### **OXO-BIODEGRADATION REPORT**

November 2015

Project: 1502005

## **TEST RESULTS**

### TESTING GUIDELINES

- ASTM D6954-04 Standard Guide for Exposing and Testing Plastics that Degrade in the Environment
- ASTM D5510-94 (2001) Standard Practice for Heat Aging of Oxidatively Degradable Plastics.
- ASTM D5208-01
  Standard Practice
  for UV Exposure.
- ASTM D3826-98 (2001) Standard Practice for Determining Degradation End Point.
- ASTM D882-09 Standard Test Method for Tensile Properties.

Report Author:

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Our laboratory received 4 sets of samples from Pet Pickups to determine the degradation properties of the material. The samples were cut to specimen sizes compatible with our test equipment and placed in accelerated storage conditions (ASTM D5510 and ASTM 5208).

In addition to the physical property testing, we conducted FT-IR analysis at the most relevant data points to chemically look for the presence of oxidation (carbonyl C=O groups) which are highlighted in the FTIR data (see report addendum for further details on page 10). Without the presence of carbonyl groups, a plastic product that has degraded will not be able to biodegrade. Once the material becomes brittle, it is ready for incorporation into an active microbial environment. This begins the second active phase—biodegradation. FT-IR graphs are displayed next to each photograph to show you molecularly what is occurring your plastic product. An explanation of the FT-IR graphs can be found on page 20, the Report Addendum.

There have been 4 individual tests conducted so far. Each test has it's own graphs and discussion of the results. A summary of the results so far:

Control bags have shown no signs of degradation or chemical change through any testing so far. Their indoor and outdoor shelf life is undetermined at this point due to no clear results and no degradation occurring.

Bare Bones Bags containing oxo-biodegradable additive at an average concentration of 5.05% will have an indoor storage shelf life of approximately 12 months and an outdoor exposure shelf life of 9 months.

Limestone Bags containing oxo-biodegradable additive at an average concentration of 3.18% will have an indoor storage shelf life of approximately 18 months and an outdoor exposure shelf life of 9 months.

The shelf life of the Recycled Bags are a bit harder to determine. Due to the nature of recycled material, the concentration of random anomalies is higher in the resin. It is always recommended that the use of oxo-biodegradable additives should be incorporated into virgin resin for the predictability it offers. However, oxo-biodegradable additives will almost always degrade recycled resin. The problem is that recycled resin can contain many different constituents and these can cause havoc on the degradation life. Some will increase the stability while others cause faster degradation.

That being said, from the results obtained so far, Recycled Bags that contain oxo -biodegradable additive at a concentration of 4.09% will have an indoor storage shelf life of approximately 18 months and an outdoor exposure shelf life of unknown quantity. The results from accelerated UV aging were inconclusive and are being retested to help determine the outdoor shelf life.

## **PRO-OXIDANT PURITY TESTING**

	Pet Pickups			
<b>Trial Run</b>	<b>Bare Bones</b>	Recycled	Limestone	Control
1	5.81%	3.71%	3.24%	0.00%
2	5.60%	4.75%	4.05%	0.00%
3	3.74%	3.81%	2.27%	0.00%
Average PDQ-M Percentage	5.05%	4.09%	3.18%	0.00%

Our laboratory received 4 plastic samples from **Pet Pickups** for testing and analysis. Oxo-biodegradable additive contain a trace element at a known concentration, that when tested, the ratios will remain proportional, and the additive concentration loading rate can be calculated by our lab technicians.

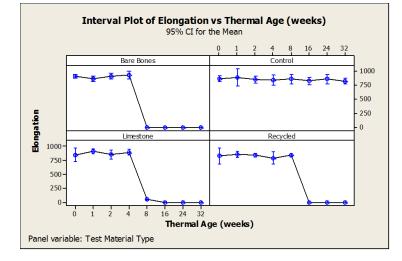
The results above indicate that the oxo-biodegradable additive was detected in the only 3 of the samples submitted for testing. The concentrations varied to a small degree in each sample, but the results were uniform. All concentrations observed in testing will allow oxo-biodegradation to occur in these samples.

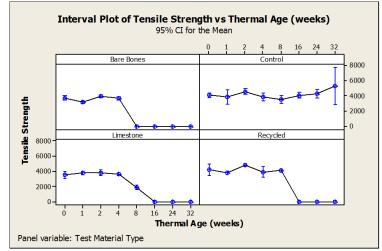
The results show that the concentration of oxo-biodegradable additive in the Bare Bones sample is around 5.05% on average. The concentration of the Recycled bags were around 4.09% on average. The concentration of the Limestone bags were around 3.18% on average.

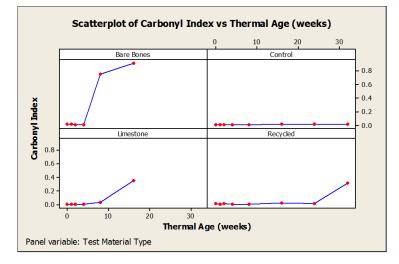
Once samples have degraded, and due to the presence of carbonyl groups, they will enter into the biodegradation phase once disposed of. Microorganisms commonly found in landfills and/or composts will feed off of the oxidized polymer chain to further reduce the material into CO<sub>2</sub>, water, and biomass (humus). The CO<sub>2</sub> is commonly used as fuel for further microbial growth.

Page 3

#### OXO-BIODEGRADABILITY ANALYSIS







Our laboratory tested 4 samples of Pet Pickups bags. Each bag was prepped and tested under ASTM 6954 guidelines at  $70^{\circ}$ C and 50% humidity.

The first and second graph show the changes over time to the structural integrity of the bags. In both graphs, all three samples that contained oxo-biodegradable additive showed dramatic changes to their structural integrity. The structural integrity of the Bare Bones bags dropped below >5% after only 8 weeks of accelerated thermal testing. That equates to a shelf life of approximately 12 months.

The Limestone and Recycled bags dropped below >5% after 16 weeks of accelerated thermal testing. That equates to a shelf life approximately 18 months.

The final graph takes a look at the chemical structure of the plastic as it is aged. A carbonyl index above 0.2 is relatively high and signals a large chemical change. It generally correlates directly to physical integrity. As you increase the number of carbon double bonded to oxygen in the plastic, the less stable a product becomes.

We see a dramatic increase in carbonyl concentration after only 8 weeks in the Bare Bones bags. The carbonyl index in the Limestone bags increases after 16 weeks. Finally, the recycled material showed an increase at the 32 week examination.

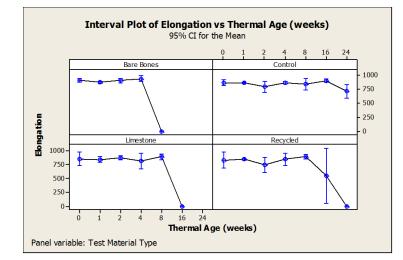
Compare all these results to the control, and you can see what effect oxo-biodegradable additives have on the product shelf life.

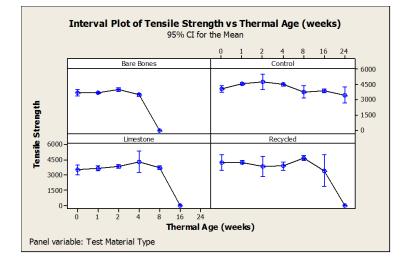
# 70 DEGREE CELSIUS RESULTS

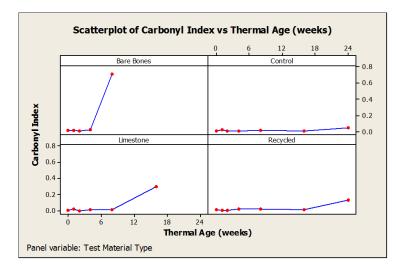
Page 4

#### OXO-BIODEGRADABILITY ANALYSIS

### **80 DEGREE CELSIUS RESULTS**







Our laboratory tested 4 samples of Pet Pickups bags. Each bag was prepped and tested under ASTM 6954 guidelines at 80°C and 50% humidity.

The first and second graph show the changes over time to the structural integrity of the bags. In both graphs, all three samples that contained oxo-biodegradable additive showed dramatic changes to their structural integrity. The structural integrity of the Bare Bones bags dropped below >5% after 8 weeks of accelerated thermal testing.

Limestone bags dropped below >5% after only 16 weeks of accelerated thermal testing.

The Recycled bags dropped below >5% after 32 weeks of accelerated thermal testing.

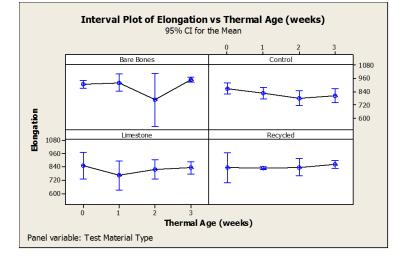
The final graph takes a look at the chemical structure of the plastic as it is aged. A carbonyl index above 0.2 is relatively high and signals a large chemical change. It generally correlates directly to physical integrity. As you increase the number of carbon double bonded to oxygen in the plastic, the less stable a product becomes.

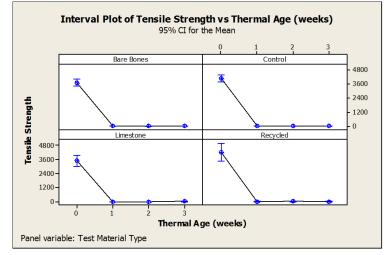
We see a dramatic increase in carbonyl concentration after only 8 weeks in the Bare Bones bags. The carbonyl index in the Limestone bags increases after 16 weeks. Finally, the recycled material showed an increase at the 32 week examination.

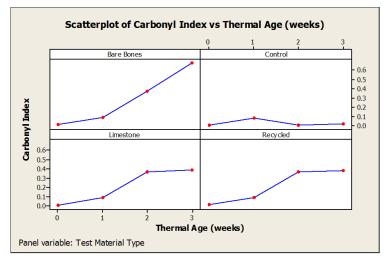
Compare all these results to the control, and you can see what effect oxo-biodegradable additives have on the product shelf life.

#### OXO-BIODEGRADABILITY ANALYSIS

**DEGREE CELSIUS RESULTS** 







Our laboratory is testing 4 samples of Pet Pickups bags. Each bag was prepped and tested under ASTM 6954 guidelines at 90°C and 50% humidity.

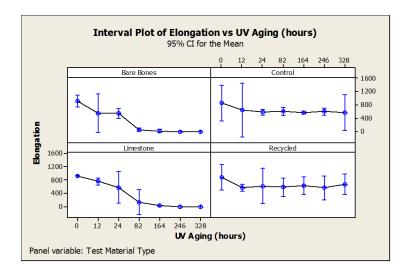
The first and second graph show the changes over time to the structural integrity of the bags. The samples have only been tested for 3 weeks of the potential 16. None of the samples so far have shown any large changes to elongation.

However, all 4 samples have lost a lot of their tensile strength through accelerated thermal aging. It can be assumed that all four samples will begin to physically fail in the very near future.

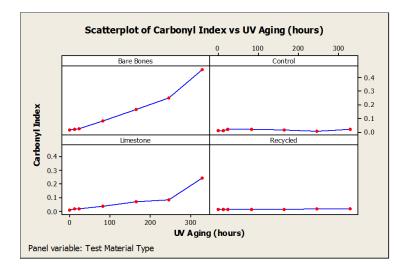
The final graph takes a look at the chemical structure of the plastic as it is aged. A carbonyl index above 0.2 is relatively high and signals a large chemical change. It generally correlates directly to physical integrity. As you increase the number of carbon double bonded to oxygen in the plastic, the less stable a product becomes.

We see a consistent increase in carbonyl concentration through all 3 weeks of testing for the Bare Bones bags. Both the Limestone and Recycled bags have also increased but plateaued shortly afterwards. In the control samples, there is very little change to the carbonyl index and that's to be expected since it is lacking any oxo-biodegradable additive.

90



#### Interval Plot of Tensile Strength vs UV Aging (hours) 95% CI for the Mean 164 246 328 82 24 Bare Bones Control 15000 10000 5000 **Tensile Strength** 0 -5000 Limestone Recycleo 15000 10000 5000 -5000 328 12 82 164 0 24 246 W Aging (hours) Panel variable: Test Material Type



Our laboratory tested 4 samples of Pet Pickups bags. Each bag was prepped and tested under ASTM 6954 guidelines for accelerated UV aging.

The first and second graph show the changes over time to the structural integrity of the bags. In both graphs, only two of the samples that contained oxo-biodegradable additive showed dramatic changes to their structural integrity. The structural integrity both the Bare Bones and Limestone bags dropped below >5% after 164 hours of accelerated UV aging. This equates to an outdoor shelf life of approximately 9 months.

The final graph takes a look at the chemical structure of the plastic as it is aged. A carbonyl index above 0.2 is relatively high and signals a large chemical change. It generally correlates directly to physical integrity. As you increase the number of carbon double bonded to oxygen in the plastic, the less stable a product becomes.

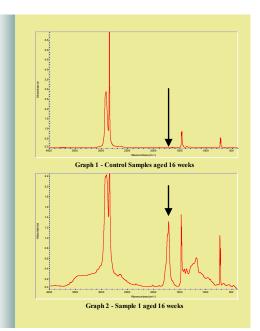
We see a continuous increase in carbonyl concentration through out testing for the Bare Bones bags. There is a slightly lower increase occurring in the Limestone bags until the 328hr examination when the concentration of carbonyl groups rises significantly.

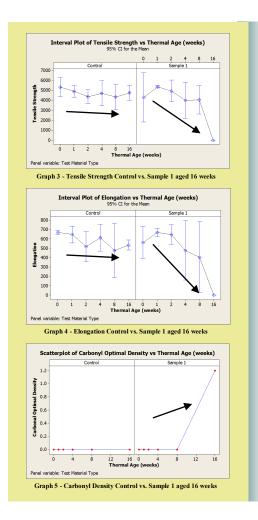
Both the recycled and control samples showed little to no change in chemical structure through testing.

## ACCELERATED UV AGING RESULTS

Graphs 1 and 2 are both FT-IR graphs collected by scanning plastic samples and documenting the amount of Carbon to Oxygen double bonds (C=O) that are present. The more C=O present in a plastic material, the more degradation that has occurred. Without these C=O sites, commonly found microorganisms would not be able to feed off the plastic once it's been disposed of. After microorganisms feed off of the oxidized polymer chains, all material is further reduced into non harmful CO<sub>2</sub>, water, and biomass (humus).

The arrows show where the C=O can be found on the graph. Notice how there is no relevant C=O peak on the Control samples even though it has been aged for 16 weeks. However, on the Samples with oxo-biodegradable additive, there is a very significant peak after 16 weeks of thermal aging. These same graphs are used to demonstrate how UV light affects plastic too.





Three properties were evaluated in comparing the degradation performance of all sample sets. Those properties are Tensile Strength, Break Strain, and the Carbonyl Density. Tensile Strength is the total area under the tensile stress versus strain curve and is related to toughness. Break strain is the elongation percentage to the breaking point. Carbonyl density is the identified absorbance value of the carbonyl peak through FT-IR analysis after considering the material's thickness.

Notice how in each of the three graphs to the left (Graph 3, 4, and 5), the trend of the control samples are fairly consistent even after 16 weeks of accelerated thermal aging. In samples containing oxo-biodegradable additive, there is a definite trend of degradation. In graph 5, there is a spike in Carbonyl Density in the oxo-biodegradable sample, signifying a larger increase in C=O groups.