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TEST REPORT 3574:

SMART SEAL IND. E COM. ARTEFATOS PLÁSTICOS LTDA

Registered in England Number 2967867 - Address as above.
A wholly owned subsidiary of Symphony Environmental Technologies Plc.

SYMPHONY ENVIRONMENTAL LTD

CONTROLLED-LIFE PLASTIC TECHNOLOGY

1.0 AIMS

To compare the oxo-degradable response of a polypropylene security seal containing d_2w prodegradant additive with respect to a non degradable control sample by means of accelerated ageing.

2.0 CONCLUSIONS

2.1 Degradation - Successive accelerated UV and thermal ageing

The results of the successive accelerated UV and thermal ageing test demonstrate that the sample containing the d_2w prodegradant additive has degraded to a greater extent than the control sample.

The sample containing the additive demonstrates a larger change in carbonyl index measurement than the respective control sample at the conclusion of the test (Figure 1). This result is consistent with the sample containing the prodegradant additive being in a more advanced state of degradation.

The sample containing d_2w reached a carbonyl index value of 0.292 after 720 hours ageing (48 hours UV ageing directly followed by 672 hours thermal ageing) where as the control sample without additive has demonstrated no significant increase in carbonyl index.

This result is consistent with inclusion of d_2w promoting degradation in the sample.

2.2 Shelf life – Accelerated thermal ageing

The sample containing the additive and the control sample demonstrated no significant increase in carbonyl index measurement at the conclusion of the test (Figure 2). This result is consistent with the samples having undergone no significant degradation.

The absence of degradation of the samples confirms that the polypropylene is intrinsically stable to thermal ageing. This is consistent with the product having a useable fit for purpose shelf–life in storage conditions away from prolonged exposure to UV light and at an average temperature not exceeding 30°C.

3.0 SAMPLE DESCRIPTION

Supplier name:	SMART SEAL IND. E COM. ARTEFATOS PLÁSTICOS LTDA
Polymer type:	polypropylene
Samples provided:	A) Green security seal with d_2w oxo-biodegradable additive
	B) Green control security seal without additive
Additive system:	93389

4.0 TEST PROTOCOL

4.1 Successive accelerated UV and thermal ageing

This method involves subjecting the substrate to 48 hours accelerated UV pre-ageing before accelerated thermal ageing and monitoring degradation as function of ageing time via changes in the carbonyl index as determined by FT-IR (Fourier Transform Infra Red) spectroscopy.

4.2 Thermal ageing

The method involves subjecting the substrate to accelerated thermal ageing and monitoring degradation as function of ageing time via changes in the carbonyl index as determined by FT-IR (Fourier Transform Infra Red) spectroscopy.

5.0 TEST METHODOLOGY

5.1 Accelerated Fluorescent UV Ageing

Samples were placed in a sample holder, in which the seal is sandwiched between two metal plates (35 x 90 mm) with four exposure windows, and exposed to ultraviolet radiation in a Q Panel QUV/se test apparatus fitted with UVA 340 lamps, in general accordance with ASTM D5208. A black panel temperature of 50ºC was used in conjunction with a humid environment. The irradiance of the lamps was 0.78 $W/m^2/nm$. Samples of the additive and control materials were withdrawn every 48 hours and their carbonyl index determined by FT-IR spectroscopy.

5.2 Accelerated Thermal Ageing

Thermal ageing of the samples was carried out in a Memmert UFE 600 fan assisted oven at a temperature of 70ºC in general accordance with ASTM D5510 Procedure B: Forced Ventilation Oven. Samples of the additive and control materials were withdrawn every 96 hours and their carbonyl index determined by FT-IR spectroscopy.

5.3 Carbonyl Index Measurement

The carbonyl index of the samples was determined by Fourier Transform infrared spectroscopy (FT-IR). Reflection spectra at the surface of the samples were obtained by a Smart Orbit diamond ATR attachment connected to a Thermo Electron Nicolett FTIR. The carbonyl index was measured as the ratio of the carbonyl peak at 1718 cm^{-1} and the carbon –hydrogen absorption peak at 2920 cm^{-1}.

Measuring changes in carbonyl index is a useful technique for monitoring the accumulation of oxidation degradation reaction by-products. These include species such as carbonyl compounds (aldehydes, ketones etc.) whose presence are all indicative of degradation. Monitoring the growth of these species as a function of time provides a good indication of the relative rate of degradation of the material.

For rigid samples such as bottles and profile, degradation proceeds via the surface, since it is controlled by the rate of oxygen diffusion into the polymer bulk. The surface layers will first be degraded and they will be removed by the action of environmental stimuli and stresses such as wind and rain. This will have the effect of exposing fresher material in the layers underneath which will initially be less degraded. However once exposed to oxygen the degradation of this layer will be facilitated. This phenomenon is well established and was reported by Cunliffe.[1]

Degradation for rigid articles will therefore proceed in a cyclical manner and this will be reflected in the profile of the carbonyl index observed. The graph would normally yield periods of increasing carbonyl index during degradation but will revert to lower values when the less degraded underlying layers are exposed.

5.4 XRF Spectroscopy

The anticipated presence of the prodegradant additive in each sample was confirmed by energy-dispersive X-ray florescence (ED-XRF) spectroscopy using a Bruker S2 Ranger A20-X10 bench top spectrometer against reference samples produced by Symphony.

The XRF spectrum of each sample was determined in air over 120 s with 40.00 kV, 250 mA X-ray source and 500.0 µm aluminum filter.

[1] *Cunliffe, A.V.* and *Davis*, A., "*Photo-Oxidation of Thick Polymer Samples—Part II: The Influence* of *Oxygen Diffusion on the Natural and Artificial Weathering of Polyolefins*," Polym. Degrad. Stab., 4, 17 (1982)

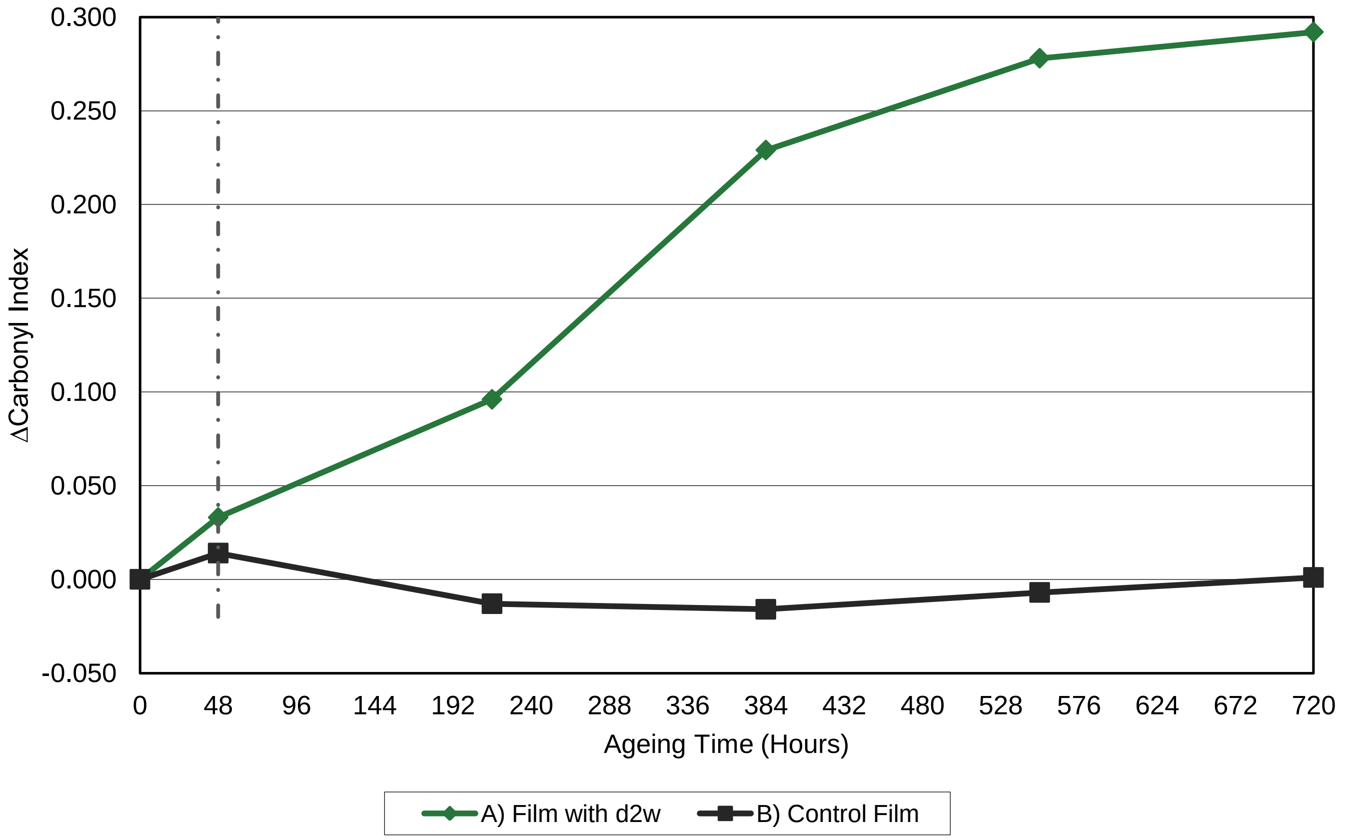
6.0 RESULTS

6.1 Successive accelerated UV and thermal ageing

Table 1: Accelerated Successive UV and thermal ageing

Sample	Δ Carbonyl Index					
	QUV Ageing		Thermal Ageing			
	0 Hrs	48 Hrs	216 Hrs	384 Hrs	552 Hrs	720 Hrs
A) Seal with d_2w	0.000	0.033	0.096	0.229	0.278	0.292
B) Control Seal	0.000	0.014	-0.013	-0.016	-0.007	0.001

Figure 1: Sucessive Accelerated UV and Thermal Ageing

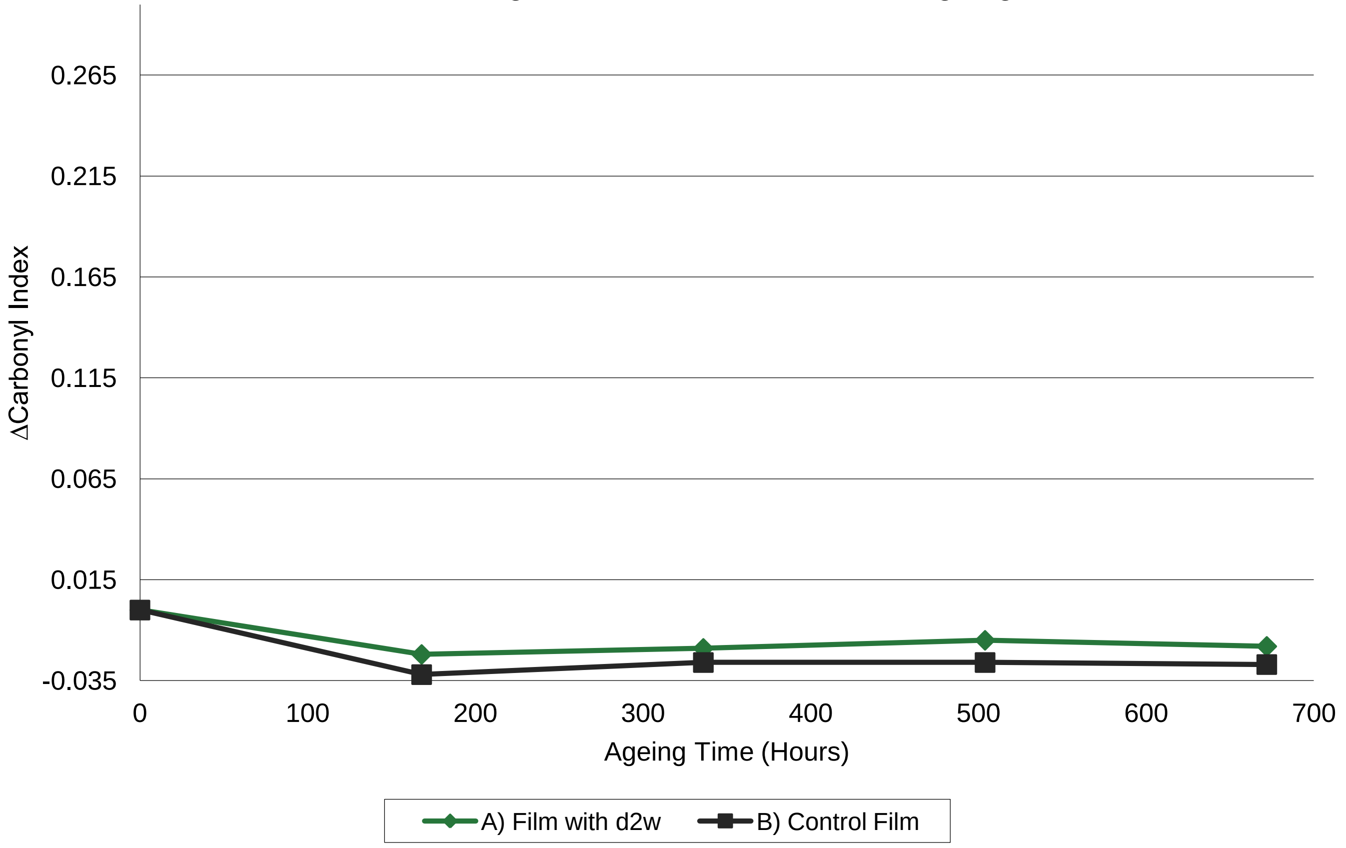


6.2 Accelerated Thermal Ageing

Table 2: Accelerated Thermal Ageing

Sample	Δ Carbonyl Index				
	0 Hrs	168 Hrs	336 Hrs	504 Hrs	672 Hrs
A) Seal with d_2w	0.000	-0.022	-0.019	-0.015	-0.018
B) Control Seal	0.000	-0.032	-0.026	-0.026	-0.027

Figure 1: Accelerated Thermal Ageing



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