### Long-term performance of the "Laguna de Barlovento" reservoir waterproofing using a PVC-P geomembrane Blanco, M., Leiro, A., Soriano, J.,

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ABSTRACT. The "Laguna de Barlovento" reservoir was one of the most important European hydraulic projects at the time of its construction because of its high capacity and challenging location. At the time, the designers decided to waterproof this reservoir with a geomembrane of plasticized polyvinyl chloride (PVC-P). This paper documents the initial characteristics of the geomembrane and its performance since its installation until 2010. The material characterization includes a comprehensive testing program, the results of which are presented. They include quantification of the geomembrane thickness, amount and nature of plasticizers, tensile properties, foldability under low temperatures, dynamic impact resistance, puncture resistance, welding strength (both in the manufacturing facility and in the field), as well as the use of techniques involving optical and scanning electron microscopy. In addition, advanced analytical techniques, such as Fourier Transform Infrared Spectroscopy (FTIR), Gas Chromatography (GC) and Mass Spectrometry (MS), were used in order to identify the plasticizers used in the geomembrane formulation. Foldability tests were found to provide early indication of degradation. Results from reflection optical and electron scanning microscopy showed that, after 19 nineteen years of installation, the geomembrane remains in good conditions, particularly on the non-exposed side.

### 1 INTRODUCTION

In Spain, Plasticized polyvinyl chloride (PVC-P) geomembranes began being used in waterproofing of infrastructure in the seventies. Early usage of PVC-P geomembranes was not particularly successful, because their contact with the bituminous sheets, usually employed in construction, led to rapid deterioration. Towards the end of that decade, the former Spanish official standardization body, IRANOR, created a working group within the Plastics and Rubbers Committee 53 to develop a standard for the PVC-P homogeneous geomembranes used in roofing. Subsequently, other standards were drafted for the same material but reinforced with fiberglass or fabrics. Ultimately, the use of PVC-P geomembranes would be extended to Civil Engineering projects.

The existence of standards for PVC-P that clearly stated the characteristics to be determined and the experimental methodology led to the gradual substitution of butyl rubber sheets, which had been used in most hydraulic projects at that time, by PVC-P geomembranes. The use of PVC-P benefited from its good resistance against ozone degradation. The ozone degradation of butyl rubber geomembranes was a major problem in reser-

voir projects. Indeed, ozone degradation was such a concern that for a number of years, laboratories were still being asked to conduct ozone resistance tests on PVC-P geomembranes. In the Spanish Peninsula, the use of PVC-P geomembranes began in the Levant, spreading rapidly throughout Spain (Blanco et al. 1998, 2009a).

The Hydrological Plan implemented in the eighties by the Autonomous Community of the Canary Islands initially involved the construction of nine reservoirs in the island of Tenerife. PVC-P geomembranes supplied by a number of manufacturers were used to waterproof all reservoirs (Amigo & Aguiar 1994, Aguiar & Blanco 1995, Blanco & Aguiar 1993, 2000, Blanco et al. 1995, 1996a). Many of these reservoirs are still in service. The Water Council of La Palma, another island in the Canary Archipelago, also conducted an extensive plan involving the construction of reservoirs. Many of them were waterproofed with PVC-P, like San Mauro, Montaña del Arco, Adeyahamen, Bediesta, Las Lomadas, Manuel Remón, El Vicario and La Laguna de Barlovento.

This paper shows the long-term performance of the PVC-P geomembrane installed in one of these reservoirs, the "Laguna de Barlovento" reservoir (Fig. 1).

This paper documents the initial characteristics of the geomembrane, the geomembrane performance

since its installation until 2010, as well as monitoring tests results. Tests conducted on PVC-P samples at the Laboratorio Central de Estructuras y Materiales (CEDEX) include the following: Thickness, amount and nature of plasticizers, tensile properties (tensile strength and elongation at maximum load), foldability under low temperatures, mechanical resistance to percussion (dynamic impact resistance), puncture resistance (static impact resistance), welding strength (conducted both at the manufacturing facility and in the field) and welding peeling resistance. In addition, optical and scanning electron microscopy techniques were used. Analytical techniques such as Fourier Transform Infrared Spectroscopy (FTIR), Gas Chromatography (GC) and Mass Spectrometry (MS) were also conducted for plasticizer evaluation.

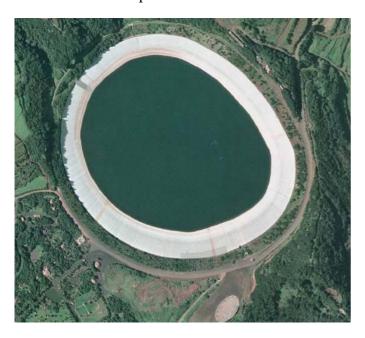


Figure 1. Aerial view of the "Laguna de Barlovento" reservoir, waterproofed with PVC-P geomembrane.

### 2 POLIVINYL CHLORIDE

While the writers have investigated a number of materials used in geomembranes (Aguiar et al. 2008, Blanco et al. 2005a, Leiro et al. 2002), significant emphasis has been placed on PVC-P because of its widespread use in Hydraulic projects across Spain over the last two decades.

Polyvinyl chloride is a macromolecule with a high tendency to have dehydrochlorination and become a conjugated diene, the structure of which is highly stable. This behaviour differentiates the PVC-P from other polymers also used as geomembrane such as the chlorinated polyethylene (CPE). In CPE, a possible dehydrochlorination would result in an isolated diene structure, which is highly unstable. A number of factors affect the instability of PVC (Castillo & Blanco 1991).

Polyvinyl chloride is originally a rigid material, being used as such in several industries ranging from the automotive to the building industry. In this physical state, the Polyvinyl chloride cannot be used in waterproofing projects. Accordingly, it should be transformed into a flexible material, i.e., it should be plasticized.

PVC plasticization can be internal or external (Wilson 1995). Internal plasticization involves attachment of the plasticizer molecules to the polymer resin by primary bonds, becoming incorporated to the polymer chain. Instead, external plasticization involves absorption or adsorption of the small molecules used as additive onto the pores of the polymer. External plasticization is the most common and cost-effective approach. In general, plasticizers involve liquid esters of high molecular weight that increase the deformability of the material and reduce its tensile strength and glass transition point.

The main problem with external plasticization is the tendency of the plasticizer to migrate. Plasticizer migration can be to air, liquid or to a solid in contact with it. The migration mechanism to air includes diffusion of the plasticizer from inside the geomembrane to its surface, and the subsequent evaporation from geomembrane surface (Sears & Darby 1982). The extraction by a liquid (e.g. water), requires that the fluid gets in contact with or enters the pores of the macromolecule, subsequently dissolves the plasticizers and finally leaches the dissolved plasticizers out to the geomembrane surface (Nass et al. 1986). Also, plasticizer loss can occur because of the contact of the geomembrane to any adjacent absorbent material with which the plasticizer has affinity. It has been verified that repeated waterproofing or contact between multiple PVC geomembranes may lead to migration between the different geomembranes (Papakonstantinou & Papaspyrides 1994, Blanco et al. 2005b).

Plasticizer loss depends on the type of ester used as additive. More specifically, it depends on its molecular weight, linearity and polarity. Considering that almost all plasticizers are polar, the loss can then be attributed to essentially one of only two factors. A plasticizer with comparatively higher molecular weight reduces the compatibility and efficiency of the plasticization processes; however, the higher the plasticizer molecular weight, the lower the migration tendency. This is because the additive diffusion into the pores of the macromolecule will be decreased by steric hindrance. Consequently, a compromise solution is needed, and the plasticizer molecular weight is recommended to exceed 400. The linearity or branches of the plasticizer is related to the shape of a plasticizer molecular structure. A plasticizer composed of straight chains is referred to as a 'lin-

ear plasticizer.' On the other hand, a plasticizer comprised of branched chains in the molecular structure is referred to as a 'branched plasticizer.' An increasing branching index of the plasticizer results in an increasing vapor pressure, which facilitates the plasticizer migration into air. However, an increasing plasticizer branching index also minimizes the plasticizer migration into liquids and solids (Stark et al. 2005).

### 3 THE RESERVOIR

The "Laguna de Barlovento" reservoir was built between 1971 and 1975, although the original waterproofing only involved a layer of compacted clay. In spite of early analyses based on laboratory test results on clay samples, leaks were detected soon after the reservoir was in service. The leaks were attributed to the volcanic nature of these clays, which included basaltic interlayers. It was then decided to waterproof the reservoir using a PVC-P geomembrane reinforced with a fabric scrim (Fayoux 2004). The geomembrane was installed in 1991, although the east portion of the reservoir waterproofed in 1992 because of the weather conditions. It should be noted that the geomembrane placed in the eastern portion of the reservoir was manufactured at a different time than the rest of the project.

The reservoir of the "Laguna de Barlovento" is located in the northeast of the La Palma Island, in the area known as "Las Cabezadas," in the municipality of Barlovento (Fig. 2). Their characteristics of the reservoir are summarized in Table 1, which includes capacity, inclination of the sideslopes, and perimeter at the crest. The reservoir location as well as the type, thickness, and quantity of geomembrane used in this project.

Table 1. Characteristics of La Laguna de Barlovento.

Location	Las Cabezadas
	(Barlovento)
Capacity, hm <sup>3</sup>	3.1
Height, m	22
Crest level, m	739
Crest perimeter, m	1,593
Slopes	2.75:1
Type of geomembrane	PVC-P
Thickness, mm	1.50
Quantity, m <sup>2</sup>	170,000
Year of installation	1991

The bottom of the reservoir was also water-proofed with 80,000 m<sup>2</sup> of PVC-P geomembrane. However, the geomembrane used in this location was unreinforced, that is, without scrim. Because this unreinforced geomembrane always remained covered with water, samples of this portion of the

reservoir were only taken at the installation time and when the reservoir had to be emptied to conduct repairs. Accordingly, a comparatively smaller volume of data were collected on the unreinforced geomembrane, which is not presented in this paper.

### 4 EXPERIMENTAL TEST RESULTS

The geomembrane used in this project was initially characterized in order to assess the conformance with the product specifications and to get initial values to be used as basis for comparison of the subsequent long-term test results. For this purpose, samples were collected periodically in different zones of the reservoirs and tests were repeated regularly to check the evolution of the various geomembrane properties.

The experimental methodology used in this research followed that developed by the European Standard EN 13361. Puncture resistance tests conducted in this study were originally developed by this research team, and now constitute the norm UNE 104 317. This standard was also adopted by the Spanish organization of normalization and certification (Standardization Regulations) AENOR (Blanco et al. 1996b).

Tensile tests, including the determination of tensile strength and elongation at break, were conducted in both the longitudinal and transverse directions, the results presented in this brief paper are only those obtained in the longitudinal direction. Also, dynamic impact tests were performed on both surfaces of the specimens, but only the values obtained on the exposed geomembrane surface will be presented in this paper. Finally, unless otherwise stated, the tests results presented herein correspond to samples collected from the north slope of the crest area. This area receives the highest incidence of ultraviolet radiation.



Figure 2. View of the geomembrane-lined reservoir.

All tests conducted using the original materials met the minimum requirements for this type of geomembrane, according to the standard specifications at that time. Those specifications would also comply with the current Reservoir Manual (2010), written by CEDEX for the Spanish Ministry of Environment and Rural and Marine Affairs.

### 4.1 Foldability at low temperature

Specimens of the geomembrane from the reservoir were subjected to folding at low temperatures. After spending 5 hours in a cold room at a temperature of -20 ° C, the specimens were folded in an angle 180°, and were inspected to identify any evidences of cracks, breaks or other surface imperfections.

Specimens collected from the crest area of the southern slope did not present any deterioration after 19 years of installation. On the other hand, specimens collected from the northern, eastern and western areas did not pass the tests. Specifically, half of the specimens showed cracks during the tests conducted 17 years after the installation. This behaviour highlights the importance of quantifying the effect of UV radiation in the type of thermoplastic geomembrane used in this project (Aguiar et al. 2002, 2003, Blanco et al. 2003).

# 4.2 *Mechanical resistance to percussion* (dynamic impact resistance)

The mechanical resistance to percussion or dynamic impact resistance was adequate according to the test results conducted with all the specimens collected in this study. This includes both initial samples as well as those collected throughout the 19 years following installation. Consistently good results were obtained regardless of the place in the reservoir where samples were collected from. The test involves dropping from a height of 500 mm a 0.5 kg plunger ending in a semi-spherical shape and a diameter of 12.7 mm. Impact zone of the geomembrane did not show holes, as was subsequently verified by a watertight test.

### 4.3 Tensile characteristics

Tensile strength results and elongation at maximum load values are showed in Figures 3 and 4 respectively. Neither the elongation at break nor the tensile strength results did present significant changes over the 19 years of service. Exposure to weathering only led to a slight increase in tensile strength values, as shown in Fig. 3. The mechanical response of the reinforced geomembrane is dominated by the fabric scrim. The good mechanical response is attributed to the good response to degradation of the resin of the geomembrane. Ac-

cordingly, since such degradation did not lead to exposure of the reinforcing polyester yarns, the mechanical benefits provided by the reinforcing scrim have not been compromised after the 19 years of service.

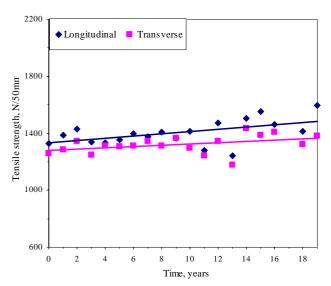


Figure 3. Tensile strength evolution over time.

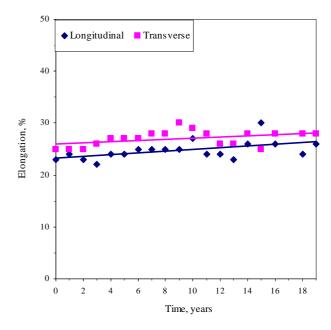


Figure 4. Elongation at maximum load evolution over time.

# 4.4 Puncture resistance (static impact resistance)

The results of puncture resistance tests are presented in Figures 5 and 6, which show the variation of resistance to static puncture and the displacement of the plunger before perforation, respectively. Puncture resistance values are observed to increase over time, while displacement of the plunger before perforation values decrease. These displacement values are comparatively small because of the presence of the Polyester reinforcement.

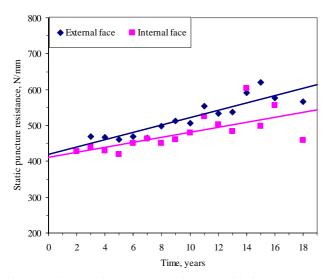


Figure 5. Changes in puncture resistance with time.

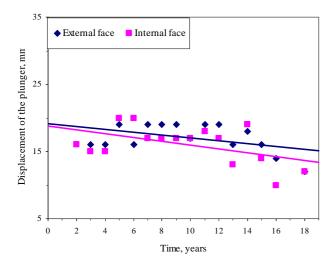


Figure 6. Changes in the measured displacement of the plunger (puncture tests) with time.

### 4.5 Welding strength

Due to the large size of the reservoir, some of the geomembrane sheets were welded in the manufacturing facility, before being carried to the site. Accordingly, two types of seams were conducted in this project: those made in the controlled environment of a manufacturing facility and those made during installation on site. The welding strength tests are considered adequate, as all the specimens broke in the edge or close to the seam, but never through the seam itself.

Test results are presented in Figures 7 and 8, which show similar trends for the seams made in the manufacturing facility and those made on site. These results indicate that the quality of the geomembrane installation was good. Figure 9 shows the test results obtained when testing the seams following the peeling procedure. The test results are comparatively high, also verifying the good execution of the seams.

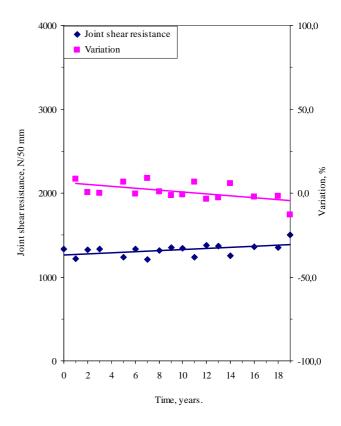


Figure 7. Changes in welding strength with time (weldings conducted on site).

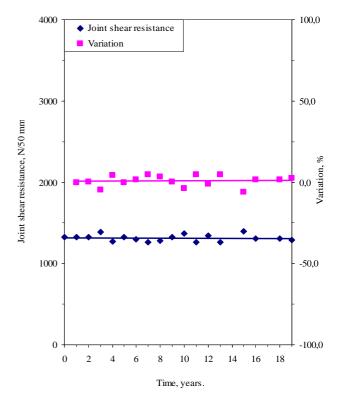


Figure 8. Changes in welding strength with time (weldings conducted in the manufacturing facility).

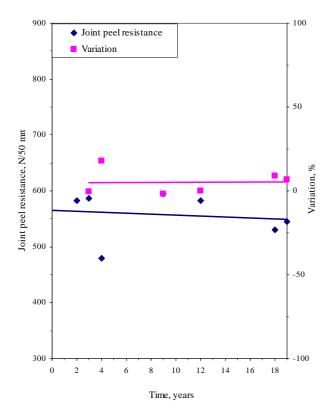


Figure 9. Changes in peeling strength with time of the weldings conducted on site.

# 4.6 Reflection optical microscopy and scanning electron microscopy.

Evaluation of the long-term performance of the geomembrane was also made using optical and electron scanning microscopy (Soriano et al. 2006, 2010). Microphotographs were taken by reflection optical microscopy (MOR) with 40x and 60x magnifications in order to visualize the geomembrane texture and morphology. Also, the geomembrane was evaluated using scanning electron microscopy (SEM) and microphotographs were obtained with 90x and 900x magnifications.

Optical microscopy results revealed a comparatively high density of superficial cracking on the exposed side of the geomembrane. On the other hand, good condition was consistently observed on the non-exposed side of the geomembrane. The results obtained using scanning electron microscopy confirmed these observations.

Figure 10 shows the exposed and non-exposed sides of the geomembrane using 60 x magnification, as obtained by reflection optical microscopy. Figure 11 also shows the two sides of the geomembrane using 90 x magnification, as obtained by scanning electron microscopy. The images shown in these figures were obtained using specimens collected 19 years after the geomembrane installation.

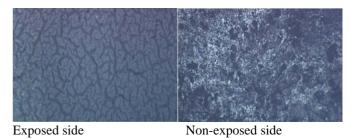


Figure 10. Microphotographs of the PVC-P geomembrane obtained using ROM (x 60): (a) Exposed side; (b) Non-exposed side.

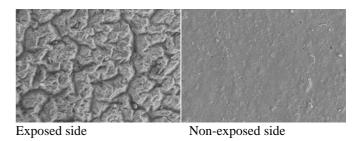


Figure 11. Microphotographs of the PVC-P geomembrane, obtained using SEM (x 90): (a) Exposed side; (b) Non-exposed side.

### 4.7 Plasticizers

The plasticizer content in the original geomembrane was determined to be 35.1%. Figure 12 shows the changes in plasticizer content (and quantification of its loss) with time. The results are presented in relation to the original value, over 19 years of the geomembrane. The results indicate a plasticizer loss of less than 40% over this period, which is considered an excellent performance for an exposed geomembrane.

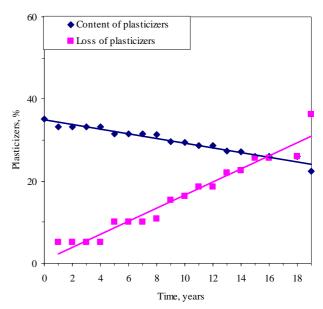


Figure 12. Content and loss of plasticizers evolution over time.

## 4.7.1 *Influence of the sample location on the test results*

Results of plasticizer content obtained over a period of 15 years after the geomembrane installation are presented in Table 2. The results are shown for four different locations in the reservoir, so the test results allow assessment of the orientation and location of the samples in the reservoir. The greatest plasticizer loss was measured in the crest area of the reservoir, then in the intermediate area and, finally, in the submerged area of the reservoir. The results also indicate that the area least affected by solar radiation was the southern portion of the reservoir. It should be noted that the eastern portion of the reservoir was waterproofed a year after than the rest of this facility. While the geomembrane used in the eastern portion of the reservoir should have had the exact same formulation as that used previously, test results indicate some differences. Specifically, the initial plasticizer content of the geomembrane used in the eastern portion of the reservoir was not the same. Accordingly, the response of this geomembrane was found to be slightly different than that observed in the rest of the project.

Table 2. Plasticizers content according to the orientation and sampling area.

TALUD	Zone		
	Crest	Intermediate	Submerged
North	26.1	27.2	28.1
South	27.4	27.6	27.9
East*	24.9	26.6	27.7
West	26.0	26.5	27.5

<sup>\*</sup>Data at 14 years

The plasticizer loss results were fitted to a straight line, as shown in Figure 12. The trend is represented by the following equation:

$$y = ax + b$$

where b is the intercept, that is, the initial plasticizer content and a represents the rate of plasticizer loss with time. Accordingly, a value of a equal to 0 (i.e. y = b) would correspond to no loss of additive. Thus, the smaller the value of a (i.e. the closer to 0), the lower the loss of plasticizers. The magnitude of this parameter allows making comparisons between results obtained from samples collected from different locations of the same reservoir. Table 3 showed the information obtained using samples collected over fifteen years after its installation. The letters (N, S) in Table 3 refer to the location of the collected samples (north, south), while the numbers in Table 3 (1, 2, 3) correspond to the areas where the samples were collected

(crest, intermediate height, and near the water). Inspection of this data indicates that the plasticizer loss of samples collected the "Laguna de Barlovento" reservoir improves towards the bottom of the facility.

Table 3. Values of the slope on the loss of plasticizers 15 years after the installation.

Reference	Square regression coefficient	Trend	
		а	В
N1	0.9711	-0.0449	34.595
N2	1.0000	-0.0439	35.100
N3	0.8128	-0.0314	35.204
S1	0.9658	-0.0421	34.491
S2	1.0000	-0.0417	35.100
S3	0.9269	-0.0351	35.168

As indicated by the values of parameter a (rate of plasticizer loss), the best response corresponds to samples located in submerged areas, while the worst case is that of samples collected from areas continuous exposed to the elements. The best preserved area is the southern portion of the reservoir.

### 4.7.2 *Identification of Plasticizer Type*

The plasticizer used in the geomembrane formulation was determined by extraction with ethyl ether, following the procedure described by Crespo (2011).

Once the plasticizer was isolated, it was analyzed using Fourier Transform Infrared Spectroscopy (FTIS) with a Nicolet 310 FTIR. The infrared spectrum was obtained, where the characteristic bands of alkyl phthalates can be clearly observed (Blanco et al. 2008, 2009b, 2010).

Identification of the additive was conducted using the Gas Chromatography technique combined with Mass Spectrometry technique (GC-MS). A gas chromatograph GC Agilent 6890N was used along with a capillary column of phenyl methylpolysiloxane DB%-MS (30 m x 0.25 x 0.25). The process was coupled to a quadrupole mass detection system Agilent 5793 MSD in electron impact mode. The first technique allows determination and separation of a number of products that constitute the plasticizer. The second technique allows identification of the actual products.

The gas chromatography technique generated a chromatogram with a single peak. This product was analyzed by Mass Spectrometry whose spectrum presented the following data: MS, m/e (relative intensity):  $446(M^+)$ ,  $307(C_{18}O_4H_{27}^+,25)$ ,  $167(C_8H_7O_4^+,19)$ ,  $149(C_8H_5O_3^+,100)$ ,  $85(C_6H_{13}^+,25)$ ,  $57(C_4H_9^+,29)$ . These fragmentations indicate that the plasticizer is a Diisodecyl phthalate.

The Diisodecyl phthalate has a molecular weight of 446, which is well above the molecular weight of 400 that is the recommended minimum value (as reported by the PVC Geomembrane Institute). Also, the protons of phthalic acid are replaced by ramified alky radicals, leading to a plasticizer migration that is greater into air than into water. Nonetheless, the radical includes ten Carbon atoms, a comparatively high length that hinders this process. These characteristics of the plasticizer used at the site are consistent with the observed good performance of the geomembrane over time.

### 5 CONCLUSIONS

The PVC-P geomembrane installed in the "Laguna de Barlovento" reservoir was monitoring during 19 years, involved collecting a significant number of samples during this time, and resulted in a significant database of long-term performance test results. These test results lead to the following conclusions:

Foldability tests under low temperature conducted on specimens collected in the southern area of the reservoir did not show cracks in the flexion zone after 19 years. On the other hand, tests conducted on specimens collected in others areas of the reservoir started to show cracks after 17 years of installation. Foldability results are considered to be particularly relevant for PVC-P geomembranes because they identified the onset of the degradation process before other geomembrane characteristics. After specimens start showing cracks after folding, continued monitoring is recommended during the following years in order to establish when it becomes necessary to replace the geomembrane.

Tensile characteristics did not present significant changes over time because the fabric used as reinforcement dominated the mechanical response, including tensile strength and the elongation at maximum load. The resin was found to protect the fabric from UV radiation, which minimized its degradation and preserved the mechanical properties of the geomembrane. The resin was found to be in good condition throughout the entire duration of this project.

The mechanical resistance to dynamic impact over time was found to be adequate due to the polyester reinforcement of the geomembrane. The resistance to puncture was found to increase with time and displacement of the plunger before perforation decreased, also as a result of the fabric reinforcement.

Welding shear resistance tests, conducted both during installation and in the manufacturing facility, were found to meet specifications. This is because breaks in all specimens were found to happen near the seams, but always outside of the seam itself. No differences were found between the two types of welding conducted at this project, which indicated that good installation procedures were followed. Shear resistance test results were deemed qualitative while the peeling tests results provided more quantitative assessment. Peeling test results led to comparatively high values in the PVC-P geomembrane, confirming an excellent installation of the material.

Results from reflection optical microscopy as well as scanning electron microscopy showed, after 19 nineteen years of installed, noticeable cracking on the exposed side of the geomembrane samples while the non-exposed side remained in good conditions.

The plasticizer of the geomembrane was identified as being disodecyl phthalate, the molecular weight of which is 446. This is a significantly higher value than that required for the geomembrane. Nevertheless, being an ester with ramified alkyl radicals this plasticizer tends to migrate into air. However, this process was minimized by the comparatively long hydrocarbon chains.

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