



Symphony Environmental Ltd
6 Elstree Gate, Elstree Way
Borehamwood
Hertfordshire WD6 1JD
England

+44 (0)20 8207 5900 Telephone
+44 (0)20 8207 7632 Facsimile
www.d2w.net
technical@d2w.net

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TEST REPORT: 6793

Sunshine Non Woven Fabric Co Ltd / Green bags Ltd



FM37939

EMS567450

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 evaluate the oxo-degradation response of PP non-woven bags made with a d₂w prodegradant additive by means of accelerated ageing in the laboratory, with comparison to an equivalent non-degradable control sample.

2.0 CONCLUSIONS

2.1 Prodegradant Catalyst Content

Evaluation of the XRF spectrum of each product sample and comparison with a calibration of reference samples has confirmed that the test sample does contain the d₂w prodegradant catalyst at the correct level; which is consistent with 1% addition of the additive masterbatch (*Table 2*).

No prodegradant catalyst was detected in the control sample.

2.2 Shelf-Life Stability

During the accelerated thermal ageing test cycle, the test sample containing the additive demonstrated no significant increase in carbonyl optical density measurement at the conclusion of the test (*Figure 1*). This result is consistent with the sample having undergone no significant degradation.

This conclusion is confirmed by observation: at the end of the accelerated thermal ageing cycle the test sample remains largely intact (*Picture 1*).

The absence of degradation confirms that the test sample is stable to thermal ageing. This is consistent with the product made with d₂w maintaining a useable fit for purpose shelf-life in storage conditions while protected from extended exposure to UV or Sunlight and excessive heat, not exceeding an average temperature of 30°C.

2.3 Initiated Thermal Degradation

Initial UV exposure was completed after 48 hours without significant evidence of degradation in all samples. Once transferred to the oven for accelerated thermal ageing, the test sample containing the additive demonstrated a significant increase in carbonyl optical density measurement which progressed throughout the remainder of test (*Figure 2*). This is consistent with the test sample having undergone significant degradation.

The non-degradable control sample demonstrated no significant increase in carbonyl optical density throughout the same ageing cycle (*Figure 2*). This result is consistent with the sample having undergone no significant degradation.

This conclusion is confirmed by observation: at the end of the UV and subsequent thermal ageing cycle the test sample show signs of physical embrittlement whilst the control sample remains largely intact (*Picture 2*).

The observation of degradation exclusively in the test sample with d₂w is consistent with the additive promoting degradation of the product at the end of its useful life or following exposure in the environment as litter, even if it is subsequently occluded or removed from sunlight.

3.0 SAMPLE DESCRIPTION

Report Prepared for: Green Bags Ltd
Product Manufacturer: Sunshine Non Woven Fabric Co Ltd
Polymer type: PP
Samples: A) 1% d₂w 93190
 B) Control
Date samples received : 31/05/2016

4.0 TEST PROTOCOL

4.1 Prodegradant Determination

The presence of the prodegradant additive in each sample is confirmed by determination of the prodegradant catalyst metal cation in the additive active ingredient by energy-dispersive X-ray fluorescence (XRF) spectroscopy.

4.2 Shelf-Life Stability

Stability of each test samples in storage conditions and during subsequent useful life of product is confirmed by monitoring degradation during accelerated thermal ageing. The storage life of the test sample is confirmed by a period under test where no significant evidence of degradation of the material is observed.

4.3 Initiated Thermal Degradation

The degradation response of a product at the end of its useful life or in the dark initiated by previous exposure to sunlight when the product was discarded as litter is demonstrated by monitoring degradation during accelerated thermal ageing following brief initial exposure to UV light for a period which is sufficient to deplete the primary phenolic stabilisers that are consumed over time during normal storage and use or rapidly destroyed by sunlight if the product is exposed to the environment as litter. The initial UV ageing cycle is not intended to bring about the onset of photo-oxidation of the test sample.

4.4 Monitoring Degradation

Throughout each test the infrared (IR) spectrum of the ageing sample is evaluated for evidence of carbonyl species (aldehydes, ketones, carboxylic acids etc.) which are reaction by-products of the oxidative degradation process and as such their presence and accumulation is indicative of ongoing degradation.

The extent and progression of degradation is monitored by calculation of the carbonyl optical density from the FT-IR spectrum of each sample. A greater magnitude in the carbonyl optical density value is consistent with greater extent of degradation.

5.0 TEST METHODOLOGY

5.1 Thickness Determination

The thickness of the sample material is determined before ageing using a digital electronic micrometer in general accordance with ISO 4593:1993 Plastics -Part 6: Dimensional properties - Method 630A: Determination of thickness by mechanical scanning of flexible sheet.

5.2 XRF Spectroscopy

The XRF spectrum of each unaged sample is recorded using a Bruker S2 Ranger A20-X10 bench top spectrometer in air over 120 s with 40.00 kV, 250 mA X-ray source and a 500.0 μm aluminum filter. Film samples were prepared in 38 mm diameter HDPE XRF sample cups and the total thickness made up to ~ 200 μm with 36 mm discs cut from the bulk material using a James Heal 230/10 sample cutter.

The concentration of the prodegradant catalyst metal cation is quantified by correlation with a calibration of known reference samples.

5.3 Accelerated ageing

5.3.1 Sample Preparation/holders

35 x 90 mm film samples were prepared in a sample holder in which the film is secured between two metal plates with four exposure windows.

5.3.2 Accelerated Thermal Ageing

Thermal ageing of the samples was carried out in a Memmert UFE 600 fan assisted oven at a temperature of 70°C in general accordance with ASTM D5510 Procedure B: Forced Ventilation Oven.

5.3.3 Accelerated Fluorescent UV Ageing

Samples were 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 was 50°C and the irradiance of the lamps was set to 0.78 $\text{W}/\text{m}^2/\text{nm}$.

5.4 FT-IR Spectroscopy

Before the commencement of ageing and at each time-point during the ageing cycle, the infrared (IR) spectrum of each sample was taken in accordance with ISO 10640 using a Nicolet iS10 fourier transform infrared (FT-IR) Spectrometer and the absorbance at 1714 cm^{-1} was recorded.

5.5 Carbonyl Optical Density Calculation

The extent of oxidation is reported as the carbonyl optical density, a function of the net increase in infrared absorbance during ageing, per unit of the path length (sample thickness):

$$\text{Equation 1: IR Carbonyl Optical Density } (\mu\text{m}^{-1}) = \frac{\Delta\text{Abs}_{1714 \text{ cm}^{-1}}}{\text{Thickness } (\mu\text{m})}$$

6.0 RESULTS

6.1 Thickness

Table 1: Sample Thickness

Sample	Thickness (μm)
A) 1% d ₂ w 93190	395.8 μm
B) Control	383.3 μm

6.2 Prodegradant Catalyst Content – XRF Spectroscopy

Table 2: Summary of Prodegradant Additive Content

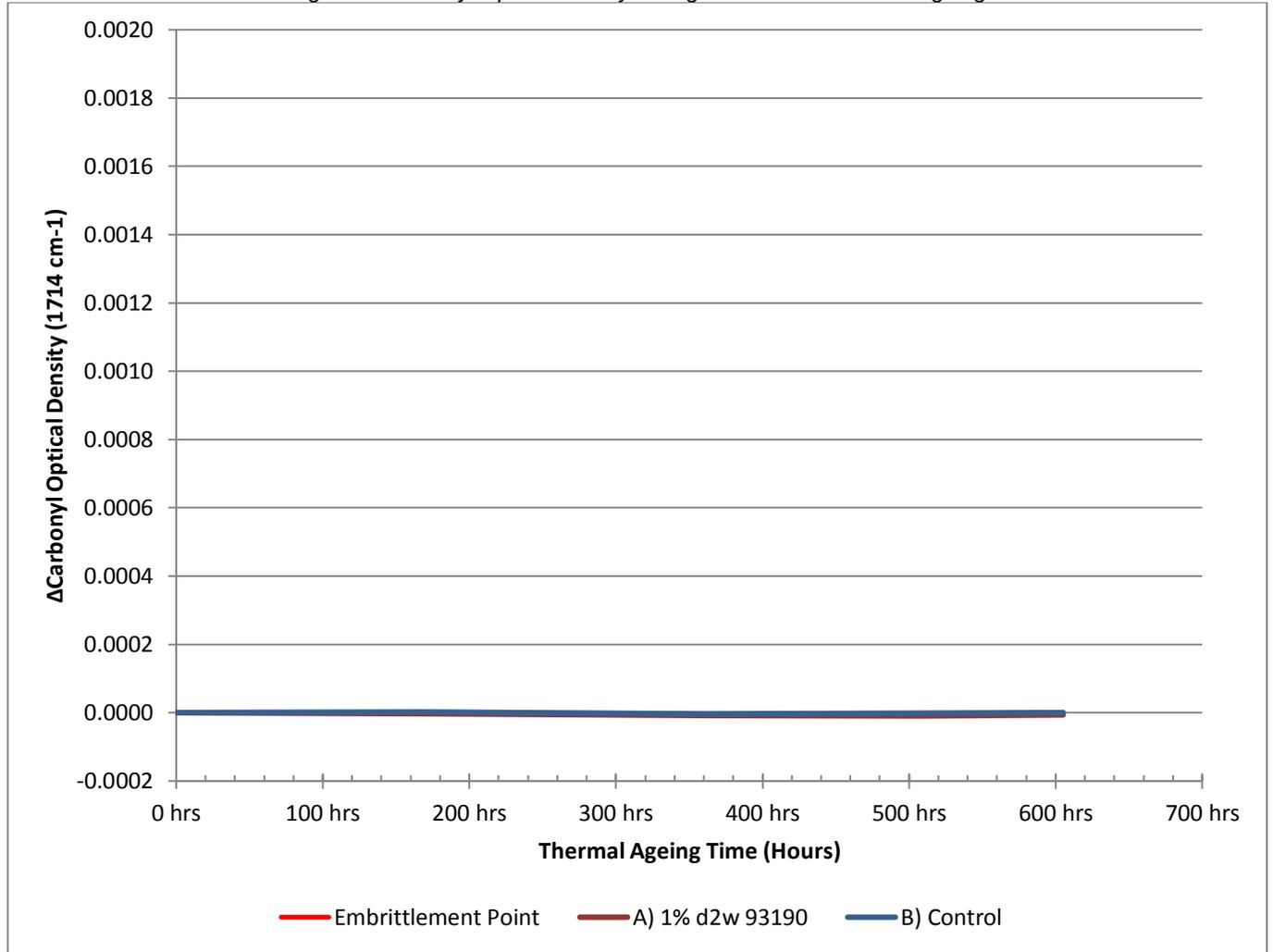
Sample	Prodegradant Content
A) 1% d ₂ w 93190	d ₂ w prodegradant catalyst present at the correct level
B) Control	d ₂ w prodegradant catalyst absent

6.3 Oxidation during Accelerated Thermal Ageing

Table 3: Carbonyl Optical Density during Accelerated Thermal Ageing

Sample	16/06/2016	23/06/2016	01/07/2016	07/07/2016	11/07/2016
	12:03	12:44	14:14	16:35	17:11
	Δ Carbonyl Optical Density (1714 cm ⁻¹)				
	0 hrs	169 hrs	362 hrs	509 hrs	605 hrs
A) 1% d2w 93190	0.0000	0.0000	0.0000	0.0000	0.0000
B) Control	0.0000	0.0000	0.0000	0.0000	0.0000

Figure 1: Carbonyl Optical Density during Accelerated Thermal Ageing



Picture 1: Test samples after accelerated thermal ageing

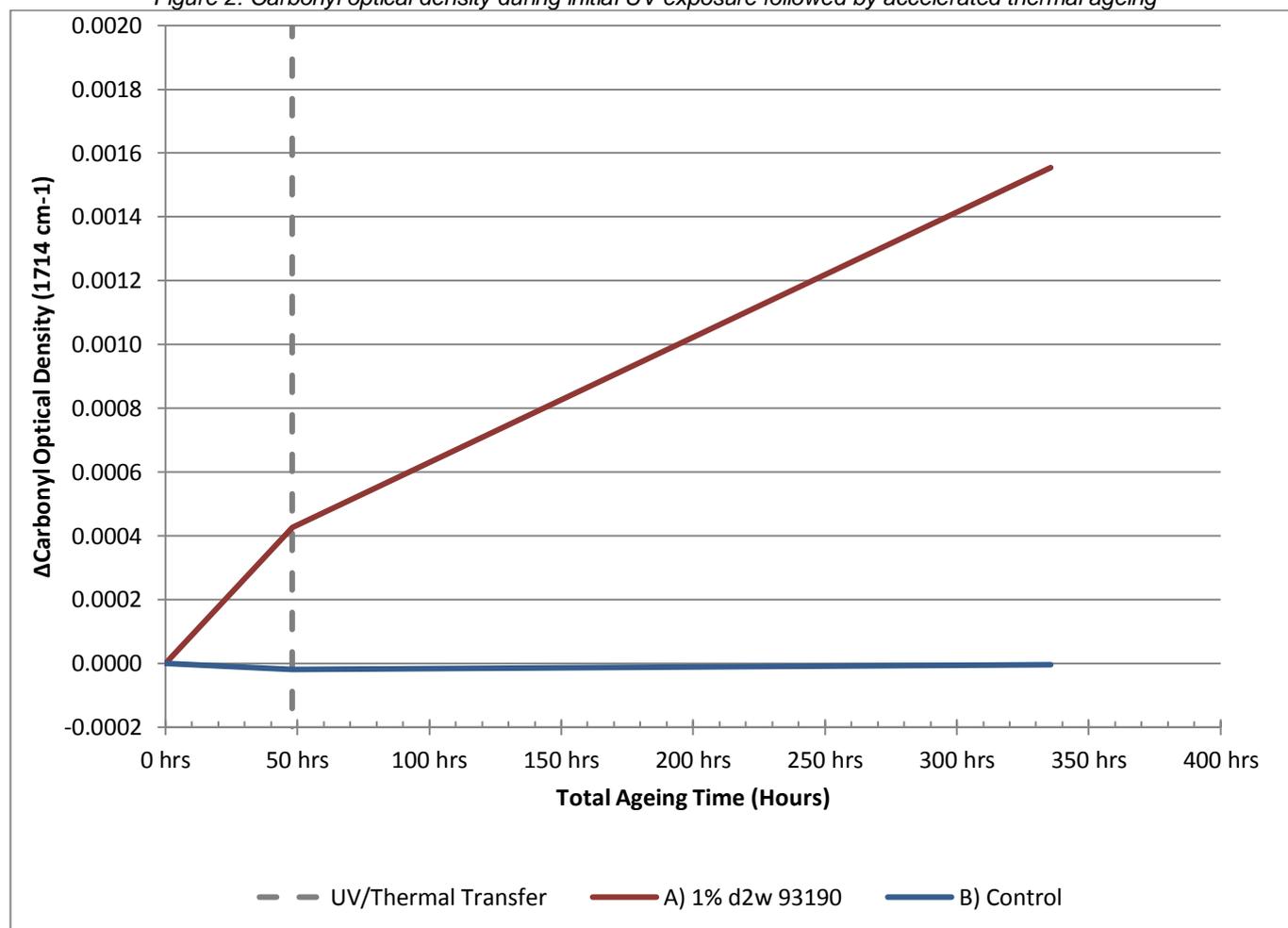


6.4 Oxidation During *Initial UV Exposure Followed by Accelerated Thermal Ageing*

Table 4: Carbonyl optical density during initial UV exposure followed by accelerated thermal ageing

Sample	22/06/2016	24/06/2016	06/07/2016
	14:48	15:28	15:01
	Δ Carbonyl Optical Density (1714 cm^{-1})		
	UV		Thermal
	0 hrs	48 hrs	336 hrs
A) 1% d2w 93190	0.0000	0.0004	0.0016
B) Control	0.0000	0.0000	0.0000

Figure 2: Carbonyl optical density during initial UV exposure followed by accelerated thermal ageing



Picture 2: Test samples after initial UV exposure and accelerated thermal ageing



Author:	Review:
Michael Skehan Laboratory Technician	Perry Higgs BSc (Hons), AUS, AMRSC Technical Executive

The information presented in this report is based on the material actually tested. Performance of finished product made with d₂w additive depends on the heat, light, stress and other conditions to which the finished product is exposed in the environment.