Symphony environmental
product testing laboratory

Date: 8 January 2021

ANALISIS BOLSA ROLLO Y CORTADA BIODEGRADABLE

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ANALISIS BOLSA FUELLE BIODEGRADABLE

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Date: 8 January 2021

Report: 11896/002

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Test Report:

- **Elemental Content Analysis**
    - Determination of Prodegradant Catalyst
- **Accelerated Ageing Studies:**
    - Thermal Stability
    - Thermal Degradation, Following Initial UV Exposure

Test Reference:	**11896 Polybags Extruidos de Leon S.A de C.V**
Product Tested:	**HDPE Natural Bags**
Prepared for:	**Polybags Extruidos de Leon S.A de C.V**
Prepared by:	**Jacob Cummings**, Scientist

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TEST REPORT

1.0 AIMS

To evaluate the presence of prodegradant catalysts in the HDPE natural bags samples by means of determination of the prodegradant catalyst metal cation by x-ray fluorescence (XRF) spectroscopy; and to evaluate the stability and degradation behaviour of the HDPE natural bags samples by means of accelerated laboratory ageing techniques, while monitoring extent of polymer oxidation by infrared (IR) spectroscopy as a function of time.

2.0 SAMPLE DETAILS

Samples Provided by:	Polybags Extruidos de Leon S.A de C.V
Product Type:	HDPE Natural Bags
Date Received:	07/09/2020

Table 1: Sample Details

SEPTL Sample ID	Sample Reference	Polymer Type	Prodegradant Additive
11896 A	Bolsa transparente control	HDPE	None
11896 B	Bolsa transparente d2w	HDPE	1% d_2w 93224

Table 1 is based solely on information provided by the customer.

3.0 RESULTS & CONCLUSIONS

3.1 Determination of Prodegradant Catalyst

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

The prodegradant catalyst content of the HDPE natural bags samples, as determined by x-ray fluorescence (XRF) spectroscopy, is shown in Table 2.

Table 2: Determination of Prodegradant Catalyst

Sample ID	Spectrum Ref.	Prodegradant Content
11896 A	2710/52293	Not Detected
11896 B	2710/52292	Prodegradant Present, Effective Concentration

3.2 Accelerated Ageing - Thermal Stability

The real-time stability of polymer products stored at ambient temperatures and protected from extended exposure to sunlight is evaluated over shorter period of time in the laboratory by monitoring degradation during thermal ageing at elevated temperatures according to ASTM D5510.

Polymer degradation is evaluated by determination of polymer oxidation by infrared spectroscopy. The increase in magnitude of features of the infra-red spectra corresponding to the carbonyl products of polymer oxidation, is recorded as carbonyl optical density. The period of accelerated thermal ageing where no significant increase in carbonyl optical density (not exceeding 0.0010) is observed is considered to be representative of the real-time period of product stability in storage conditions.

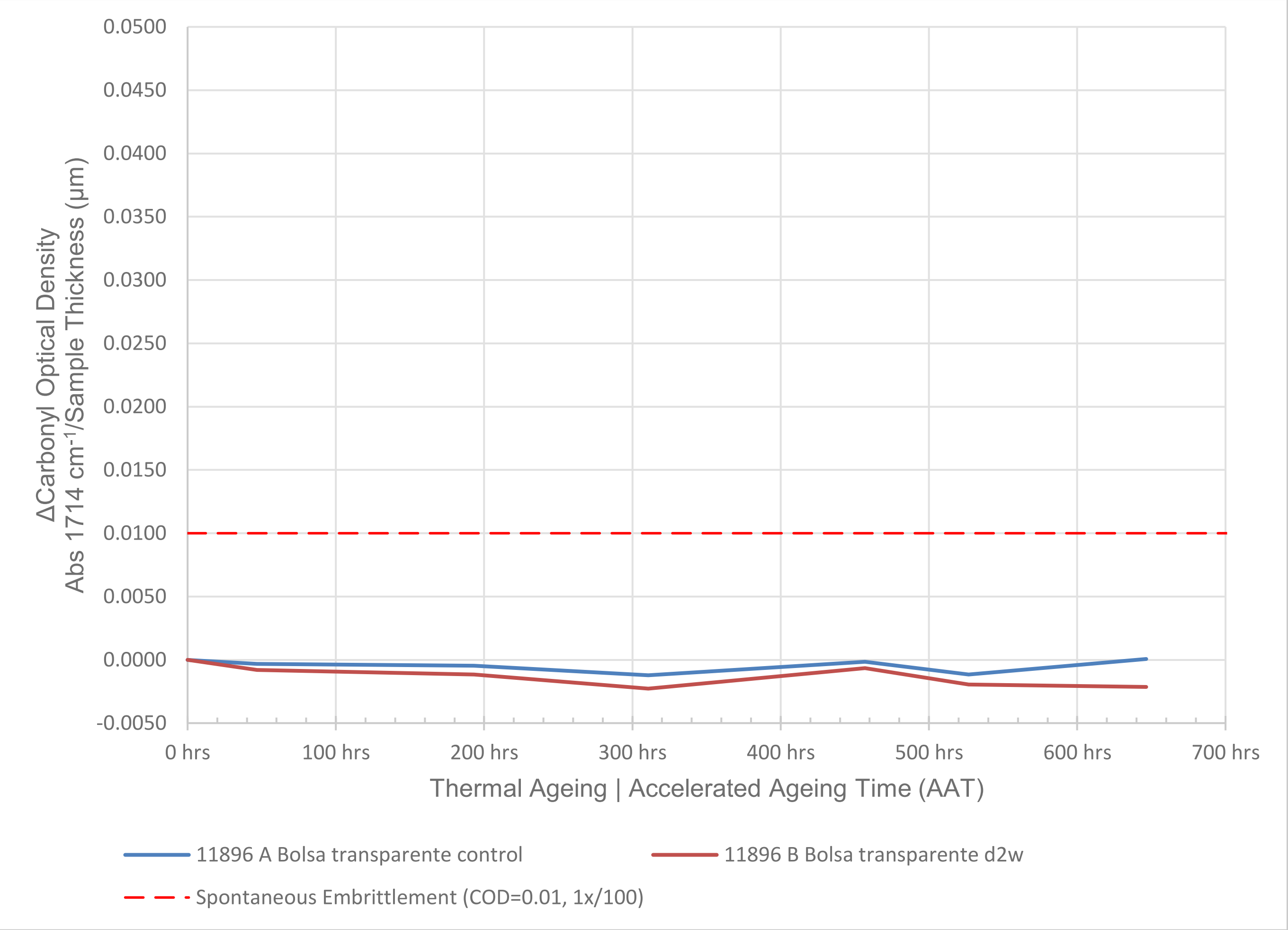
Samples 11896 A & B demonstrated no significant oxidation during the accelerated stability test. The samples demonstrated an average carbonyl optical density value which did not exceed 0.0001 up to 647 hours accelerated ageing (Table 3). This result is consistent with the samples having undergone no significant degradation.

The absence of degradation in the samples for the duration of this test confirms that the products are stable in dark conditions, at ambient temperatures for an initial life period corresponding to the products life.

Table 3: Carbonyl Optical Density during Accelerated Thermal Ageing

Accelerated Ageing Time (AAT)	**ΔCarbonyl Optical Density (IR Abs 1714 cm^{-1}/Thickness)**	
	11896 A Bolsa transparente control	**11896 B Bolsa transparente d2w**
0 hrs	0.0000	0.0000
47 hrs	-0.0003	-0.0008
193 hrs	-0.0005	-0.0012
311 hrs	-0.0012	-0.0023
457 hrs	-0.0002	-0.0007
527 hrs	-0.0012	-0.0020
647 hrs	0.0001	-0.0021

Figure 1: Carbonyl Optical Density during Accelerated Thermal Ageing



3.3 Accelerated Ageing - Thermal Degradation, Following Initial UV Exposure

Polymer degradation in dark conditions, following initial exposure to sunlight is evaluated in a shorter period of time in the laboratory by monitoring degradation during accelerated fluorescent UV ageing in accordance with ASTM D5208 followed by thermal ageing at elevated temperatures according to ASTM D5510.

Polymer degradation is evaluated by determination of polymer oxidation by infrared spectroscopy. The increase in magnitude of features of the infra-red spectra corresponding to the carbonyl products of polymer oxidation, is recorded as carbonyl optical density. A carbonyl optical density value of 0.0100 is considered indicative of advanced degradation as such to bring about spontaneous embrittlement. The rate of degradation is evaluated and compared by monitoring carbonyl optical density as a function of ageing time.

The samples were initially exposed to constant fluorescent UV ageing for a period of 48 hours. The samples were then exposed to accelerated thermal ageing in dark conditions. During accelerated ageing, test sample 11896 B demonstrated a significant increase in **carbonyl optical density** (Table 4**).**

Sample 11896 B demonstrated a carbonyl optical density value of 0.0127 in 267 hours exposure (inclusive of 48 hours initial fluorescent UV exposure). This **is consistent with this sample having undergone significant degradation.**

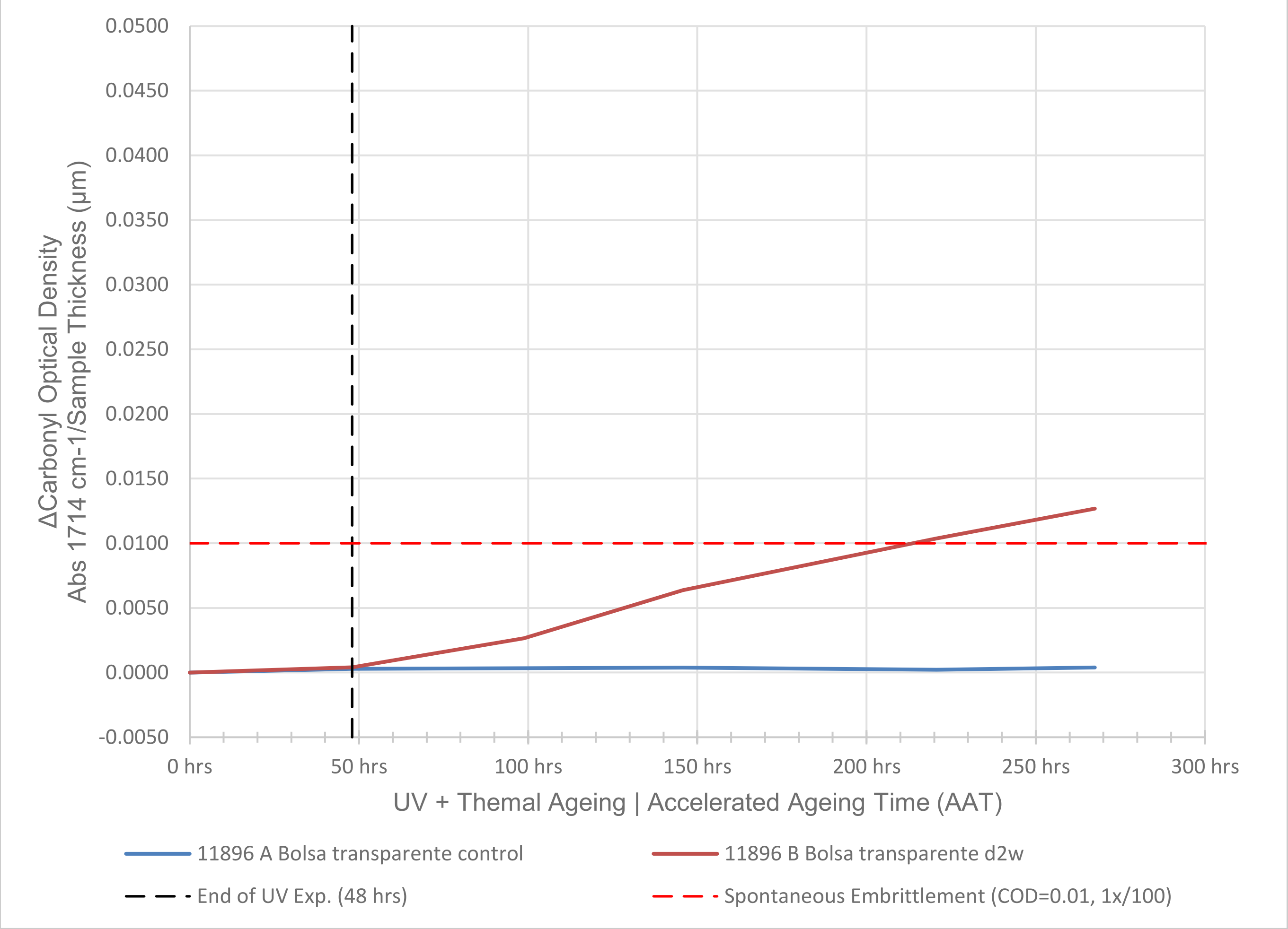
Sample 11896 A demonstrated no significant increase in carbonyl optical density throughout the test (Table 4). This result is consistent with this sample having undergone no significant degradation.

The observation of degradation exclusively in the sample which contains the prodegradant additive is consistent with the additive promoting degradation of the product in dark conditions, following initial exposure to sunlight in the environment as litter.

Table 4: Carbonyl Optical Density during Accelerated Thermal Ageing, Following Initial UV Exposure

Accelerated Ageing Time (AAT)	**Exposure**	**ΔCarbonyl Optical Density (IR Abs 1714 cm^{-1}/Thickness)**	
		11896 A Bolsa transparente control	**11896 B Bolsa transparente d2w**
0 hrs	UV	0.0000	0.0000
48 hrs		0.0003	0.0004
99 hrs	Thermal	0.0003	0.0027
146 hrs		0.0004	0.0064
221 hrs		0.0002	0.0104
267 hrs		0.0004	0.0127

Figure 2: Carbonyl Optical Density during Accelerated Thermal Ageing, Following Initial UV Exposure



4.0 TEST METHODOLOGY

4.1 XRF Spectroscopy

4.1.1 Determination of Prodegradant Catalyst

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 aluminium 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 validated reference samples.

4.2 Accelerated Ageing Exposure

4.2.1 Sample Preparation/Holders

35 x 90 mm film samples were cut using a scalpel and secured in a sample holder with four exposure windows.

4.2.2 Thickness Determination

The thickness of the sample material is determined before ageing using a digital electronic micrometer, in not less than four random locations across the test sample and the average value recorded.

4.2.3 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 accordance with ASTM D5510 Procedure B: Forced Ventilation Oven.

4.2.3.1 Thermal Stability Real Time Exposure Prediction

The real time period (RT) at ambient temperature (T_{RT}) represented by accelerated ageing time (AAT) at elevated accelerated ageing temperature (T_{AA}) is predicted in accordance with ASTM F1980 using the relationship outlined in Equation 1.

The calculation is performed using an anticipated average storage exposure temperature (T_{RT}) of 30°C and a conservative ageing factor (Q_{10}) of 2.

$$\text{Equation 1: } AAT = \frac{RT}{Q_{10}{}^{[(T_{AA}-T_{RT})/10]}}$$

4.2.4 Accelerated Fluorescent UV Ageing

Samples were exposed to ultraviolet radiation in accordance with ASTM D5208 in a Q Panel QUV/se test apparatus fitted with UVA 340 lamps, set to a black panel temperature of 50ºC and irradiance of 0.78 W/m^2/nm @ 340 nm.

4.3 FT-IR Spectroscopy

Infrared (IR) spectra is recorded in accordance with ISO 10640 using a Nicolet iS10 fourier transform infrared (FT-IR) Spectrometer and the absorbance at 1714 cm^{-1} was recorded.

4.3.1 Carbonyl Optical Density Calculation

The extent of oxidation is reported as the carbonyl optical density, a function of the net increase in infrared absorbance (ΔIR Abs) at 1714cm^{-1} during ageing, per unit of the path length (sample thickness in μm):

Equation 2: $$\textbf{Carbonyl Optical Density} \ \frac{\Delta \textbf{IR Abs}_{1714\ \textbf{cm}^{-1}}}{\textbf{Thickness}\ (\mu\textbf{m})}$$

5.0 REPORT HISTORY

Date	Report Ref.	Comments
Previous Versions		
08/01/2021	11896/001	Prodegradant Content Report and Edited Product Description
Current Version - This report supersedes all previous versions		
08/01/2021	11896/002	Added Accelerated Ageing Data and Edited Product Description

6.0 REPORT AUTHORISATION

Author:

Jacob Cummings
Scientist

"The information presented in this report is based on the material actually tested. Performance of finished product made with d$_2$w® additive depends on the conditions under which and length of time for which the additive is stored and on the method of manufacture of the finished product and the heat, light, stress and other conditions to which the finished product is exposed. Nothing in this report constitutes or implies a license to use Symphony's intellectual property".

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ANALISIS BOLSA FUELLE BIODEGRADABLE

Date: 7 September 2020

Report: 11886/001

Test Report:

- **Elemental Content Analysis**
    - Determination of Prodegradant Catalyst

Test Reference:	**11886 Polybags Extruidos de Leon S.A de C.V**
Product Tested:	**HDPE T-Shirt Bags**
Prepared for:	**Polybags Extruidos de Leon S.A de C.V**
Prepared by:	**Jacob Cummings**, Scientist

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TEST REPORT

1.0 AIMS

To evaluate the presence of prodegradant catalysts in the HDPE t-shirt bags samples by means of determination of the prodegradant catalyst metal cation by x-ray fluorescence (XRF) spectroscopy.

2.0 SAMPLE DETAILS

Samples Provided by:	Polybags Extruidos de Leon S.A de C.V
Product Type:	HDPE T-Shirt Bags
Date Received:	01/09/2020

Table 1: Sample Details

SEPTL Sample ID	Sample Reference	Polymer Type	Prodegradant Additive
11886 A	Bolsa camiseta control	HDPE	None
11886 B	Bolsa camiseta d2w	HDPE	1% d_2w 93224

Table 1 is based solely on information provided by the customer.

3.0 RESULTS & CONCLUSIONS

3.1 Determination of Prodegradant Catalyst

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

The prodegradant catalyst content of the HDPE t-shirt bags samples, as determined by x-ray fluorescence (XRF) spectroscopy, is shown in Table 2.

Table 2: Determination of Prodegradant Catalyst

Sample ID	Spectrum Ref.	Prodegradant Content
11886 A	2710/52289	Not Detected
11886 B	2710/52290	Prodegradant Present, Effective Concentration

4.0 TEST METHODOLOGY

4.1 XRF Spectroscopy

4.1.1 Determination of Prodegradant Catalyst

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 aluminium 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 validated reference samples.

5.0 REPORT HISTORY

Date	Report Ref.	Comments
Previous Versions		
04/09/2020	11886/001	Prodegradant Content Report
Current Version - This report supersedes all previous versions		
07/09/2020	11886/001	Prodegradant Content Report

6.0 REPORT AUTHORISATION

Author:

Jacob Cummings
Scientist

"The information presented in this report is based on the material actually tested. Performance of finished product made with $d_2w^{®}$ additive depends on the conditions under which and length of time for which the additive is stored and on the method of manufacture of the finished product and the heat, light, stress and other conditions to which the finished product is exposed. Nothing in this report constitutes or implies a license to use Symphony's intellectual property".