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Comparative study of three different kinds of geomembranes (PVC-P, HDPE, EPDM) used in the waterproofing of reservoirs.

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ABSTRACT. This work describes the long-term behaviour of three kinds of geomembranes which are constituted by plasticized poly vinyl chloride (PVC-P), high density polyethylene (HDPE) and terpolymer rubber of ethylene-propylene-dienic monomer (EPDM), used as the waterproofing system of the reservoirs “Los Llanos de Mesa”, “San Isidro” and “El Golfo”, respectively.

Characteristics of the three original geomembranes and their behaviour along time are presented. Thicknesses, content and nature of the plasticizers (in PVC-P), tensile properties, dynamic and static puncture, foldability at low temperature, shore hardness, tear resistance and carbon black (in HDPE), joint strength (shear and peeling test) and microscopy, both optical and electronic scanning tests were carried out. Results obtained conclude with a long-term durability of geomembranes, independently of their macromolecular nature.

These characteristics were determined by advanced analytical techniques in PVC-P samples, such as Fourier Transform Infrared Spectroscopy (FTIR), Gas Chromatography (GC) and Mass Spectrometry (MS).

1 INTRODUCTION

Plasticized polyvinyl chloride geomembranes (PVC-P) have been the most used material in the waterproofing of reservoirs in Spain in the 70's and 80's. It was possible because of two different facts: a new normative frame for this product and the Hydrologic Plan of Canarias Islands (Spain).

From that point of view, IRANOR institution (“Instituto para la Racionalización y Normalización del Trabajo”) created a working group in the late seventies who elaborated a PVC-P regulation in order to use those geomembranes for waterproofing of reservoirs and published a standard for homogeneous sheets. Afterwards, other methods were developed, for instance, related to glass fibre and synthetic fabric reinforcements, both resistant and non-resistant to bituminous materials and, for different applications like Civil Engineering or Building Works.

When Spain took part in the European Union, the IRANOR institution disappeared and, because of the European requirements, AENOR (Spanish organization of normalization and certification) appeared as private institution for this business. The existence of these clear regulations and right experimental methodology induced the substitution

of butyl rubber sheets into PVC-P geomembranes (Blanco 2005).

On the other hand, the Hydrologic Plan of Canarias managed water resources in order to take the maximum benefit and built some reservoirs with this sort of material. Periodic controls both at the beginning and on the long-term contributed to use and develop different geomembranes and, particularly, PVC-P geomembranes (Amigó & Aguiar 1994, Aguiar & Blanco 1995).

The use of this vinyl product had its maximum peak in the indicated dates of this epigraph and it has been used until nowadays (Zapata et al. 2010) not only in this technology field but in tunnels (Usillos 2008), canals (Machado 2004, Levita 2008), mineral waste deposit liners (Rubín de Célix 2005) and dams (Scuero et al. 2010, Thanopoulos et al. 2010, Cazzuffi et al. 2010).

Although different densities of polyethylene materials are used as waterproofing systems (Blanco et al. 2010a), the extensive use of high density polyethylene (HDPE) can be observed. Geomembranes of this nature have been used with abundance for years (Blanco & García 2010) and some of them with excellent results over time, as the one installed in the reservoir of “Plá de Sant Jordi” (Palma de Mallorca, Spain) (Blanco et al. 2010b). The reason why this material is so extensively used nowadays can be due to two factors. First, the economic point of view, as big

First, the economic point of view, as big supply and competence causes good prices in contrast to other kind of geomembranes. Second, the material is a very stable polyolefin, inert chemically and with long-term durability (Koerner 1999) so landfills are usually waterproofed with it (Giroud & Touze-Foltz 2003, Zornberg 2010).

Terpolymer rubber (EPDM) is the macromolecular compound of a geomembrane used in Hydraulic Works and Building Works (Puig 2010); in this last application, EPDM has an extensive use and it could be considered to be the most used synthetic barrier in countries like USA (Davis et al. 1998). In Spain, its used has been increasing along time in reservoirs and a lot of experience has been gained as regards its behaviour once installed (Abad et al. 2010, Blanco et al. 2010). The paper describes the long-term behaviour of three kind of geomembranes which are constituted by plasticized polyvinyl chloride (PVC-P), high density polyethylene (HDPE) and terpolymer rubber of ethylene-propylene-dienic monomer (EPDM) which are part of the waterproofing system of “Los Llanos de Mesa” (Fig. 1), “San Isidro” (Fig. 2) and “El Golfo” reservoirs (Fig. 3) respectively.

The purpose of this study is not to say which material performs better or is having a superior behaviour. The geomembrane nature is not a decisive factor, it can be thermoplastic or thermostable but with a correct formulation.



Figure 1. Reservoir of “Los Llanos de Mesa” waterproofed with PVC-P.

2 THE RESERVOIRS

The follow up of the behaviour of different geomembranes along time was presented in recent research works in order to check their durability since installation (Blanco et al. 2005). This work is a collaboration project between “Centro de Estudios y Experimentación de Obras Públicas

(CEDEX) (Spain)”, “Balsas de Tenerife (BALTEN) (Spain)”, “Universidad Complutense de Madrid (UCM) (Spain)” and “IRSTEA (France)”.



Figure 2. Reservoir of “San Isidro” waterproofed with HDPE.

The reservoirs selected for this study are located in The Canary Islands (Spain) and their features are presented in Table 1, such as: capacity, slopes, crest perimeter and height, location, nature of geomembrane and thickness, material used and year of installation.

Table 1. Reservoirs whose geomembranes are studied in this work.

Characteristics	Reservoir		
	Los Llanos de Mesa (LM)	San Isidro (SI)	El Golfo (EG)
Location	San Juan de la Rambla	Granadilla de Abona	La Frontera
Capacity, m ³	175,961	49,799	145,000
Height, m	16.0	11.0	13.0
Crest Perimeter, m	681.0	212.7	--
Perimeter, m	585.00	328.19	--
Slope	--	--	2:1
Type of geomembrane	PVC-P	HDPE	EPDM
Thickness, mm	1.50	1.50	1.52
Material used, m ²	22,930	8310	20,000
Year of installation	1986	1991	1995

3 EXPERIMENTAL

Firstly, the initial characteristics of the geomembranes were determined in order to check their validity and to get initial values to use as a basis for comparison while monitoring them along time. For this purpose, samples were taken periodically in different zones of the reservoirs and tests were repeated along time to check the

along time to check the evolution of the same parameters.



Figure 3. "El Golfo" reservoir which is waterproofed with an EPDM polymeric geosynthetic barrier.

The experimental methodology used in this research project was developed by the European standard EN 13361 (2005). Puncture resistance tests were performed according to UNE 104 317 (2011), the method developed by this research team which, nowadays, belongs to the standards applied by the Spanish organization of normalization and certification AENOR (Blanco et al. 2006). Tensile tests and the determination of elongation at break were made in both longitudinal and transverse direction, the values presented in this work will be those obtained in the longitudinal direction.

Besides, the tests related to dynamic impact were performed on both surfaces of the specimens, internal and external, but only the results obtained on the external surface will be presented in this research work.

Tests carried out at the beginning, had passed the minimum requirements established for these types of geomembranes according to the Reservoirs Handbook (Manual de Balsas, 2010) written by CEDEX following a request of "Ministerio de Medio Ambiente y Medio Rural y Marino".

3.1 Common tests

3.1.1 Folding at low temperature

Specimens of the three materials of the reservoirs were tested with a folding at low temperature test. This test consists in introducing the specimens in a cold store during 5 hours at a given temperature and after a flexing of 180° during 3 seconds. This test was conducted on both faces of the samples, external and internal. After that, it was checked if the specimens presented cracks, fissures or any other sign of deterioration

The folding temperature depends on the material and its macromolecule; It is thus a test to check the

suitability of the material, not expected to representative of the temperature at which the geomembrane will be exposed on site. Folding temperatures are presented in Table 2. The test was passed by all the specimens at the beginning but, while HDPE and EPDM geomembranes continued to pass the requirements, PVC-P north-samples did not pass the test after 13 years and the south-samples presented cracks after 20 years of installation.

Table 2. Folding temperature depending on the type of synthetic geomembrane.

Material	Test Temperature, °C
EPDM	-55
HDPE	-75
PVC-P	-20

3.1.2 Dynamic Puncture

Original thermoplastic specimens passed the resistance to dynamic puncture test as none of them suffered perforation in the impact area after throwing a 0.5 kg plunger drop ended in a semi sphere ball whose diameter was 12.7 mm from a 500 mm height. This was checked with a watertight test. In the thermostable sample which was located in "El Golfo" reservoir, the height was 350 mm and the test was passed by this one too.

This is an interesting test to take into account the problems that could occur during the installation of the sheet in the reservoir and afterwards, such as vandalism.

In general, the height from which the plunger drop felt without causing any damage on the impact area decreased as a consequence of resin ageing. In the particular case of PVC-P, it is necessary to add to resin ageing the loss of plasticizers over time.

EPDM had better behaviour over time. Indeed two years after installation it was able to exceed the height of 500 mm, like thermoplastics materials. This fact is a consequence of the end of the vulcanization process in the elastomeric material which occurs after installation.

3.1.3 Tensile strength characteristics

Values for tensile strength and elongation at break are presented in Table 3. In the elastomeric material, important changes in tensile strength values were not observed twelve years after installation. The HDPE geomembrane was able to maintain the load over years in spite of an important decrease at the beginning.

The PVC-P geomembrane did not exhibit really significant changes in tensile strength and elongation at break values in relation with the

tion at break values in relation with the polyester fabrics reinforcement responsible for these properties. After twelve years, the polyester fabrics had not been attacked by ultraviolet radiation proceeding from the sun because they were protected by the resin which was in a good state.

The evolution of both the shear and elongation at yield point were determined for the HDPE geomembrane; shear values ranged between 22.9 MPa and 21.3 MPa after twelve years, while elongation in this point fluctuated around 13%.

Elongation at break of the two homogeneous geomembranes decreased over time, especially in the thermostable material.

Table 4 intends to evince the influence of the geographical orientation in the deterioration of geomembranes. In this attempt results over nine years of open air exposure are presented.

Samples were taken in north and south slope in the coronation area. The test considered to be the most significant in order to demonstrate the influence of ultraviolet radiation over materials for this work were: tensile strength, elongation at break and static puncture.

It could be checked that the influence in the rubber material is null and is really small in HDPE. The reason for not detecting appreciative changes in PVC-P is due to the reinforcement of the geomembrane, so that, in this case, these properties are not significant during the first years in use. Consequently, tests to quantify the loss of plasticizers and folding at low temperature should be carried out to detect the mentioned changes.

3.1.4 Static Puncture resistance

Figures 4 and 5 represent the variation of resistance to static puncture and the displacement of the plunger before perforation, respectively. In general issues, resistance to static puncture values increased over time in the same way as displacement of the plunger before perforation values decreased. This decrease is more important in the case of EPDM but its value over twelve years is better than the original results of the considered thermoplastic materials.

The behaviour at static impact is considerably better in elastomeric than in thermoplastic materials such as HDPE and PVC-P, while in dynamic impact it happens the other way round.

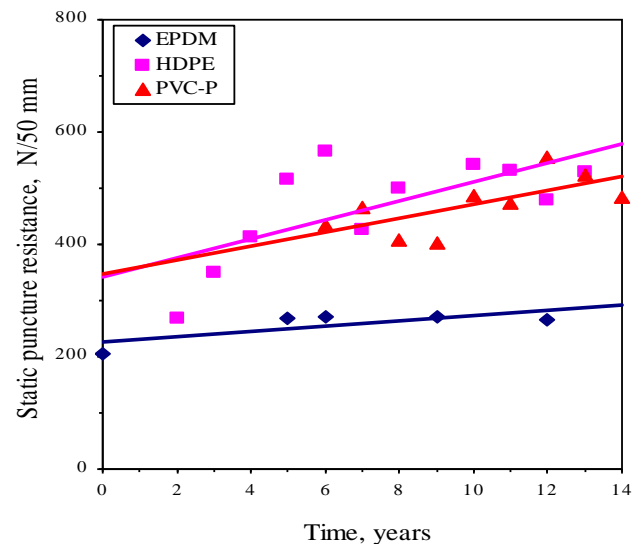


Figure 4. Static puncture resistance evolution, time function.

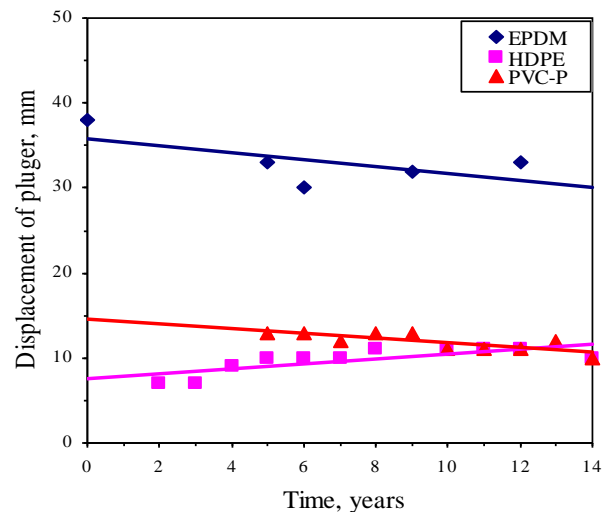


Figure 5. Displacement of plunger before perforation, time function.

3.1.5 Seams resistance

Seams resistance determined by shear resistance test presented acceptable values as samples broke in the edge or close to the seam, but always outside of it. Values reached are shown in Figure 6.

Table 3. Evolution of tensile strength characteristics over time.

Years	Tensile Strength, MPa			Elongation at break, %		
	EPDM	HDPE	PVC-P*	EPDM	HDPE	PVC-P*
0	10.0	38.0	1325	553	1083	22
5	10.1	24.1	1630	410	612	23
6	10.3	27.1	1493	358	689	22
7	10.9	26.4	1478	359	692	23
8	10.8	27.6	1460	396	725	23
9	10.7	21.1	1440	352	410	23
10	10.4	24.2	1488	325	532	24
11	10.3	23.4	1433	337	610	24
12	9.4	34.5	1465	283	639	23

* Because it is a reinforced sheet the tensile strength is expressed in N/50 mm and the elongation is the point of maximum load.

Table 4. Geographic orientation influence in the behaviour of EPDM, HDPE and PVC-P geomembranes nine years after installation.

Characteristics	Type of geomembrane		
	EPDM	HDPE	PVC-P
Tensile strength, MPa			
- North	10.7	29.9	1440*
- South	10.6	34.3	1485
Elongation at break, %			
- North	352	705	23*
- South	359	888	21
Displacement of plunger, mm			
- North	32	12	13
- South	32	10	15

* Because it is a reinforced sheet the tensile strength is expressed in N/50 mm and the elongation is the point of maximum load.

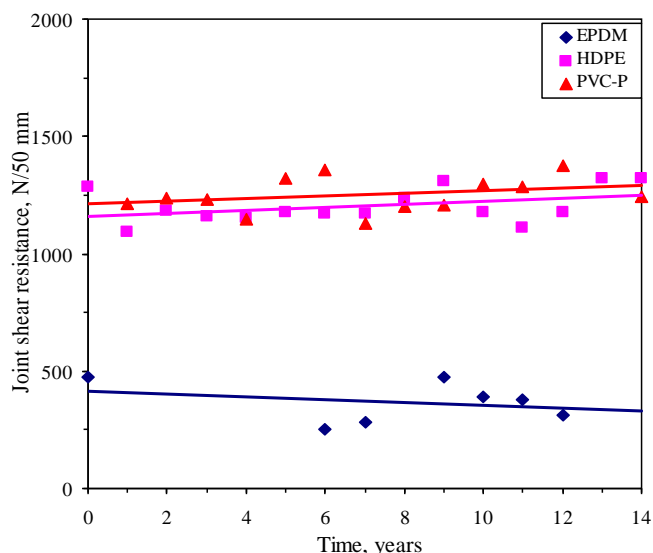


Figure 6. Joint shear resistance evolution, time function.

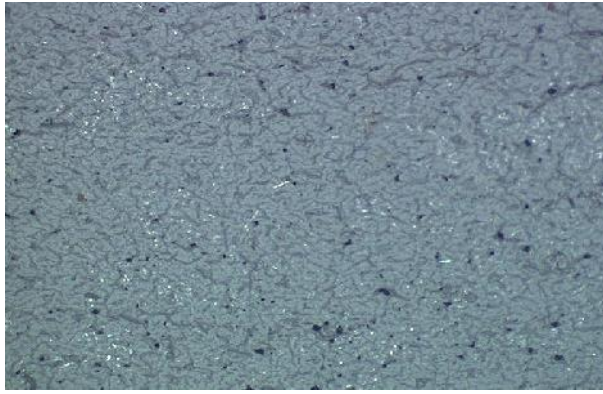
3.1.6. Reflection optical Microscopy and Scanning Electron Microscopy

Microscopy evaluation of EPDM geomembranes was carried out according to the literature (Soriano et al. 2006, 2010). Microphotographs were taken by Reflection Optical Microscopy (ROM), at x40

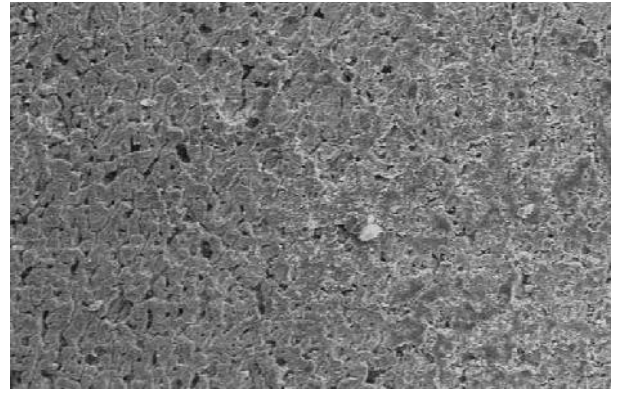
and x60 magnifications; the purpose was to see the texture and morphology of the samples. Figure 7 represents the external surface at x60 magnifications of the different geomembranes after ten years of installation. Besides, the behaviour of these geomembranes ten years after installation was also checked by Scanning Electron Microscopy (SEM) at x90 and x900 magnifications, Figure 8 represents the external surface at a x90 magnification of these materials.

Reflection Optical Microscopy shows a PVC-P geomembrane with important ageing symptoms as by the presence of abundant micropores and superficial cracking. The HDPE geomembrane presents grooves of manipulation, although both this material and the rubber show uniform and homogeneous surfaces.

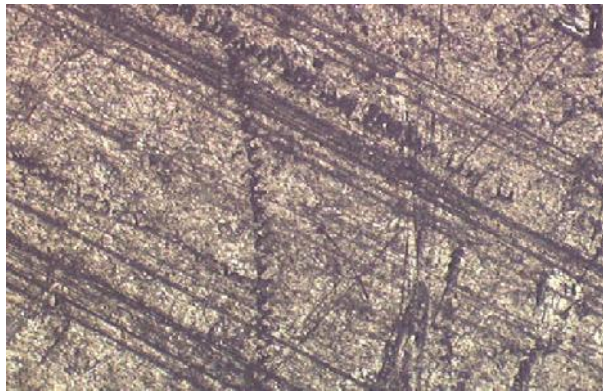
Scanning Electron Microscopy (SEM), like Reflection Optical Microscopy confirms the facts cited above and besides, SEM detected microcracks in the elastomeric geomembranes.



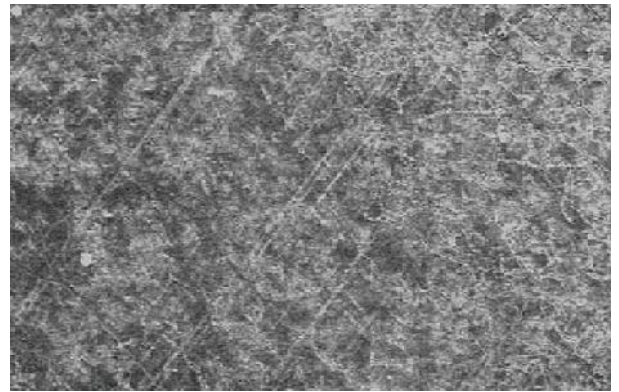
PVC-P



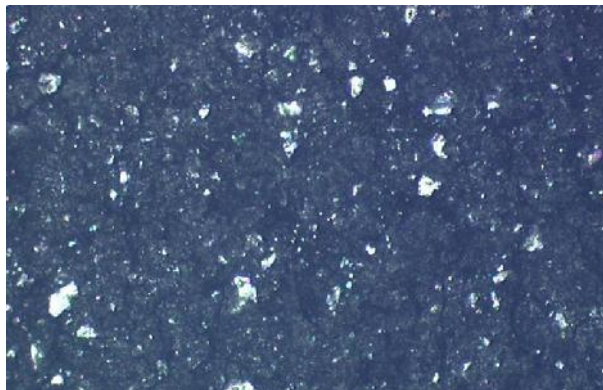
PVC-P



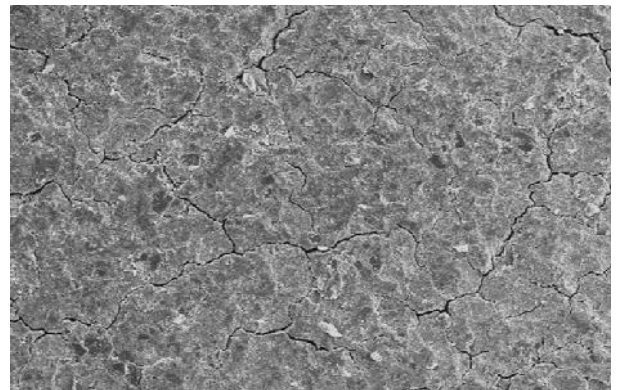
HDPE



HDPE



EPDM



EPDM

Figure 7. Microphotographs ROM (x 60) of the external face ten years after installation.

Figure 8. Microphotographs SEM (x 90) of the external face ten years after installation.

3.2 Particular tests

3.2.1. Shore hardness

Table 5 presents the values reached after twelve years of service life for both EPDM (Shore-A Hardness) and HDPE (Shore-D Hardness) geomembranes. In EPDM the hardness had a trend to increase over time. In HDPE the hardness values were very similar or with a trend to decrease slightly over time.

Table 5. Shore Hardness evolution over time.

Years	Shore Hardness	
	A (EPDM)	D (HDPE)
0	64	59
5	72	61
6	71	60
7	70	58
8	70	56
9	74	61
10	71	57
11	72	58
12	76	58

3.2.2 Tear resistance

The HDPE tear resistance evolution is shown in Figure 9 during the fourteen years of service life of the geomembrane in the reservoir. No appreciable variation is observed during this period. This test was carried out in HDPE for periodic controls because some isolated cases of tearing occurred close to the seams due to the weakening of seam process.

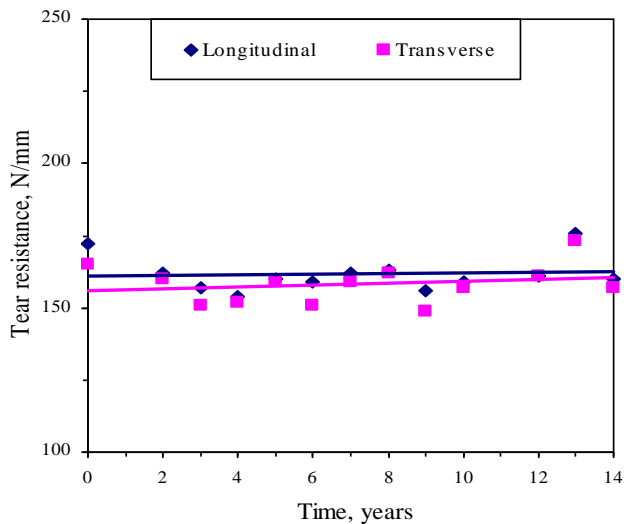


Figure 9. HDPE tear resistance evolution.

3.2.3 Carbon black

The carbon black content was 2.5% in the HDPE samples, which is a correct value. Indeed a value in the range 2 to 3%, is recommended. Figure 10 presents the way carbon black was dispersed in the geomembrane without accumulation points of this additive. It could be observed that the dispersion of carbon black had a value of 2 on a 1-7 scale, the lower the value, the better the dispersion.



Figure 10. Photograph of carbon black dispersion.

3.2.4 Content and nature of plasticizers.

The initial content of plasticizers determined in the original PVC-P geomembrane was 32.3%. Periodic controls have been carried out for twenty years in order to follow the plasticizers content evolution after installation in the reservoir. Figure 11 shows the loss of this additive in both north and south samples; the loss obtained were 38.8% in south-slope samples and 45.3% in the north ones. North-slope is the area the most affected by radiation proceeding from the sun so it is the most deteriorated zone of the reservoirs' geomembrane.

First of all, plasticizers included in the geomembrane formulation were determined by extraction with ethyl ether, following the procedure described in the literature (Crespo 2011). Second, they were analysed by Fourier Transform Infrared Spectroscopy (FTIR) where characteristic bands of alkyl phthalates were obtained (Blanco et al. 2008).

The additive identification was carried out with Gas Chromatography technique combined with Mass Spectrometry technique (CG-MS). The first technique allows to know and to separate the number of products which take part in the plasticizer and the second one, permits to identify these products (Blanco et al. 2010d).

Gas Chromatography technique generated a chromatogram where two peaks representative of retention times of 57.61 and 82.03 minutes appeared, in percentages of 3.0% and 97.0%, respectively. This fact indicates the existence of two different plasticizers in the geomembrane formulation.

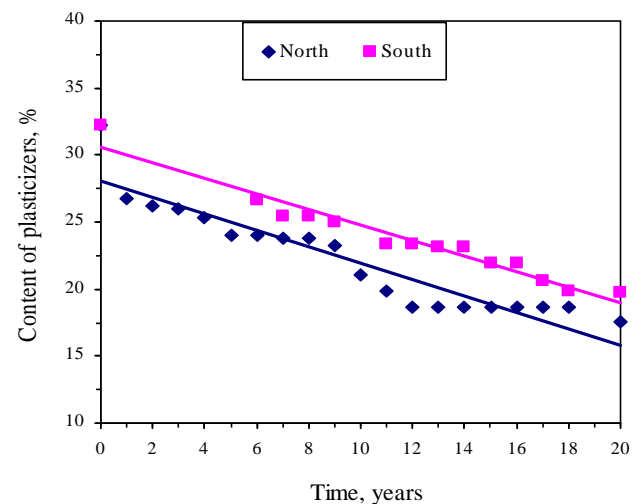


Figure 11. Loss of plasticizers over time.

Afterwards, the two isolated products were analysed by Mass Spectrometry whose spectrum results were the following:

- Spectrum results of the peak at 57.61 minutes (retention time in the chromatogram) were: 390(M⁺), 279(C₁₆H₂₃O₄⁺,12), 167(C₈H₇O₄⁺,39), 149 (C₈H₅O₃⁺,100), 71(C₅H₁₁⁺,14), 57 (C₄H₉⁺,18). All of these results referred to MS versus m/z (relative intensity). These fragmentations indicated that it was *Bis(2-ethylhexyl) phthalate*, and
- Second peak whose spectrum is presented in Figure 14, presented the following results: MS, m/z (relative intensity):446(M⁺), 307(C₁₈O₄H₂₇⁺,25), 167(C₈H₇O₄⁺,19), 149(C₈H₅O₃⁺,100), 85(C₆H₁₃⁺, 25), 57(C₄H₉⁺,29). The fragmentations indicated that it was *Diisodecyl phthalate*.

In both cases, alkyl radicals that substitute the proton of phthalic acid were branched, so they were susceptible to air migration.

Besides, the average molecular weight (Wilson 1995) whose value was 440.44 was calculated from the existence of two additives. As this value is higher than 400, the geomembrane presents a huge durability according to the literature (PGI 2004, Blanco et al. 2008).

4 CONCLUSIONS

At the beginning, all geomembranes passed the minimum requirements established in the current law; however, macromolecular material always suffers an ageing process due to its organic nature which will be higher or lower depending on the adverse conditions of the installation place, particularly, the incidence of solar radiations.

After carrying out the comparative study about the behaviour of the three materials, it can be concluded that:

- Folding at low temperature test presented correct values over time, excepting in the PVC-P geomembrane whose north-slope samples exhibited cracks after thirteen years of service life while cracks only appeared in the south-slope samples after twenty years.
- Tensile strength characteristics pointed out a decrease of elongation values in all the samples except for the PVC-P geomembrane. The reason is that this is a reinforced sheet and fabrics had not been attack by solar radiation because it was protected by the resin and therefore, no change occurred.
- The best behaviour to static impact was for the EPDM geomembrane for which a displacement of the plunger before perforation higher than for the other geomembranes, even

considering the initial values was observed along time.

- In contrast, the resistance to dynamic impact was better in the PVC-P and the HDPE than in the EPDM.

- The microscopy test showed homogeneous and uniform surfaces, with slight ageing symptoms and some microcracks.

- The shore hardness increased over time in the elastomeric material. On the other hand, the values in the HDPE geomembrane were very similar to the initial values or with a slight trend to decrease over time.

- Additives of plasticizers were determined in the PVC-P geomembrane. The plasticizers were a mixture between *Bis(2-ethylhexyl) phthalate* and *Diisodecyl phthalate*, whose average molecular weight was a high value. This fact indicates that the geomembrane should exhibit a significant durability. However, alkyl radicals that substitute the proton of phthalic acid are branched, and thus are susceptible to air migration.

The three materials considered are suitable for waterproofing of hydraulic works. The choice of one of them will be a function of some different factors, as economic ones or the own function of the reservoir itself.

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