much different time till they derive the results.

It was seen that ESCR method is not appropriate for the evaluation of stress cracking resistance because it showed the difference from the actual cracking in the field. In USA, its performance evaluation method and criteria were changed after the experimental verification.

The purpose of this study is to propose the level-up of product criteria in consideration of the safety rate applied to the field construction by evaluating the performance level of the products manufactured in Korea and used in the field.

Before studying the products used in the field, it is necessary to examine the appropriateness of ESCR method, which is actually being used to check the stress cracking of the product in the field, by collecting the resins used as raw material for Korean products and measuring their characteristic values related with stress cracking to compare and analyze them.

## 2 EXPERIMENTAL

To compare ESCR method and SP-NCTL method that have been proposed as the evaluation method for stress cracking behavior and performance of HDPE water-blocking material which is used for the construction of waste landfill in Korea, two kinds of chip were manufactured: one that the additives were mixed with the raw material resin with the additive (RWA) used for Korean products and one the raw material resin without the additives (RWOA).

The chips were compressed with the compression molder for 30 min at the pressure of 500psi and the temperature of 170°C to have the form of sheet and then cooled at the rate of  $(15 \pm 2)^\circ\text{C}/\text{min}$ .

The specimen without the additives (RWOA) did not contain the carbon black and the antioxidant but the specimen with the additives (RWA) contained both of them. Additionally, GM1 and GM2 regional HDPE GM products were prepared for comparing with resin samples.

Table 1. Characteristics of raw material resin

Test Samples	Thickness	Density
	ASTM D 5199	ASTM D 1505
Resin without additives (RWOA)	1.85 mm	0.931 g/cm <sup>3</sup>
Resin with additives (RWA)	2.51 mm	0.945 g/cm <sup>3</sup>
Geomembrane 1 (GM 1)	2.01 mm	0.946 g/cm <sup>3</sup>
Geomembrane 2 (GM 2)	2.05 mm	0.946 g/cm <sup>3</sup>

The stress cracking characteristics of two specimens were evaluated to analyze the mechanism necessary for the selection of appropriate evaluation method and the set-up of criteria. For this, the content of carbon black, the melt flow index and the oxidative induction time were checked along with ESCR test and NCTL test.

To find out the contents of carbon black, the polyethylene was carbonized and the weight of residual ashes was measured with the thermo gravimetric analysis (TGA) using the principle in ASTM D1603. For the melt flow index, the load of 2.16kg was applied for 10 min, as regulated in ASTM D1238, and the weight of extruded polyethylene resin was measured.

The oxidative induction time was obtained by measuring the oxidation time of each specimen under the standard compressive pressure in ASTM D3895. For ESCR test, it is necessary that the specimen folded and fixed with a jig is immersed in the Igepal CO-630 solution in a test tube and sealed to be treated for a long time at 50°C. So, the thermostat chamber which is regulated in ASTM D1693 was used. In SP-NCTL test, the specimen is immersed in the test solution of 50°C for a long time while receiving a certain amount of load and its cracking time is measured. For this, the NCTL-dedicated equipment regulated in ASTM D5397 was used. For ESCR test, the test condition in the standard of the waste landfill installment was applied; for SP-NCTL test, 30% of the yield stress strength was applied.(Table 1 and Figure 1)

Table 1. NCTL test condition

pH	Applied stress (yield%)	Temp. (°C)	Duration (hour)
4	25%, 30%, 35%, 40%	50°C	1,200
12	25%, 30%, 35%, 40%	50°C	1,000



Figure 1. Apparatus for NCTL test of geomembrances

### 3 RESULTS AND DISCUSSION

#### 3.1 Physical properties

The carbon black is used in the resin to prevent the embrittlement of the material by the ultraviolet rays while being exposed outside for a long time when the HDPE GE product is covered on the field. In the product containing a certain amount of carbon black, the embrittlement of the material by UV rays can be prevented. And the result value of SP-NCTL gets less due to the carbon black. RWOA specimen did not contain the carbon black at all, but the carbon black contents of RWA, GM1 and GM2 specimen was 1.8%, 2.1% and 1.7%, respectively.

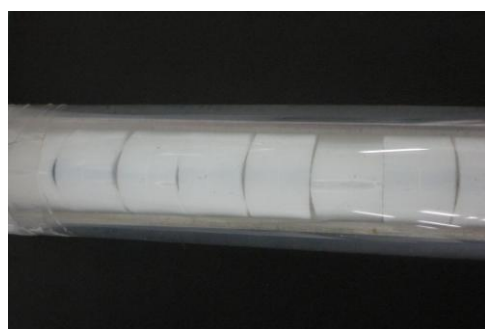
According to some reports, if the carbon black mixed to increase the resistance against UV goes over a certain amount, the stress cracking of the material decreases. So, it is important to adjust its mixing amount for the management of product quality. In case of specimens used for this test, it seems very probable for the specimen with carbon black to have the stress cracking. MI value which is determined to some degree at the production stage of the resin has a relation with the molecular weight. As the MI value gets higher, the stress cracking tends to increase. RWOA specimen had MI value of 0.063 g/10min but the MI value of RWA, GM1 and GM2 specimen were 0.241 g/10min, 0.224 g/10min and 0.191 g/10min, respectively. RWA, GM1 and GM2 specimens showed a relatively higher value in comparison with RWOA specimen. The increase seems to be caused by the addition of carbon black, and so the possibility of the stress cracking can be said to get relatively higher.

#### 3.2 Oxidative induction time

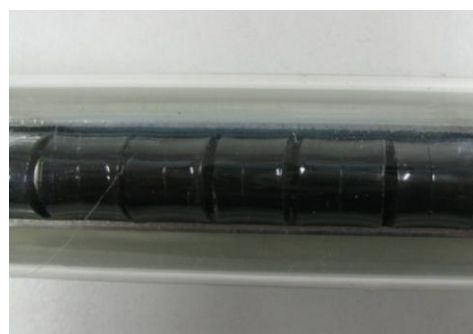
It is known that the material with UV stabilizer and antioxidant suffers less embrittlement by the oxidation than the materials without them when exposed to the external environment for a long time. But, once the antioxidant is totally dissipated, the oxidation occurs rapidly to degrade the physical properties. Oxidative induction time of RWA, GM1 and GM2 specimen were measured as 120min, 150min and 193min, respectively but in case of RWOA, it was about 5min. When comparing with RWOA and RWA specimens, we can see that the antioxidant prevents the embrittlement of the material for a considerable time and thus the stress cracking, too.

#### 3.3 Environmental stress crack resistance

After giving a notch on its center, the specimen was folded by 180° and fastened with a holder. Then, it was immersed in 10% Igepal CO-630 solution. The container with the specimen was put into the thermostat of 50°C for 1,500 hours and then the cracking of the specimen was observed. Both of RWOA, RWA, GM1 and GM2 specimens did not show the stress cracking due to the long aging. As mentioned in the previous study, it seems that the stress relaxation of specimen has happened in the process of ESCR test. RWOA specimens were failed after 1,800 hours.( Figure 2)



(a) RWOA



(b) RWA

Figure 2. ESCR Specimens after 1,800 hours.

### 3.4 Notched constant tensile load

To find out the load equivalent to 30% of the yield tensile stress, a tensile test was carried out according to ASTM D 638. In accordance with the SP-NCTL test method in the appendix of ASTM D 5397, small-sized test specimens were collected for the tensile test and were immersed in the 10% Igepal CO-630 surfactant solution of the test equipment while the calculated stress is applied to each specimen according to the measured yield tensile strength. For RWOA specimen, the stress cracking was generated after around 150 hours; for RWA, GM1 and GM2 specimens did not happen even after 200 hours. With this, it could be said that the additive gave an influence on the stress cracking.(Table 2)

Table 2. Test Results in Characteristic Items of each Specimen

Test Properties	Units	Test Method	Test Samples			
			RWOA	RWA	GM1	GM2
Carbon Black Contents	%	ASTM D 1603	0	1.8	2.1	1.7
Melt Flow Index	g/10min	ASTM D 1238	0.063	0.241	0.224	0.191
Oxidative Induction Time	min	ASTM D 3895	5	120	150	193
ESCR	hrs	ASTM D 1693	over 1,500	over 1,500	over 1,500	over 1,500
<b>SP-NCTL</b>	<b>hrs</b>	<b>ASTM D 5397</b>	<b>151</b>	<b>over 200</b>	<b>Over 200</b>	<b>Over 200</b>

In the basic condition of the specimen, RWOA has the relatively lower density and melting index than RWA, GM1 and GM2, therefore the stress cracking is hard to happen. But, in NCTL test, the stress cracking happened faster in RWOA than in RWA, GM1 and GM2. It is because, even though the high density and melting index of RWA, GM1 and GM2 specimen may increase the occurrence possibility of stress cracking, the antioxidant added with the carbon black increased the oxidation resistance and thus the stress cracking resistance.

As a factor in the extruding process, RWOA specimen was compressed and molded about 0.5mm thinner than RWA specimen. Thus, RWOA specimen showed higher result in NCTL and ESCR tests than expected. It seems because RWOA specimen which was thinner than RWA specimen got cooled faster in the process and its crystallinity degree became lower.

With this, we could confirm the problem that, in ESCR test, the stress of the specimen became relaxed, making the test period longer, and the stress cracking phenomenon that can generate in the real field could not be quantified because the cracking time of the specimen was not known correctly. On the other hand, in SP-NCTL test, the time necessary for quantifying the results and confirming the quality for the characteristics of specimens was relatively short. Therefore, it seemed more appropriate method to evaluate the quality of the products which are used in the actual field.

## 4 CONCLUSION

This study has some limit in understanding the stress cracking because it compared and analyzed just two resin specimens that are used as the raw material of HDPE GM. According to the results of this study, we can propose the revision of specification and test method of stress cracking resistance to SP-NCTL method instead of ESCR method in KPS M 6000 standard for the stable quality management of waste landfill.

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