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## EVALUATION OF STRESS CRACKING ON GEOMEMBRANES AFTER ACCELERATED TESTS

# EVALUACIÓN DE FISURACIÓN BAJO TENSIÓN DE GEOMEMBRANAS POS ENSAYOS ACELERADOS

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**ABSTRACT:** This paper presents results of stress cracking tests performed in high density polyethylene (HDPE) geomembranes (GM). Stress cracking tests were performed in accordance to ASTM D5397: Notched Constant Tensile Load Test (NCTL) and Single Point-Notched Constant Tensile Load Test (SP-NCTL). Tests were conducted to the fresh sample at 50°C (standard test) and at 70°C (accelerated condition) in order to compare the SC values. Results from accelerated tests (NCTL) showed, for instance, a total economy of 390 hours (comparing load stages of 25% yield stress) to perform the tests.

Key words: HDPE geomembranes, stress cracking, accelerated tests.

**RESUMEN:** Este trabajo presenta resultados de fisuración bajo tensión realizados con geomembranas (GM) de polietileno de alta densidad (HDPE). Las pruebas de tensión fueron realizados de acuerdo a la norma ASTM D5397: Notched Constant Tensile Load Test (NCTL) and Single Point-Notched Constant Tensile Load Test (SP-NCTL). Estos ensayos fueron realizados en muestras de control a 50 ° C (ensayo padrón) y a 70 ° C (condición acelerada) para comparar valores de fisuración bajo tensión. Los resultados de estos ensayos acelerados (NCTL) mostraron una economía total de 390 h (comparando etapas de carga de 25% de la tensión de ruptura) para los ensayos realizados.

Palabras clave: Geomembranas de polietileno de alta densidad, fisuración bajo tensión, pruebas aceleradas.

## 1. INTRODUCTION

Stress cracking (SC) is an external or internal cracking in plastic induced by tensile stress less than its short-term mechanical strength [1]. Stress cracking occurs in a brittle manner with little or no elongation near to the crack surface [2].

Halse et al. [3] and Peggs and Carlson [4] claim that failure due to stress cracking is associated with defects or imperfections that cause the stresses to be enhanced to higher values with up to a 6-fold magnification of tensile stress (relative to the average global stress) depending on the geometry of the defect. The defects may be of various types and shapes and generally include surface scratches, grinding gouges, patches, and seams. In addition, the presence of an external chemical environment such as detergents, surfactants, leachate,

polar vapor, or liquid, may accelerate stress cracking. Stress cracking in the presence of chemicals is called "environmental stress cracking" [5, 6].

Polymers used in the fabrication of geosynthetics, such as polyethylene (PE), polyester (PET), unplasticized and plasticized poly vinyl chloride (PVC), are subjected to environmental stress cracking [7]. Among polyolefin polymers, polypropylene (PP) is less sensitive to SC than PE when it is associated with a very aggressive chemical environmental. The deformation limit that activates the phenomenon in PP is not yet well-known. The other polymer properties that affect susceptibility to stress cracking include the molecular weight and the co-monomer content [8, 9].

Higher molecular weight corresponds to longer chains [10], resulting in more tie molecules and more effective

tie molecule entanglements [11]. Similarly, high comonomer content and longer co-monomer short-chain branches provide better cracking resistance, most likely because portions of the long-branch chains cannot be folded into the lamellae and therefore contribute to the amorphous tie molecules [11, 12].

High density polyethylene (HDPE) is a widely used polymer for manufacturing geomembranes used in liquid and waste containment facilities and/or used as a part of liner systems in modern landfills [13]. The primary function of GM is to provide a barrier to advective and diffusive migration of contaminants [14]. The relatively high crystallinity (40 to 50%) of the material provides both high chemical resistance and low diffusion rates, which are required in most containment facilities [6, 15]. However, despite its good chemical resistance, one of the concerns raised in using HDPE geomembranes is their susceptibility to stress cracking (SC) which, in turn, is a consequence of their high crystallinity [2, 16, 6, 13, 15].

The evaluation of stress cracking is performed according to ASTM D5397 [17]. The test is called Notched Constant Tensile Load (NCTL) and uses notched dumbbell shaped specimens placed under various tensile stresses. The tensioned specimens (usually 20) are immersed in a bath containing 10% Igepal / 90% water solution at 50°C to accelerate the crack growth. A notch is introduced at the central constant-width section on the face of the specimen. The depth of the notch is such that the ligament thickness is equal to 80% of the nominal sheet thickness. The applied stresses typically range from 20% to 50% of the room temperature yield stress ( $\sigma_{vield}$ ) in increments of 5%. Three replicate specimens are tested at each stress level, and the failure time of each individual specimen is recorded to an accuracy of 0.1 hour. When a specimen fails, its failure time is recorded by a timer. The test data is presented by plotting the applied stress versus average failure time on a log-log scale. Unfortunately, the full test takes a long time to complete (generally over 10,000 hours). Thus, the Single Point Notched Constant Tensile Load (SP-NCTL) test was developed and is included in ASTM D5397 [17] as an appendix (to be used as a quality control or conformance test). The concept is to select a stress level near, but slightly lower than the transition stress, and to specify the minimum failure time at that stress. A single applied stress of 30% yield stress is utilized with a minimum failure time in GRI-GM13 [18] (Specification for HDPE Geomembranes). In 2003, the specification was revised by increasing the minimum failure time from 200 hours to 300 hours to further enhance the SCR of HDPE geomembranes [6, 19].

Rowe and Sangam [12] concluded that stress cracking is important because: (a) even short cracks can allow excessive leachate through the geomembrane that may readily move laterally in areas of poor contact between the geomembrane and the underlying clay; and (b) short cracks can grow with time eventually allowing excessive leakage through the geomembrane even in areas of good contact with the clay. In either case, once the leakage increases substantially, the geomembrane ceases to perform the barrier function for which it was designed as discussed by Rowe et al. [20].

Several investigators have reported the vulnerability of HDPE geomembranes to stress cracking: Fisher [21], Peggs and Carlson [4], Hsuan et al. [2], Hsuan [6], Rowe and Sangam [12] and Rowe et al. [15].

The transition from ductile-to-brittle failure requires the knowledge of stress level, stress concentration factor, temperature and surrounding environment. However, the fundamental governing factor is the polymer's characteristics, among which crystallinity and molecular weight are the most important. In this sense, it is important to evaluate the SCR of an HDPE GM to assess its long-term performance.

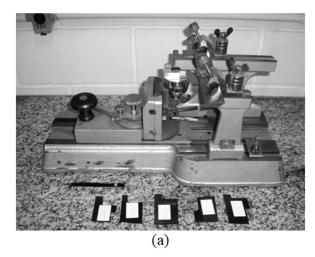
As previously mentioning the standard test is performed in a bath containing 10% Igepal / 90% water solution at 50°C to accelerate the crack growth. However, the effect of higher temperatures are not yet well know.

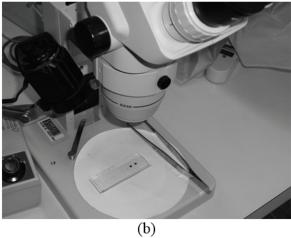
This paper presents results of SC tests performed in HDPE samples. The tests were conducted at 50°C (standard test) and at 70°C (accelerated condition) in order to verify the effect of the temperature and compare the SC values in both conditions. For this purpose, equipment was developed to process 20 specimens simultaneously. This equipment includes electronic acquisition of the failure times.

#### 2. MATERIAL AND METHODS

Smooth HDPE geomembranes of 2.0-mm nominal thickness were used. Tests of SC (NCTL and SP-NCTL) were conducted at 50°C and at 70°C (accelerated

condition) to compare the SC values according to ASTM D5397 [17]. Additionally, the dispersion of carbon black (Fig.1) was evaluated in accordance with ASTM D5596 [22] to verify the degradation of the material.

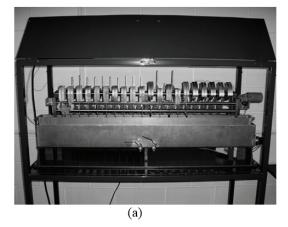




**Figure 1.** Preparation of the dispersion of carbon black test (a) cut specimens (b) microscopic evaluation.

## 2.1. Developed stress cracking test equipment

The equipment used in the SC tests was developed to process 20 specimens simultaneously. This equipment includes electronic acquisition of the failure times (Fig. 2). Force is applied to the specimen by a lever with metallic weights in its extremity. This lever applies a force equal to three times the load. The incubation system allows the mechanical control of temperature.



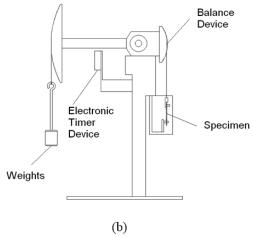
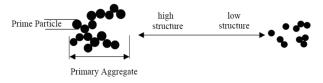


Figure 2. Test Setup

## 3. RESULTS AND DISCUSSION

## 3.1. Carbon black dispersion

Carbon black is the most common type of UV protection for polymeric products, and it consists of very fine particles (the primary particles) fused together to form the primary aggregates (Fig. 3). The UV absorbing efficiency of carbon black is governed by the average prime particle size. Primary aggregates composed of finer prime particles present a greater surface area for optical absorption than the primary aggregates composed of larger prime particles.



**Figure 3.** Schematic drawing of carbon black particles [23]

Thus, UV absorption increases as prime particle size decreases. However, with prime particles below 20 nm, the UV stabilizing efficiency tends to level off as light scattering becomes more significant with a further decrease in particle size. The carbon black particle size used for the UV protection of polymers used for geosynthetics is typically in the range of 22–25 nm [24, 23, 19]. The results obtained show that all specimens belong to the category I, which contains carbon black particles of circular geometries with diameters less than

or equal to 35  $\mu m$ . In this research, the results of carbon black tests were not efficient in detecting degradation in the samples.

## 3.2. Accelerated NCTL and SP-NCTL tests

Fig. 4 presents the NCTL curves and the failure times of the SP-NCTL tests. Table 1 and 2 presents the parameters of the NCTL tests and the SP-NCTL results concerning the samples at 50°C and 70°C, respectively.

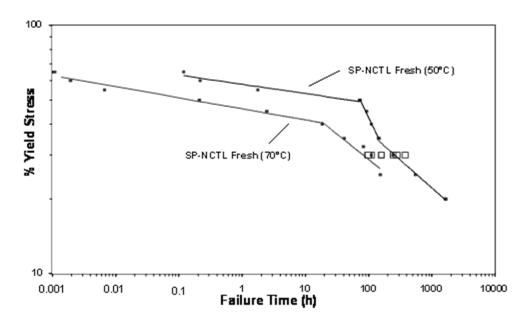


Figure 4. Results of NCTL tests at 50°C and 70°C and failure times (SP-NCTL)

**Table 1.** Parameters of the NCTL tests

Sample	Ductile Slope (%/h)	Brittle Slope (%/h)	$T_{t}(h)$	σ <sub>t</sub> (%)
HDPE (50°C)	-0.038	-0.23	143	35
HDPE (70°C)	-0.045	-0.20	18	40

 $T_t = \text{Transition time}; \ \sigma_t = \text{stress transition}$ 

**Table 2.** Average failure times (T<sub>s</sub>) of the SP-NCTL tests

Sample	$T_f(h)$	CV (%)	Variation (%)		
HDPE (50°C)	243	38	-		
HDPE (70°C)	101	6	58 (decrease)		
CV = another interference					

CV = coefficient of variation

The accelerated test (70°C) presented a bi-linear curve (different than the test at 50°C). The highest loading stages at 70°C presented 6 stages of loading in the ductile region and only 4 stages of loading at 50°C. The slope of the straight ductile accelerated test showed a slight increase when compared to the sample at 50°C.

The transition time ( $T_i$ ) obtained in the accelerated test presented an 87% decrease when compared to the test at 50°C and had a higher stress transition ( $\sigma_i$ ). The brittle region of the curve for the accelerated test presented 5 stages of loading and 4 stages for the test at 50°C. The slope of the accelerated tests decreased

when compared to the tests at 50°C due mainly to the loading stage of 32.5%  $\sigma_{vield}$ .

Accelerated test results have shown time savings when tests with high temperature are used: a total economy of 390 h, comparing load stages of 25%  $\sigma_{\text{yield}}$ . However, the behavior of the sample under high temperature was completely different when it was compared to the test at 50°C (the type of curve and in the stress transition,  $\sigma_{\text{i}}$ , obtained).

The increase in the incubation temperature (70°C) decreased the average failure times by 58%. Accelerated test results provided more homogeneous results, resulting in lower coefficients of variation as well.

Regarding the accelerated tests, the average time to failure (SP-NCTL) was close to the failure time of the NCTL (30%).

The results show that the sample maintains the same trend verified in the NCTL tests. There was a total economy of 265 h compared to the standard test (50°C).

## 4. CONCLUSION

From the data presented in this paper the following conclusions can be drawn:

- The SC equipment developed to register the failure times worked well producing precise failure times;
- The carbon black tests were not efficient to detect degradation in the GM samples;
- Results from accelerated tests (NCTL) showed time savings (390 h less compared to a load stage of 25% σ<sub>yield</sub>). However, the behavior of the GMs under high temperatures was completely different when compared to the standard test at 50°C. This fact is discussed and evaluated by Hsuan and Koerner [25] who say the transition time changes in a systematic manner with changes in test temperature. The recommended test temperature for performing the control NCTL test (and the associated SP-NCTL test) will be material dependent and must be decided upon accordingly.

 Results from accelerated SP-NCTL tests showed a total economy of 265 h (compared to the standard test at 50°C), and the same trend was verified in the NCTL tests.

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