

# Durability of reinforced PVC-P geomembranes installed in reservoirs in eastern Spain

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**ABSTRACT:** The aim of this paper is to study the durability of polyvinyl chloride (PVC-P) geomembranes reinforced with a synthetic fabric in hydraulic works in the Spanish Mediterranean basin. Therefore, a set of six geomembranes installed in irrigation reservoirs for 18–31 years were analysed. The initial characteristics of the geomembranes were determined to verify fulfilment of the Spanish regulations in force at the time. The characteristics were then assessed, and the results were interpreted with reference to the loss of plasticisers, tensile characteristics, foldability at low temperatures, dynamic impact resistance, puncture resistance, seam strength, reflected optical microscopy (ROM) and scanning electron microscopy (SEM). Additionally, the identification of the plasticisers in the geomembranes involved Fourier transform infrared spectroscopy (FTIR), gas chromatography (GC) and mass spectrometry (MS) tests. For the analysed samples, the loss of plasticisers was significant, ranging from 71.0% to 84.3%. However, the tensile strength results indicated current, regular waterproof working performances in the reservoirs. The results suggest that the durability of PVC-P geomembranes is a function of the loss of plasticisers and the state of the synthetic reinforced fibres.

**KEYWORDS:** Geosynthetics, Geomembrane, PVC-P, Waterproofing, Water reservoir, Durability

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## 1. INTRODUCTION

The use of synthetic geomembranes in hydraulic works began in the 1940s, and development increased in the 1960s and 1970s. Butyl rubber (IIR) geomembranes were used for waterproofing in the 1930s, and polyvinyl chloride linings started to appear in the 1940s. There were some problems with the first generation of polymer geomembranes, such as the minimal resistance of IIR to ozone and the brittleness and propensity to cracking

of PVC-P, which mainly occurs when used in contact with bituminous materials (Noval 2015a).

The first documented application of a polymer geomembrane was for waterproofing the Contrada-Sabetta dam in Italy (Cazzuffi 1987; Cazzuffi *et al.* 2010). The geomembrane that was used was polyisobutylene (PIB), and it was applied in 1959. In its Bulletin No 78, the International Commission on Large Dams (ICOLD 1991) cited the Kualapuu Reservoir in Molokai (Hawaii) as the first reservoir to be

waterproofed using an IIR synthetic geomembrane in 1969.

The use of synthetic geomembranes in hydraulic works first arrived in Spain in the 1970s in the Southeast of the peninsula, specifically in the Ibi district of Alicante province, where IIR-based elastomeric barriers were installed (Blanco 2005). Previously, the reservoirs had used liquid waterproofing systems, for example, at El Saltadero. Additionally, low-density polyethylene (LDPE) films were applied in buried systems, and some have survived perfectly, fulfilling their function to the present day. This is the case in the El Fraile reservoir (south of Tenerife island) and Plá Mateos (Alicante). Before this, albeit uncommonly, bituminous geomembranes modified with rubber were used in some dams in the Pyrenees, such as the Aiguamoix dam in Lerida. In 1968, the Odiel Perejil dam in Campofrío (Huelva province) was built using a thermoplastic chlorinated polyethylene (CPE) geomembrane (ICOLD 2010). In 2002, a survey was carried out to establish the performance of geomembranes over time, and the results showed excellent preservation of the membranes (Blanco and Zaragoza 2003). In 1974, the Matavacas irrigation weir (Sanlúcar de Guadiana-Huelva) was waterproofed with an IIR synthetic geomembrane, which is still in good condition apart from the joints between the sheets (de Cea *et al.* 2003; Blanco *et al.* 2010).

PVC-P is considered the successor to IIR, and it is resistant to ozone. It was initially introduced as a synthetic thread reinforcement (Crespo 2011) and then as a homogeneous material (Blanco *et al.* 2013a) or fibreglass insertion (Blanco *et al.* 2016a). This material was widely supported for use in Mediterranean countries, for example, Italy (Cazzuffi 2013).

Consequently, high-density polyethylene (HDPE) geomembranes began to be used in some waterproofed reservoirs in Castilla-León and the Canary Islands (Noval *et al.* 2014a, 2014b).

Next, a move into the field of elastomers began with the appearance of ethylene propylene diene monomer rubber (EPDM), which contains a minimum number of double bonds in its side chain and is not attacked by atmospheric ozone (Noval *et al.* 2014c, 2015; Blanco *et al.* 2015).

In Spain, the installation of PVC-P geomembranes was highest in the last two decades of the past century. The most widespread areas of use were in the southeast of the peninsula and the Canary Islands because of the large quantity of agricultural irrigation reservoirs (Amigó and Aguiar 1994; Aguiar and Blanco 1995; Blanco *et al.* 2003a, 2012).

Nevertheless, PVC-P geomembrane may have some disadvantages, because the material can occasionally deteriorate relatively quickly due to the loss of plasticisers. In building construction applications, the installation of a PVC-P lining sheet over a bituminous geomembrane leads to accelerated depletion because of the interactions between the bitumen components (maltenes, asphaltenes, oils) and those of the geomembrane (resins and additives).

By the 1980s, PVC-P was widely used in the region of Valencia, and some of those reservoirs still have the same

waterproofing system currently (Blanco *et al.* 2016b). In other cases, the reservoirs were re-waterproofed using a different procedure (Méndez *et al.* 2008).

In this paper, we analyse the behaviour over time of six PVC-P geomembranes reinforced with polyester fabric. Their ages range from 18 to 31 years, and they were all installed in irrigation reservoirs located in the Alicante province (southeast Spain). The survey was carried out in the following reservoirs: El Hondón de las Nieves, El Rollo, Plá Aceituna, La Caseta de Mira, El Cid-III and El Rabosero. The aim of this paper is to demonstrate that a PVC-P geomembrane reinforced with synthetic thread fabric remains effective despite the fact that its loss of plasticisers is greater than 50%. Therefore, based on experience with exposed PVC liners in Spain installed according to European practice, their durability is not only dependent on the loss of plasticisers but also on the condition of the textile reinforcement.

## 2. THE RESERVOIRS

### 2.1. Location

The studied reservoirs are El Hondón de las Nieves, El Rollo, Plá Aceituna, La Caseta de Mira, El Cid-III and El Rabosero. All are located in Alicante province (southeast Spain) (Supplemental Graphical Data). All these reservoirs have been waterproofed with a PVC-P geomembrane reinforced with a synthetic thread fabric. The installation of the geomembranes ranges from 1984 (Cid III) to 1997 (Caseta de Mira). The technical characteristics of the reservoirs are shown in Table 1 and include the location, capacity, elevation, height, crest perimeter and date of installation.

### 2.2. Climate

The exposure conditions of all the geomembranes are very similar, since all of them were installed on uncovered reservoirs. Additionally, their site locations share similar semi-arid climatic conditions characterized by high annual solar radiation (approximately 2700 h of sunlight per year) and severe summer drought. The total annual precipitation is less than 400 mm, which comes mainly in the autumn and the spring, and shows an irregular rainfall distribution out of sync with the times of maximum irrigation needs.

The maximum ultraviolet index for clear weather in Alicante is between 4.5 and 6.5, which is relatively significant in terms of the scale of 0.5 (low UV) to 14.5 (extreme UV) (UNEP 2007).

### 2.3. PVC-P geomembrane

Reservoirs were lined with a 1.50 mm thick PVC-P geomembrane from VICON plastic manufacturing using the 'impregnation' process. Although the original composition of the geomembranes is unknown, a typical composition is 50–70% PVC resin, 25–35% plasticisers and 2–5% other additives, which include UV light absorbers such as carbon black, pigments such as titanium dioxide, stabilizers such as calcium stearate, and fillers

**Table 1. Characteristics of the reservoirs waterproofed with PVC-P**

Reservoir	Hondón de las Nieves	El Rollo	Plá Aceituna	Caseta de Mira	Cid-III	El Rabosero
Location	Hondón de las Nieves	Aspe	Agost	Agost	Monforte del Cid	Aspe
Capacity (m <sup>3</sup> )	1 213 280	536 000	118 132	269 717	531 810	240 772
Elevation (m)	19.50	16.00	8.25	9.00	12.0	12.80
Height of crest (m)	464	399	388	419	404	260
Crest perimeter (m)	1068	834	535	780	950	691
• Slope gradient						
• Exterior	1.50:1	1.50:1	1.30:1	1.75:1	1.50:1	1.50:1
• Interior	2.50:1	1.50:1	2.50:1	2.00:1	2.50:1	3.00:1
Geomembrane installation year	1990	1987	1994	1997	1984	1994

such as calcium carbonate (Koerner *et al.* 2005; Blanco 2015).

### 3. EXPERIMENTAL

The experimental method followed the guidelines of European regulation EN 13 361 (AENOR 2013a). All samples were extracted from the northern slopes of the reservoirs in the crest area, which faces south and receives the most solar radiation (Aguar *et al.* 2003; Blanco *et al.* 2003b; Noval *et al.* 2014d). In the case of the Hondón de las Nieves reservoir, a sample was also taken from the intermediate area on the northern slope, which is sometimes covered by water and sometimes in contact with solar radiation. The exhumed samples represent the worst-case scenario, since most of the installed geomembranes perform better under less solar radiation.

First, the original characteristics of all the lining systems were determined. These values are shown in Table 2. It should be noted that the previously existing regulations included tests that have currently fallen out of use, for example, water behaviour and plasticiser migration.

The water behaviour was tested in accordance with regulation UNE 53 028, method B (AENOR 1990), which has since been modified. The test is used to assess the absorption of water by the geomembrane and the extraction of additives by the water; the results for both the absorption and extraction were obtained over 1 to 6 days. The water behaviour fundamentally affected the dimensional stability of the geomembranes.

Plasticiser migration was tested in accordance with regulation UNE 53 095 (AENOR 1981) using circular test specimens 60 mm in diameter at a temperature of (50 ± 1) °C and using expanded polyethylene as the contact material. The test sought to determine the loss of plasticiser in contact with a solid at a certain temperature. This test was mandatory in both the first draft of the current European regulation and in the Swiss and Spanish regulations. However, after some years, it was determined that the laboratory values had no relation to the field installation results. For this reason, this test does not appear in the current regulations.

In the case of these geomembranes, very high migration values were initially observed, and some years later, this

**Table 2. Geomembrane characteristics before installation**

Characteristic	Value
Plasticiser content (%)	34.1
Traction resistance (N/50 mm)	
Longitudinal	2208
Transversal	2203
Elongation at maximum strain (%)	
Longitudinal	18
Transversal	33
Puncture resistance (N/mm)	
External side	531
Internal side	450
Plunger displacement before perforation (mm)	
External side	8
Internal side	5
Seam resistance (N/50 mm)	
By traction	1800
Peeling	438
Migration of plasticisers (%)	
Water behaviour	4.06
Absorption (%)	
1 day	4.00
6 days	9.60
Extraction (%)	
1 day	0.19
6 days	0.30

value had not increased. However, the authors are aware that other materials that initially showed low migration values experienced high levels of migration when they were finally installed in waterworks (Blanco and Aguar 1993).

#### 3.1. Plasticisers

##### 3.1.1. Content

The determination of the plasticiser content levels was carried out in accordance with the Spanish regulations in force at the time, UNE 104 306 and ASTM D2124 (AENOR 2000; ASTM D2124). Extraction with ethyl ether removes all low molecular weight organic products, but it does not extract the vinyl macromolecule and non-organic materials. However, it does extract other additives distinct from the plasticiser, such as UV light absorbents and antioxidants. For this reason, the loss of plasticisers calculation uses a correction in accordance with the scientific literature (Giroud 1995).

### 3.1.2. Identification

Once the plasticisers were isolated, the process of identification began, using Fourier transform infrared spectroscopy (FTIR) (Ortega and Blanco 1982; Blanco *et al.* 1989). The identification is based on the presence of absorption bands that are characteristic of different organic groups. The analysis was performed using a Nicolet 310 spectrometer.

Then, to complete the identification of the additive, gas chromatography (GC) was used in combination with mass spectrometry (MS). An Agilent 6890 N Network was used with a capillary column of phenyl methylpolysiloxane DB%-MS (30 m × 0.25 × 0.25) coupled with an Agilent 5793 MSD quadrupole mass detector system in electron impact mode. The GC allows us to observe and separate the products in the plasticiser, and the MS identifies the elucidated fractions (Blanco *et al.* 2008, 2009).

### 3.2. Folding at low temperatures

PVC geomembrane specimens were folded in accordance with the standard EN 495-5 (AENOR 2013b) method. This test involved maintaining the specimens at  $-20^{\circ}\text{C}$  for 5 h, after which the specimens were folded at an angle of  $180^{\circ}$  and held at this angle for 3 s. The specimens were subsequently inspected for any evidence of cracks, fissures or any other signs of surface imperfections.

The temperature used for the folding tests depended on the geomembrane material and its macromolecular characteristics. Thus, the objective of this test was to assess the suitability of the material, instead of reproducing the temperature conditions the geomembrane would be subjected to during its service life. The dimensions of the test specimens were  $200 \times 200$  mm.

### 3.3. Dynamic impact

The dynamic impact test is a useful parameter for evaluating the capacity of a geomembrane to resist punctures caused by stones, pebbles, or foreign objects that may be thrown into the reservoir by vandals.

This test was performed per the standard EN 13361 (AENOR 2013a) method. A geomembrane passes this test if it is not perforated after dropping a 0.5 kg plunger five times into a hemisphere 12.7 mm in diameter from a height of 500 mm. The concave surface of the hemisphere is placed facing upwards. The presence of punctures was determined by a watertight test.

### 3.4. Puncture resistance

The static puncture resistance and displacement of the plunger before the perforation were measured per the UNE 104317 (AENOR 2011) method. This measurement method was developed by CEDEX and is now part of the standards applied by AENOR (Blanco *et al.* 1996). The method uses an INSTRON dynamometer (model 556). The upper clamps have a cylindrical rod with an irregular pyramid shape ending that allows test specimens to be fitted over a hollow device in the lower part. Ten circular samples (five for each side) that were 50 mm in diameter were tested at low speeds (5 mm/min). A larger plunger

displacement prior to perforation indicated a better puncture resistance.

### 3.5. Tensile characteristics

The tensile characteristics of the specimens were measured per the standard EN ISO 527-3 (AENOR 1996) method using an INSTRON dynamometer (model 5569). The displacement speed was 100 mm/min. The tensile characteristics were determined both longitudinally and transversally. Rectangular test specimens were used because the geomembranes were reinforced with synthetic threads. The polyester fabric used for the reinforcement is responsible for the load and elongation performances, which were measured at the maximum load point. The tensile strength results are expressed as N/50 mm, which is the width of the rectangular test specimen used.

### 3.6. Seam resistance

Seam resistance tests for both the shear resistance and peeling resistance were carried out using an INSTRON dynamometer, model 5569, with a clamp separation speed of 100 mm/min and rectangular test specimens (EN 13 361).

The tensile method is considered a valid qualitative approach to assess the behaviour of the seams between geomembranes. The resulting values reflect the tensile strength in the transverse direction. The specimens broke at the edge or close to the seam, which confirmed the acceptable findings of the test.

Using the allowed material quantity, the peeling resistance test was carried out. The quantitative nature of this test allows comparisons to be made between different materials or between the same materials from different reservoirs or from different areas of the same reservoir when appropriate.

### 3.7. Reflection optical microscopy (ROM) and scanning electron microscopy (SEM)

The microscopic structures of the geomembranes were determined using ROM and SEM. Microscopic evaluations of the geomembranes were carried out according to the literature (Blanco *et al.* 2002; Soriano *et al.* 2010, 2012).

ROM was assessed using a LEICA optical microscope, model DMRX, which was equipped with an automatic photographic system. Microphotographs were taken at  $\times 40$  and  $\times 60$  magnifications.

Likewise, SEM images were acquired using a ZEISS SEM (model EVO 50) equipped with an Oxford Instruments dispersive X-ray spectrometer (model INCA Pentafet X3). However, because these materials are electrical insulators, they were sputtered with a 100–200  $\mu\text{m}$  layer of gold palladium. This task was automatically performed using an Emitech metalliser (model K550). In this case, microphotographs were taken at  $\times 90$  and  $\times 900$  magnifications. Both techniques were used to determine the structures of the geomembranes to assess their deterioration over time.

## 4. RESULTS AND ANALYSIS

The results of the initial material conditions (i.e. measurements performed when the geomembranes were initially installed) indicated that, at this point, the geomembranes fully satisfied the minimum requirements established for these types of geomembranes in the Reservoirs Handbook (MARM 2010) published by CEDEX upon the request of the Spanish Ministry of Environment, Rural and Marine Affairs. The results of the previously described measurements are presented below.

**Table 3. Loss of plasticisers in the geomembranes**

Reservoir	Loss of plasticisers (%)	
	Year sample was taken	
	2012	2015
Hondón de las Nieves	66.6	71.6
El Rollo	84.3	84.3
Plá Aceituna	66.4	70.9
Caseta de Mira	78.5	80.0
El Cid-III	—	78.5
El Rabosero	—	71.1

**Table 4. Characteristics of the Hondón de las Nieves geomembrane 25 years after installation**

	Area the samples were taken	
	Crest	Intermediate
Loss of plasticiser (%)		
Traction resistance (N/50 mm)	71.6	71.4
Longitudinal	3420	3147
Transversal	2121	2425
Elongation at maximum load (%)		
Longitudinal	16	15
Transversal	19	24
Puncture resistance (N/mm)		
External side	351	431
Internal side	320	417
Plunger displacement before perforation (mm)		
External side	7	8
Internal side	5	9

**Table 5. Main FTIR bands for the PVC-P plasticisers**

Wavenumber (cm <sup>-1</sup> )	Chemical group	Bands
3080	H-C=	Stretching vibration (in aromatics)
2965–2870	H-C	Stretching vibrations (methyl and alkyl groups)
1720	C=O	Stretching vibration (carbonyl group)
1600–1585	C=C	Skeletal vibrations (ring stretch)
1460	C=C	Skeletal vibrations (ring stretch)
1455	CH <sub>2</sub>	Scissoring deformation
1375	CH <sub>3</sub>	Symmetrical bending
1300–1075	C-O-C	Symmetrical and asymmetrical stretching
740	C-H	Out of plain ring bending vibrations
720	CH <sub>2</sub> for (CH <sub>2</sub> ) <sub>n</sub> with n ≥ 4	Rocking vibration

### 4.1. Folding at low temperature

Based on the low temperature folding test, all the test specimens from the six reservoirs showed breakages, cracks, fissures, and other signs of surface imperfections. Initially, all the specimens passed the test.

When a homogenous geomembrane begins to have surface imperfections in this test, we observed that a re-waterproofing process should be performed in the reservoir within a few years (Crespo 2011). However, in the case of geomembranes that have been reinforced with a polyester thread fabric, that is, in this case, many years can pass before re-waterproofing is necessary.

### 4.2. Plasticisers

The loss of plasticisers in the samples taken in 2015 is shown in Table 3. The geomembrane from the reservoir Caseta de Mira has not suffered any changes since 2012. However, in other cases, a decrease of 5% was observed.

The most significant characteristics of the samples taken from the northern slope (crest and intermediate areas) of the Hondón de las Nieves reservoir after 25 years of use are summarized in Table 4. It can be observed that while the loss of plasticisers is almost the same for both areas, the mechanical characteristics are better in the intermediate areas. Basically, the puncture resistance is higher, with higher plunger displacement values prior to perforation.

Once the plasticisers were isolated, they were identified via FTIR, which detected the bands that are listed in Table 5. It should be noted that the ester carbonyl absorption frequency is at 1720 cm<sup>-1</sup>. The characteristic ester group vibrations appear in the region of 1300–1050 cm<sup>-1</sup>. In this region, two significant absorption bands appear as a result of the symmetric and antisymmetric vibrations of the ester C-O-C group. The diverse bands clearly indicate an aromatic compound. The 740 cm<sup>-1</sup> band clearly indicates an ortho di-substitution on the aromatic ring. The results indicate that the plasticiser is an alkyl phthalate, which was also verified by the MS results.

Figure 1 shows the MS spectrum of the plasticiser. The following fragmentations were observed: MS, m/z (relative intensity): 390 (M<sup>+</sup>), 279 (C<sub>16</sub>H<sub>23</sub>O<sub>4</sub><sup>+</sup>, 12), 167 (C<sub>8</sub>H<sub>7</sub>O<sub>4</sub><sup>+</sup>, 39), 149 (C<sub>8</sub>H<sub>5</sub>O<sub>3</sub><sup>+</sup>, 100), 113 (C<sub>8</sub>H<sub>17</sub><sup>+</sup>, 9), and 57 (C<sub>4</sub>H<sub>9</sub><sup>+</sup>, 18). This was identified as the bis(2-ethylhexyl) phthalate. Its molecular weight is 390,

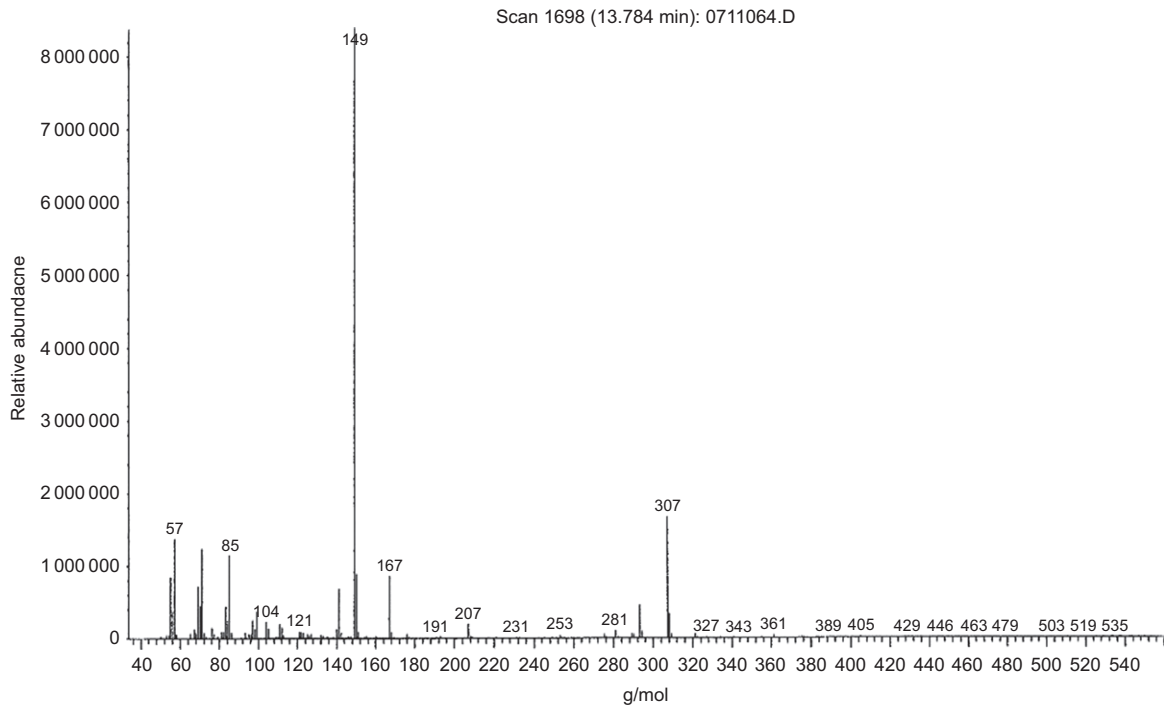


Figure 1. Mass spectrum of the plasticiser extracted from the geomembrane

which is lower than the current recommended value of 400 for high-durability PVC-P geomembranes (PGI 2004; MARM 2010).

The low molecular weight of this additive justifies its tendency to fundamentally migrate to the air (Stark *et al.* 2005). Additionally, this behaviour is consistent with the high losses of plasticisers found in all the geomembranes. GC showed a chromatogram with a single peak at a 82.93 min retention time.

When the PVC-P geomembranes were installed, and even a decade after their installation, bis(2-ethylhexyl) phthalate was the most widely used plasticiser. However, it has since ceased to be used as a unique plasticiser. Currently, plasticisers with the alkyl radicals that contain 10 and 12 carbon atoms are typically used. The higher molecular weights of these plasticisers result in slower migration, and they are more environmentally friendly.

4.3. Dynamic impact

All the test specimens passed the dynamic impact test. Cracks, fissures, or other signs of deterioration were not observed in the impact area after the test device was dropped from a height of 500 mm. Despite the high degree of plasticiser loss, the geomembranes could still withstand the dynamic impact action. This highlights the good condition of the textiles reinforced with fabrics.

4.4. Puncture resistance

The puncture resistance and plunger displacement prior to perforation values are summarized in Table 6. It can be observed that the plunger displacement values are relatively low, and only the membranes from El Rollo would initially pass the Spanish standards for newer geomembranes in the Reservoirs Handbook (MARM 2010).

Table 6. PVC-P geomembrane mechanical and puncture characteristics

	Reservoir					
	Hondón de las Nieves	Plá Aceituna	El Rollo	Caseta de Mira	El Cid-III	El Rabosero
Traction resistance (N/50 mm)						
Longitudinal	3420	3214	3919	2888	2470	2999
Transversal	2121	2333	3977	1943	902	3145
Elongation at maximum load (%)						
Longitudinal	16	23	18	21	14	20
Transverse	19	25	34	24	10	33
Puncture resistance (N/mm)						
External side	351	472	829	499	560	445
Internal side	320	433	812	386	378	318
Plunger displacement before perforation (mm)						
External side	7	5	12	8	11	4
Internal side	5	5	12	7	8	3

It is noteworthy to mention that the reinforcement used in the geomembranes also initially showed low puncture resistance values.

#### 4.5. Tensile characteristics

The tensile values for the samples are shown in Table 6. Only the geomembrane from the El Cid-III reservoir showed low values for both the load and elongation, which agreed with its older installation age (31 years). The membrane has already lost part of the resin covering the reinforced polyester, and the textile is directly exposed to UV radiation, which further degrades it and leads to a reduction in both the load and elongation.

Even in the initial geomembranes, the textile reinforcement showed a high tensile behavior, with values higher than 2200 N/50 mm, which easily fulfilled the minimum Spanish requirements (1100 N/50 mm). However, these values have increased over time due to the higher rigidity of the sheets and the loss of plasticisers.

#### 4.6. Seam resistance

The seam resistance values observed from the tension procedure are shown in Table 7. The obtained values are relatively high because of the reinforcement textile. All of the values surpass 2000 N/50 mm, with the exception of those from the El Cid-III and El Rollo reservoirs, which have the oldest installation age (1980).

The peeling method resistance test was only performed for the El Rabosero reservoir because of the joining between the panels. The geomembrane from this reservoir provided a moderately acceptable value of 302 N/50 mm considering its long installation time (21 years).

**Table 7. Joint shear resistance**

Reservoir	Joint shear resistance (N/50 mm)
Hondón de las Nieves	2178
El Rollo	1119
Plá Aceituna	2062
Caseta de Mira	2036
El Cid-III	1651
El Rabosero	2593

#### 4.7. ROM and SEM

The original samples observed using SEM at  $\times 90$  and  $\times 900$  magnifications are shown in Figures 2 and 3, respectively. In the first image ( $\times 90$ ), the presence of micropores can be observed, and they are clearly evident in the second microphotograph taken at a higher magnification ( $\times 900$ ).

For all the 2015 samples, Figure 4 depicts the ROM images for the external side at  $\times 60$  magnification. Likewise, Figure 5 shows the appearance of the external side of the materials by SEM at  $\times 90$  magnification. Both the ROM and SEM images reveal a significant deterioration in the external side of the geomembranes, with visual evidence of surface cracking.

For the samples exhumed from the El Cid-III reservoir 31 years after installation, the microscopy images of the internal side show good material conditions (Figure 6). The comparison between the two sides of the test specimens illustrates the effect of UV radiation on the deterioration of the organic material.

Finally, Figure 7 shows a set of SEM ( $\times 90$ ) microphotographs of the geomembrane from the northern slope (south facing) of the Hondón de las Nieves reservoir. The microphotographs in the upper part correspond to the crest area of the reservoir, which is where the geomembrane was always in contact with solar radiation. The microphotographs in the lower part were taken of the membrane from the intermediate area, which is where the geomembrane was exposed to weathering and covered by water.

The external side shows notable cracking, which was slightly greater in the crest sample, and for the internal side, some craters can be observed. Possibly, these craters are related to the micropores that were initially observed in the geomembrane and are a consequence of the impregnation process used in the manufacturing. The coalescence of these pores and material ageing cause the formation of craters and cracking over time.

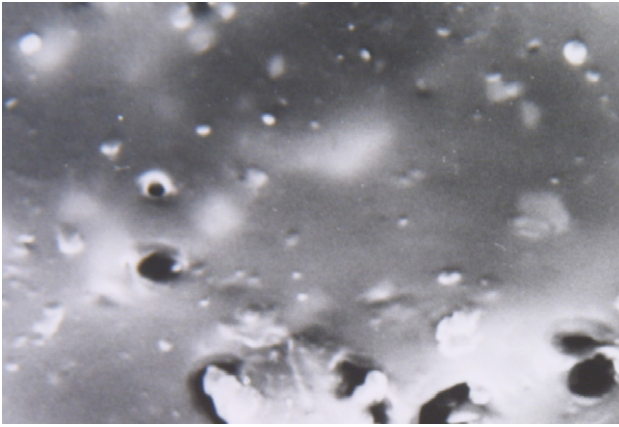
## 5. GEOMEMBRANE END OF LIFE

The durability of geomembranes depends on intrinsic factors, such as their composition and thickness, and extrinsic



**Figure 2. SEM microphotograph of the original geomembrane at  $\times 90$  magnification**



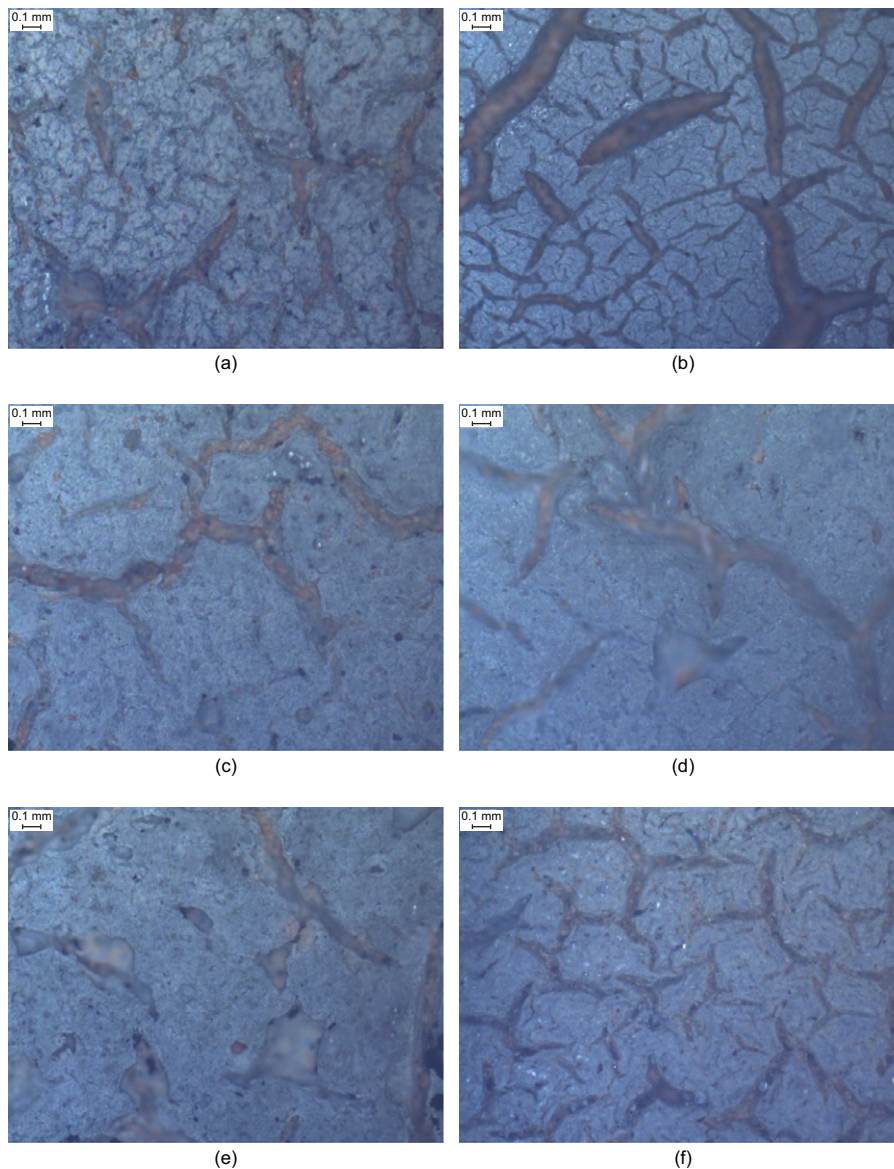


**Figure 3. SEM microphotograph of the original geomembrane at  $\times 900$  magnification**

factors, such as stresses and different environmental agents (Cazzuffi 2014). The latter will affect all types of geomembranes, irrespective of the nature of the resin. A photo- and thermal oxidation process will produce a series of free radicals that cause a haemolytic chain reaction, which deteriorates the macromolecule. However, some intrinsic factors are specific to each geomembrane.

The lifetime of a geomembrane can be assessed via the evolution of one or several fundamental characteristics, depending on the nature of the geomembrane (Noval 2015b). Different authors have proposed that the service life of a geomembrane comes to an end when this fundamental characteristic loses 50% of its value. However, this value is not defined. The scientific literature considers two possibilities, which can be defined as

- Theory A. The service life ends when the fundamental characteristic under consideration has lost 50% of its original value (Hsuan and Koerner 1998).



**Figure 4. ROM microphotographs of the external sides at  $\times 60$  magnification. (a) Hondón de las Nieves; (b) Plá Aceituna; (c) El Rollo; (d) Caseta de Mira; (e) El Cid-III; (f) El Rabosero**



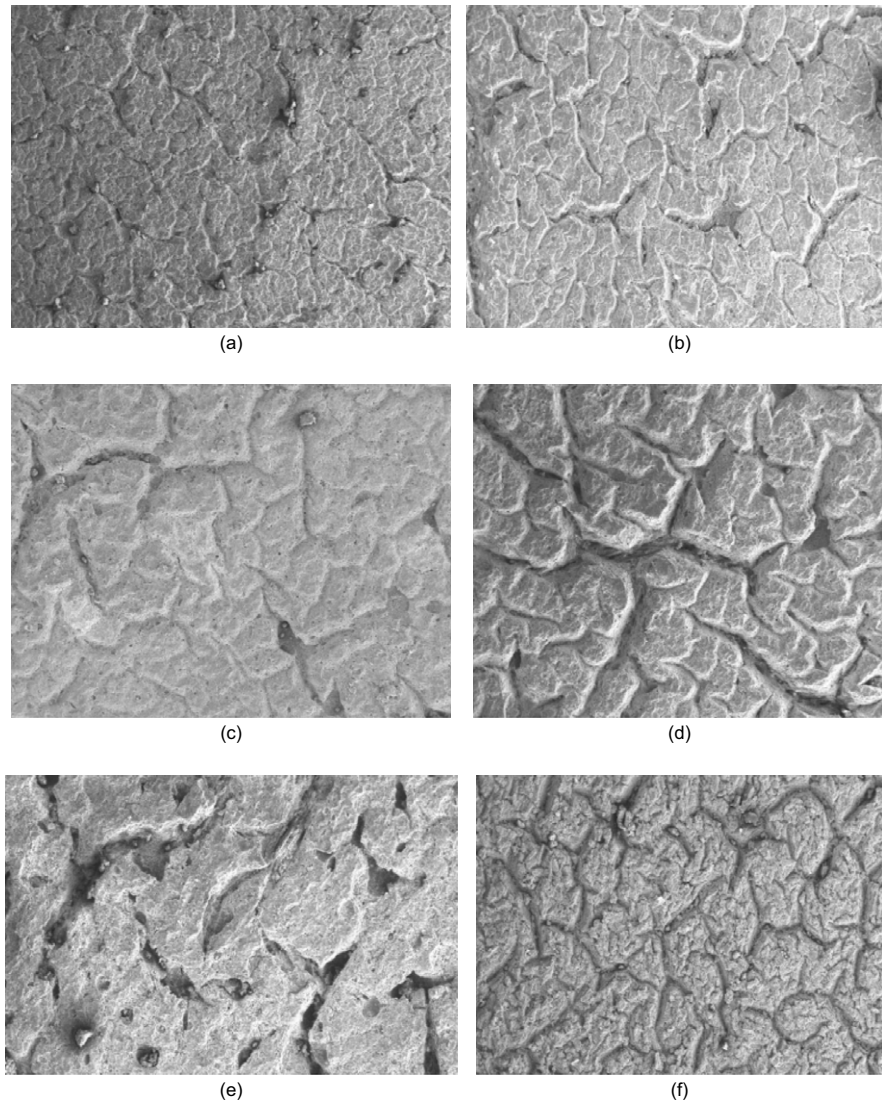


Figure 5. SEM microphotographs of the external side of the geomembranes at  $\times 90$  magnification. (a) Hondón de las Nieves; (b) Plá Aceituna; (c) El Rollo; (d) Caseta de Mira; (e) El Cid-III; (f) El Rabosero

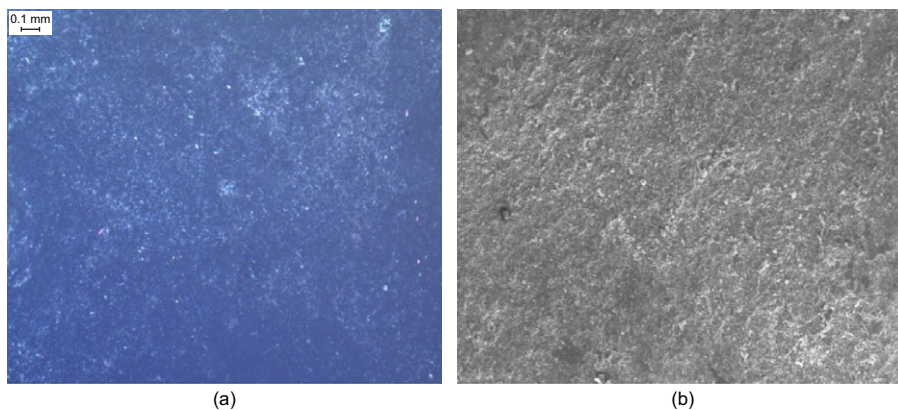
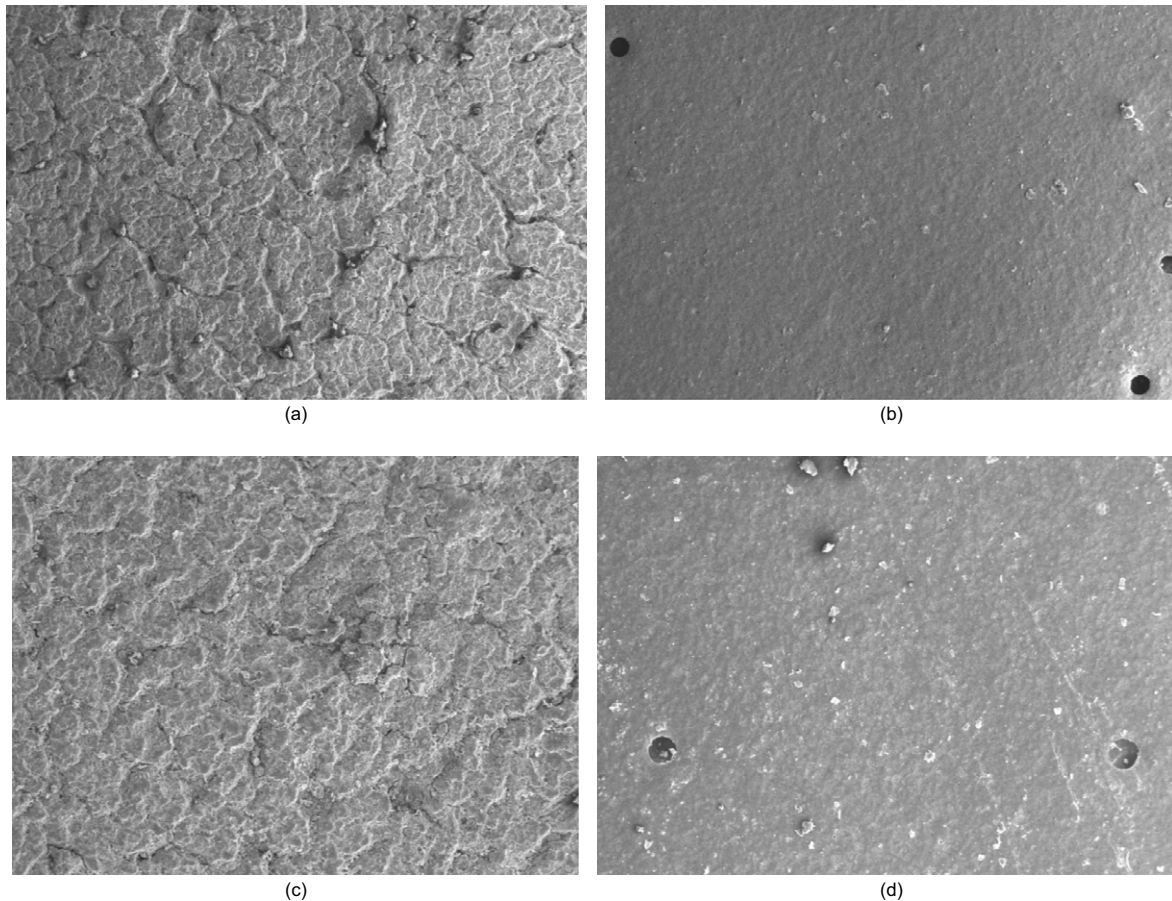


Figure 6. (a) ROM and (b) SEM microphotographs of the internal side of samples from the El Cid III reservoir at  $\times 60$  and  $\times 90$  magnification, respectively

- Theory B. The service life ends when the fundamental characteristic under consideration has lost 50% of the value required by current regulations (Rowe *et al.* 2009).

The above theories refer to HDPE geomembranes. Regarding the key parameter under consideration, different international forums refer to stress cracking and oxidation induction times. However, some authors



**Figure 7.** SEM microphotographs of the geomembrane from Hondón de las Nieves reservoir at  $\times 90$  magnification. (a) External side at crest, (b) internal side at crest, (c) external side in the intermediate area, (d) internal side in the intermediate area

reference the carbonyl index and durability factor, which is defined as the coefficient between stress cracking and the carbonyl index (Blanco 2015).

Based on this approach, we extended these theories to other types of geomembranes. EPDM geomembranes have been successfully evaluated using the elongation at break, since this fundamental characteristic significantly decreases over time as the polymer vulcanisation increases (Blanco 2015; Noval 2015a).

For PVC-P, in addition to the folding at low temperature, we also considered the loss of plasticisers. Our field experience, based on periodic monitoring of more than two-hundred exposed hydraulic structures throughout Spain, indicates that when the folding begins to fail, the geomembrane service life is nearing the end. The geomembrane may last a few more years depending on the geographic location of the structure and the mechanical requirements of the reservoir, but overall, the integrity of the material is severely damaged (Blanco *et al.* 2003b, 2012). In contrast, the reservoirs studied in this research showed significant durability levels although the samples broke in the folding test. The polyester-reinforced textile contributes to the better performance of the geomembranes, and additional key characteristics should be considered in the durability assessment. Thus, the loss of plasticisers was also considered in the PVC-P weathering approach.

**Table 8.** PVC-P values of the main characteristics after losing 50% of their original values according to Theory A & B

Characteristic	Theory A	Theory B
Plasticiser content (%)	17.1	—
Traction resistance (N/50 mm)		
Longitudinal	1104	550
Transverse	1102	550
Elongation at maximum load (%)		
Longitudinal	9	8
Transversal	17	8
Puncture resistance (N/mm)		
External side	266	—
Internal side	225	—
Plunger displacement before perforation (mm)		
External side	4	6
Internal side	3	6

The values for the most important characteristics of the PVC-P geomembranes for both theories are summarized in Table 8. These technical requirements are fully described in the Spanish Reservoirs Handbook (MARM 2010). In theory B, the plasticiser content is not considered a fundamental requirement because any manufacturer can change rigid PVC into flexible PVC using different methods, including internal plasticisation. Additionally, the puncture resistance results for theory B (Table 8) only consider the data from the plunger

displacement prior to perforation, since this characteristic best defines the puncture behaviour (MARM 2010). All the geomembranes studied surpass the minimum required values for both theories. However, as shown in Table 3, all the tested geomembranes have suffered a loss of plasticiser that is far higher than 50% of its original content.

The straightforward application of the above theories for an exposed PVC-P geomembrane installed in an irrigation reservoir is too restrictive. As mentioned above, the authors have assessed and monitored different geomembranes in Spain that do not meet the requirements of the aforementioned theories over time. However, the geomembranes continue to fulfil their waterproofing purpose. For PVC-P, several cases support this conclusion since more than 50% of the additive was lost but the geomembrane has not yet been removed. Moreover, some PVC-P geomembranes installed in Spain exhibited losses higher than 80%, but they still fulfil their working requirements in the reservoirs (Blanco *et al.* 2013b; Noval *et al.* 2014c). Nonetheless, it is important to stress that these geomembranes are reinforced with a synthetic thread textile.

## 6. CONCLUSIONS

The work reported in this paper is based on studying exposed PVC liners in the southeast of Spain and installed according to European practice. The applications, design practice, materials and performance may not apply to other regions and jurisdictions.

The tests carried out on the original geomembranes verified that they fully satisfied the Spanish regulations in force for a PVC-P geomembrane at the time of their installation. Accordingly, the mechanical characteristics of these materials are excellent, and they have remained well preserved over time. The tension resistances of the geomembranes are very high; that is, double the minimum values required by regulations for this type of geomembrane, except for the geomembrane installed 31 years ago. This is due to the geotextile reinforcement. The results, based on the location the samples were exhumed from in the reservoirs, show that the samples from the intermediate area have a better mechanical performance, but the differences were not significant. Regarding the hydraulic working performance of these waterproofed reservoirs, water leakage over time through the drainage monitoring system has not been observed.

The data obtained in the microscopic analysis show geomembranes with internal sides that are in good condition with isolated craters; however, the external sides show a high level of deterioration and surface cracking.

The plasticiser was identified after extraction with ethyl ether using FTIR, GC and MS, and it was bis(2-ethylhexyl) phthalate, which has a low molecular weight of 390 and a branched alkyl radical that promotes air migration. The alkyl radical substitutes the phthalic acid protons, leading to a significant loss of this additive.

The high durability of these geomembranes is due to the behaviour of the reinforcement polyester textile.

The reinforced textile is still covered with vinyl resin, which prevents solar radiation exposure.

The durability assessment of the geomembranes is based on the loss of fundamental characteristics. The plasticiser content was the key parameter analysed. The results revealed that the loss of plasticisers was greater than 70% in all cases and reached 84% in one sample. Therefore, the loss of plasticisers is significantly greater than the 50% threshold referred to in scientific recommendations, but all the geomembranes still provide containment.

In conclusion, the geomembranes are still installed in reservoirs and still fulfil their waterproofing function, which implies that the current loss of plasticisers durability criteria needs to be further discussed based on the extra field reservoir data.

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## ABBREVIATIONS

AENOR	Asociación Española de Normalización y Certificación
BALTEN	Balsas de Tenerife
CEDEX	Centro de Estudios y Experimentación de Obras Públicas
CPE	Chlorinated polyethylene
EN	European Norm
EPDM	Ethylene-propylene-diene terpolymer
FTIR	Fourier transform infrared spectroscopy
GC	Gas chromatography
HDPE	High density polyethylene
ICOLD	International Commission on Large Dams
IIR	Butyl rubber
LDPE	Low density polyethylene
MARM	Ministerio de Medio Ambiente y Medio Rural y Marino
MS	Mass spectrometry
PIB	Polyisobutylene
PVC-P	Plasticised poly(vinyl chloride)
ROM	Reflection optical microscopy
SEM	Scanning electron microscopy
UNE	Spanish Standard
UV	Ultraviolet

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