



## STATION D'ESSAIS DE VIEILLISSEMENT NATUREL DE BANDOL

OUTDOOR AND ACCELERATED AGEING TESTS ON POLYMERS AND OTHER MATERIALS

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### TEST REPORT

## Comparison of behaviour of low density polyethylene films containing oxo-additive or not, when submitted to wet environment (sea water)

This test report concerns only the samples effectively submitted to test

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# FTIR TEST REPORT

**Client :** Symphony Environmental Ltd  
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**Test methodology :** Natural ageing in specific wet conditions  
Determination of carbonyl index by FTIR spectroscopy

**Samples :** Polyethylene film containing oxo-degradable additive + control

## I. TEST PROTOCOL

### 1. Samples

*Table 1 : Samples description*

Reference	Supplier	Polymer type	Oxo-additive system	Thickness (*)	Date recieved
FA6224 A	Symphony	Borealis LDPE/CLEAR	1% DG 12-08	50 µm	09/06/2015
FA6224 NA	Symphony	Borealis LDPE/CLEAR	-	50 µm	09/06/2015

(\*) micrometer measurement

These films are prepared in specific floating holders (dimensions 20 cm x 10 cm), fitted to our exposure system.

### 2. Ageing test

The test is of surface weathering on sea water, in outdoor conditions of Bandol.

A tank filled with sea water, of capacity 70 liters and height 40 centimeters is used for the experiment (the water level, decreasing by evaporation, is adjusted by adding fresh water, thus the salinity in sea water tests remains constant).

Sample holders enable the films to float on the surface of sea water throughout the test.

The UV rate received by the samples is the sun's one.

The exposure began on 19/06/2015 and lasted 82 days.

### 3. Carbonyl Index measurement

The polymer oxidation is determined by FTIR spectroscopy in transmission mode, according to ISO 10640. The spectrometer used is an Avatar 360 (Nicolet) working with the following conditions :

- Scans number : 16
- Resolution : 4 cm<sup>-1</sup>

The carbonyl index is measured one a week, as the delta of the ratio of the carbonyl peak at 1713 cm<sup>-1</sup> and the thickness of the sample in micrometers.

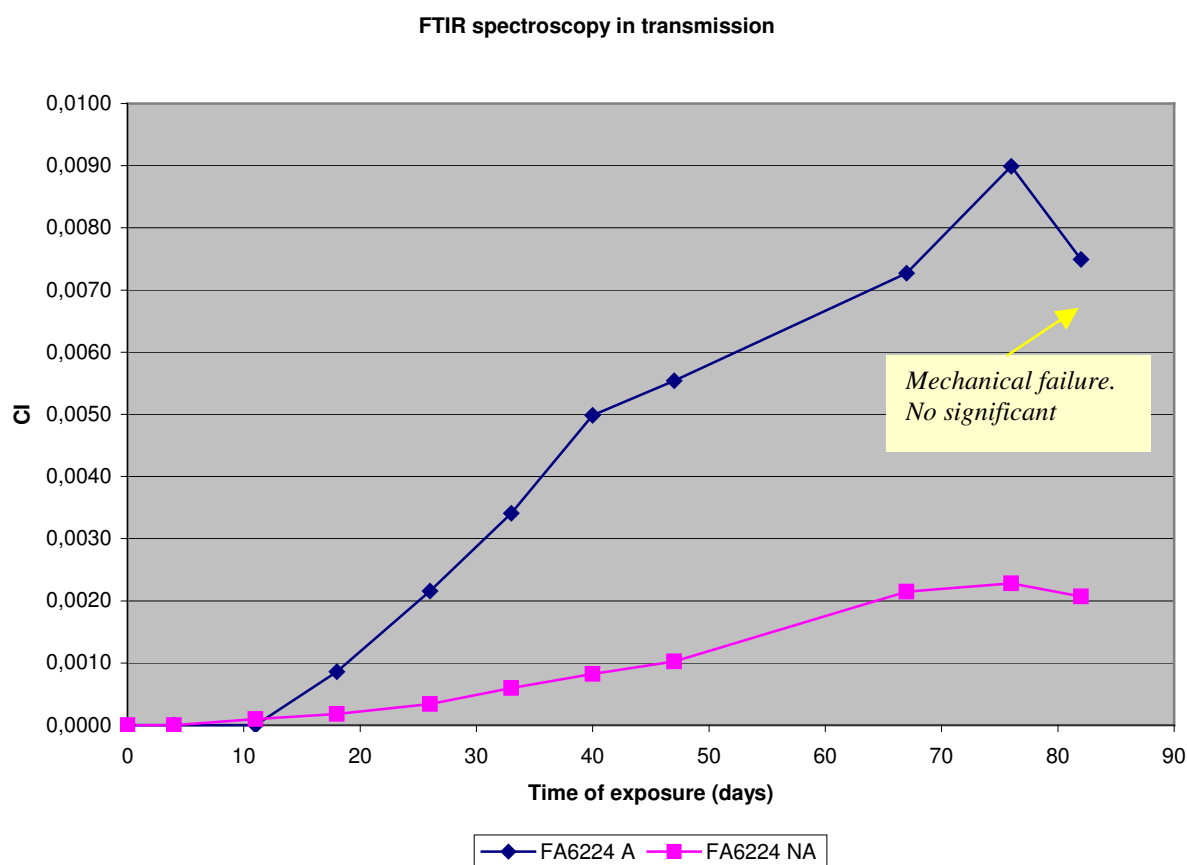
$$CI = \Delta (DO_{1713}/e)$$

*Notice that all the samples have been washed once a week before FTIR measurement.*

## II. RESULTS

Figure 1 shows the evolution of carbonyl index during natural ageing at the surface of sea water, for the two samples FA6224 A/NA.

**Figure 1 : Outdoor ageing on surface of sea water (beginning : 19/06/15)**



Induction period is similar for the two samples (11 days) but degradation kinetics are then quite different. In particular, the lineic oxidation rate of 0,001 (corresponding to 50% loss of elongation at break) is reached after 19 days by the oxo-degradable additivated film (FA6224A) and 46 days by the control one (FA6224 NA). After 76 days of surface weathering on sea water, FA6224 A presents a lineic oxidation rate of 0,009 and shows dramatic mechanical failure.

These FTIR results can be related to molecular weight determination (Smithers Rapra, CTR 58653 on date of 22/10/2015).

The comparison of molecular weight distributions, using conventional high temperature gel permeation chromatography, was performed on three samples : FA6224 A and FA6224 NA after 82 days of surface weathering on sea water in Bandol, FA6224 NA unaged.

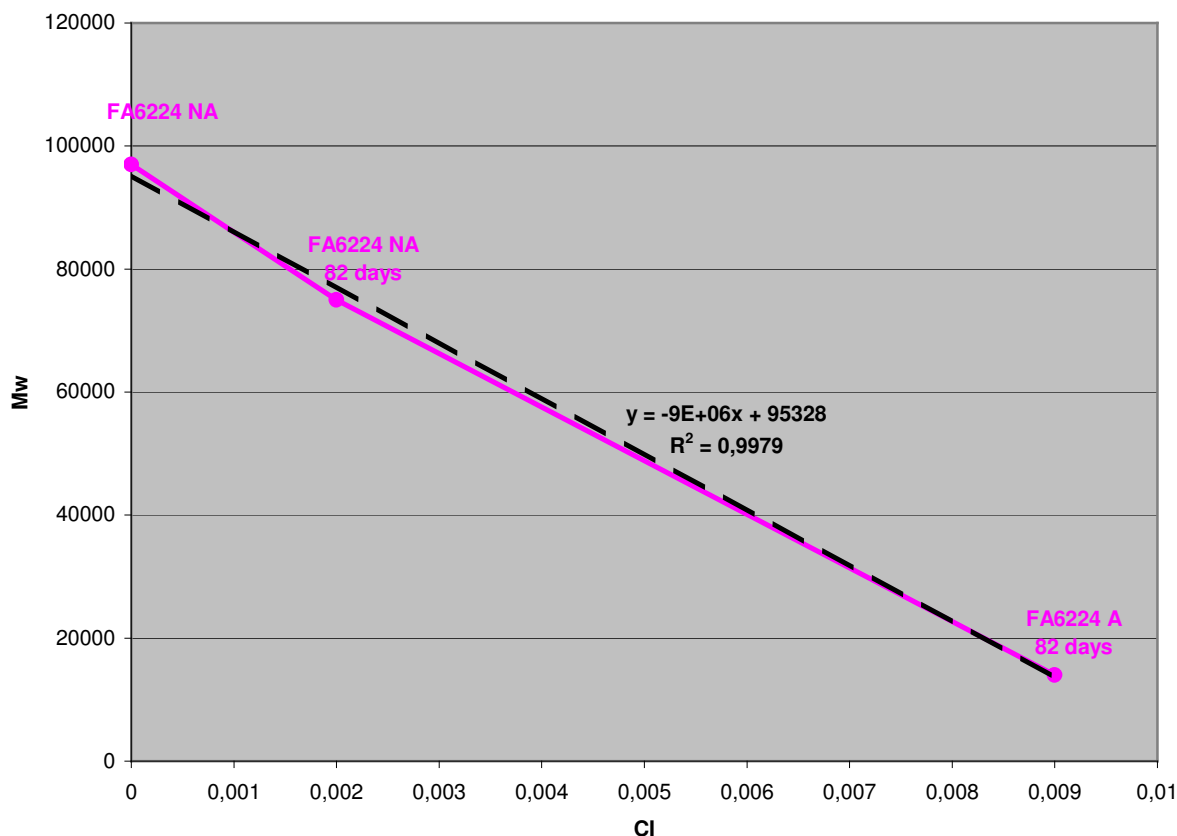
Table 2 summarizes the whole results.

**Table 2 : FTIR / GPC results**

Sample	CI	Mw	Observation
FA6224 NA (control) unaged	0,000	97000	-
FA6224 NA (control) 82 days sea water ageing	0,002	75000	-
FA6224 A (1% DG 12-08) 82 days sea water agein	0,009	14000	Mechanical failure

Figure 2 represents Molecular weight versus Carbonyl Index.

**Figure 2 : Correlation Mw / CI**



A perfect correlation exists between oxidation rate measured by FTIR in transmission and molecular weight determined by GPC.

This result, fully compatible with the theoretical mechanisms of chemical degradation, enable to validate the two methods of characterization and to put its trust in measurements of the present study.

### III. OPINION AND INTERPRETATION

FTIR analysis points out the capability of LDPE film to degrade quickly in outdoor wet environment when containing 1% DG 12-08 additive.

Compared with control one, this high oxidation rate is to be related to a lower molecular weight, representative of chain breaking reactions.

The weathering test on sea water surface, performed to point out the behaviour of samples containing prodegradant d2w<sup>®</sup> additive in wet environments (films and bags accidentally released in oceans or lakes), points out very promising behaviours.

Assuming that there is correlation between oxidation rate and elongation at break, film FA6224 A would present a 50% loss of mechanical properties in three weeks, and a total loss in three months, when exposed in summer period in Mediterranean climate.