BD Report

Evaluation of the Oxo-degradable characteristics of a green PE stretch film sample containing 1% Reverte™ manufactured by PT Trimitra Cikarang and supplied by PT Neochem Indonesia

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Sample details:

Received supplied from: PT Trimitra Cikarang
Project Reference No.: BD 1999
Sample(s) received: 30/04/2019

Sample description:

Sample Number	Material Type	Sample Form	Thickness	Base Colour	Print	Reverte™ Grade	Reverte™ Batch No.	Addition Level (wt%)
14522	*PE*	*Stretch film*	*~23 µm*	*Green*	*No*	*BD92771CC*	*24045*	*1%*

	Prepared by	Authorised by
Signature		
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Date	19th September 2019	19th September 2019

علامة الجودة الإماراتية
Emirates Quality Mark

The testing detailed in this report was performed wholly at:

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This report is applicable to the unique sample supplied to Wells Plastics Ltd by PT Trimitra Cikarang via our local distributor PT Neochem Indonesia. It should be noted that the report does not guarantee that subsequent production batches manufactured by PT Trimitra Cikarang will contain Reverte™ oxo-biodegradable masterbatch manufactured by Wells Plastics Ltd at the correct dosage level.

Unless otherwise stated, the testing, analysis and reporting of the results of the evaluation reported here were performed in accordance with the referenced applicable methods (internal, national or international.)

Work Instructions used in this evaluation:
QWI82 Production of BD graph & test report
QWI85 Accepting a BD sample
QWI86 BD Sample preparation
QWI91 Operating FT-IR for analysis of BD samples
QWI92 Printing BD sample FT-IR scans
QWI93 Conducting UV degradation testing

Evaluation of the oxo-biodegradable characteristics of a green PE stretch film sample supplied by PT Trimitra Cikarang, care of PT Neochem Indonesia

1. Background

PT Trimitra Cikarang is interested in evaluating Wells' "Reverte™" oxo-biodegradable masterbatch for use in PE shopping bags.

Reverte™ oxo-biodegradable masterbatch BD 92771 has been recommended for their application. This product contains a mixture of a metal ion pro-oxidant and a photoinitiated initial degradation inhibition package.

The grade utilised is polyethylene based and is generally suitable for inclusion in a broad range of polyethylenes including high density polyethylene (HDPE), low density polyethylene (LDPE) and linear low density polyethylene (LLDPE).

The formulation has been developed for a 1% addition to give thin section PE films maintained at 20ºC a controlled in-house shelf life of approximately 18 months, a further dwell time, normally around 2 to 6 months after photoinitiation and then a rapid breakdown of film properties resulting in acute embrittlement, normally after around 8 - 18 months. However, thicker section products, some stabiliser packages and specific polymer blends and pigmentation can significantly extend this embrittlement period and thinner sections, mineral fillers etc. can shorten it.

PT Trimitra Cikarang supplied one sample for evaluation of its oxo-biodegradable properties. The sample was a green PE stretch film. It measured ~23 µm in thickness and was submitted as containing 1% BD 92771CC. A section was cut out and labelled with a description/internal testing number for identification. The sample was subjected to testing of its oxo-degradable properties in Wells Plastics' laboratory at their plant in Stone, Staffordshire, UK.

2. Samples as received / before testing

3. Method

The high molecular weight of commercial grades of polymers render them fundamentally hydrophobic and, therefore, very resistant to direct microbial attack. A reduction of the polymer chain length from its initial value of around 250,000 to a value between 4,000 and 10,000 increases its intrinsic microbial accessibility and enables subsequent biodigestion.

Reverte™ products initially catalyse the oxo-degradation of the polymer chains and then promote the growth of microbial colonies to expedite the second biodegradation stage. The initial chain scission (degradation) of the polymer chain causes a serial reduction in polymer molecular weight which ultimately results in acute embrittlement, micro-fragmentation and biodigestion.

This degradation can be tracked by the measurement of critical physical properties, using test methods such as ASTM D3826 to measure properties such as elongation, but this method is somewhat flawed because as the degradation gets underway the test sample becomes too friable for physical testing.

However, because oxo-degradation causes the formation of a carbonyl group at the point of every scission, measurement of the onset and level of this carbonyl group development in the test product is a more accurate direct measure of its induced degradation by the metal ion pro-degradant system within the Reverte™ masterbatch. This carbonyl index, as it is directly proportional to the elongation at break, can be used to determine the elongation when the sample is too weak to be conventionally tested. The point of embrittlement in polyolefins is defined as the point at which the elongation at break is ≤ 5%.

Polyolefins are generally reduced to the embrittled state of ≤ 5% elongation when the carbonyl index is greater than approximately 0.1 to 0.6 depending on the type, grade, pigmentation and thickness of the product under consideration. Thicker sections, stabiliser packages and heavier pigmentations can give critical carbonyl indices far greater than the range given, but the actual critical carbonyl index is readily determined empirically during the testing procedure.

The sample was aged using a modified ASTM D 5208-01 (Cycle C) test method. The ageing cabinet utilised contained UV lamps to simulate gentle outdoor sunlight. The temperature of the cabinet was maintained at 50ºC according to the test method. Results from Wells' standard PE film without any Reverte™ addition was presented alongside the test specimen as a comparative control.

It should be noted that the level of UV exposure generated in the ageing cabinet is very low and should not be compared with the levels generated, for example, in QUV ageing experiments.

In effect, the UV exposure level is around 26kLy per year in the cabinet. To put this in perspective, to simulate a full year's outdoor exposure in the UK the samples would have to be in the cabinet for around 3 to 4 years, to match a year in mainland Europe they would have to be in the cabinet for around 4 to 5 years and a year in Florida USA would be simulated by 9 to 10 years in the cabinet.

The test piece in this experiment only spent just over 4 weeks in the cabinet; so we can see that the actual UV exposure was relatively low and that the acceleration of the ageing process should be largely attributed to the higher temperature (50ºC) following the photo-triggering stage of the breakdown reaction.

The test specimen was removed after fixed time periods and the carbonyl index determined by Infra-red analysis, using a modified ASTM D 5576 test method. In addition, the sample was empirically assessed for friability and state of embrittlement. The carbonyl index at the point at which the test piece was fully embrittled was noted and presented as 100% embrittlement. The remaining carbonyl indices were calculated as a percentage of this and presented as "Degree of Embrittlement".

Finally, Arrhenius principles were applied to the results obtained at 50ºC, transposing them into the real-time results that would be expected at 20ºC.

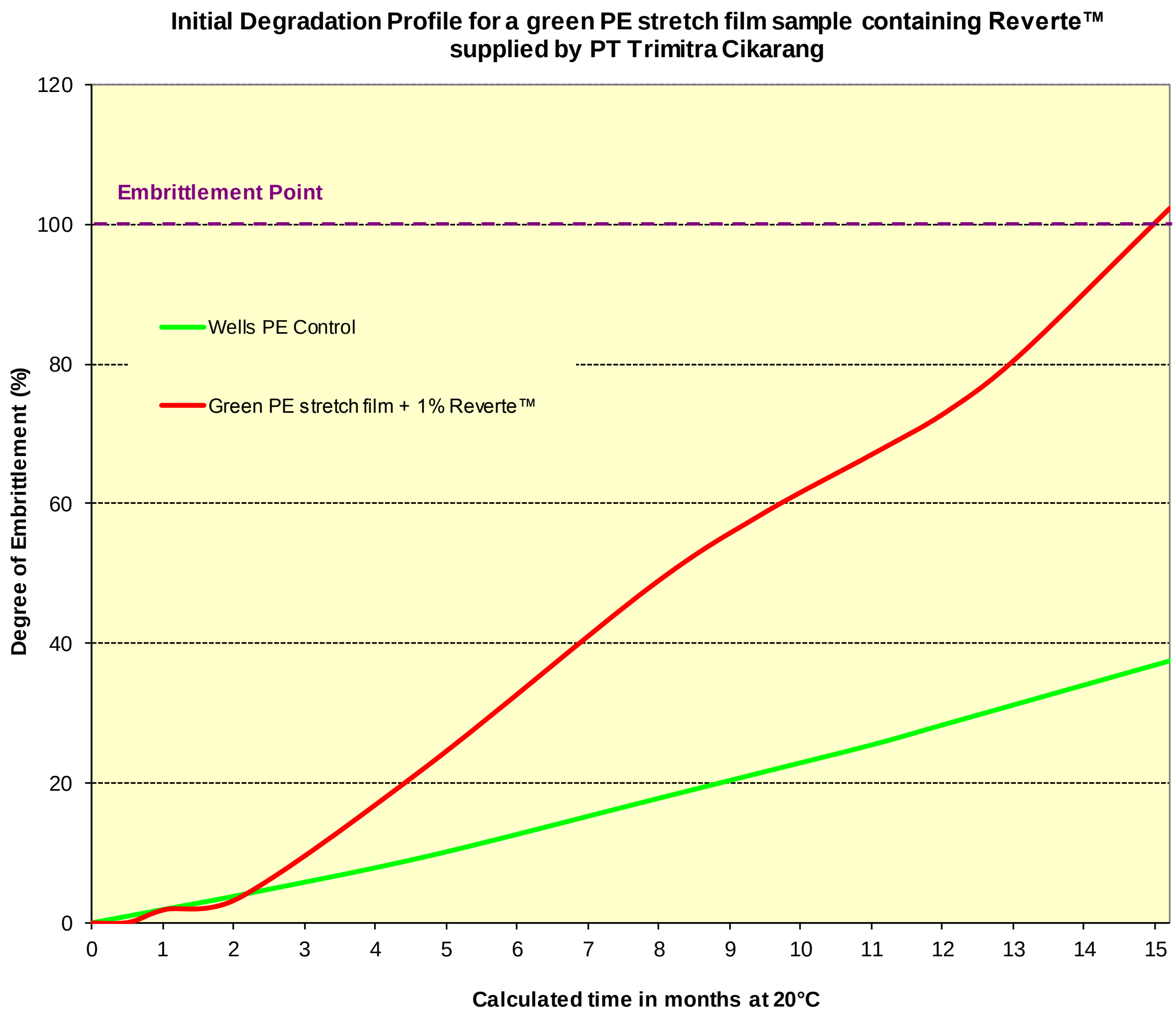
4. Results

4.1 Degree of Embrittlement

	Degree of Embrittlement (%)										
Accelerated Ageing Time (hours at 50°C)	**0**	**24**	**48**	**96**	**216**	**360**	**432**	**504**	**552**	**600**	**696**
Calculated Time (months at 20°C)	**0.0**	**0.5**	**1.0**	**2.1**	**4.7**	**7.9**	**9.4**	**11.0**	**12.1**	**13.1**	**15.2**
Wells PE Control	0.0	1.0	2.0	4.0	9.5	17.5	21.5	25.5	28.5	31.5	37.5
Green PE stretch film + 1% Reverte™	0.0	0.1	2.0	3.7	22.4	48.0	58.5	67.2	73.3	81.5	102.4

A photograph of the test specimen after its accelerated ageing period may be found in Appendix 1.

4.2 Graph of results



5. Discussion of Results

It is always difficult to precisely quantify results obtained in terms of real-time degradation due to the vagaries of natural conditions. However, the Arrhenius principles that we have applied to the accelerated ageing results enable us to present the results that would be expected from ageing in a real environment at a constant temperature of 20ºC in sunlight.

The Wells PE control film sample demonstrated fairly typical behaviour, not reaching a point of embrittlement during the test period and only reaching a level of embrittlement of around 37.5% after the 696 hour accelerated ageing period (calculated to around 15.2 months at 20ºC).

In marked contrast the green PE stretch film sample containing Reverte™ demonstrated a greatly enhanced degradation profile. It showed a well-defined "dwell time" of ~101 accelerated ageing hours (calculated to around 2.2 months at 20ºC) during which time no induced degradation was evident over the control. This was followed by a steady degradation in physical properties with the sample reaching 100% embrittlement after ~686 accelerated ageing hours (equivalent to around 15 months at 20ºC).

It can be seen that the green PE stretch film sample containing Reverte™ displayed the characteristic "dwell time" normally exhibited by Reverte™ containing products. This dwell time gives a greatly enhanced window of confidence in the use of oxo-biodegradable additive technology as no induced degradation is evident for the first time period of the product's calculated lifetime following its photo-triggering.

Finally, it should be noted that even when a control film may have degraded through normal UV/oxidative attack, this doesn't mean that the chain scission will continue in a uniform and controlled manner until the chains are short enough for microbial digestion.

This is what the use of Reverte™ additive does and it is this which speeds up and facilitates the ultimate biodegradation of the plastic following the initial oxo-breakdown.

It should be re-stated that these are idealised real-time projections based on accurate accelerated laboratory ageing and, as previously stated, natural climatic conditions of sunlight, soil temperature etc do vary. These extrapolated results have, therefore, been prepared in good faith, but any potential user would have to carry out his own empirical observations to ensure that the product was fit for his purpose in the precise ageing regime employed.

6. Conclusions

1. The addition of Reverte™ masterbatch to the green PE stretch film sample submitted by PT Trimitra Cikarang has been shown to be effective in introducing an oxo-biodegradable characteristic, giving a readily distinguishable dwell time after photoinitiation followed by a controlled progression towards full embrittlement.

END OF REPORT*

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Appendix I

Photograph of the sample after ageing

Following its ageing process of 696 accelerated ageing hours (calculated to around 15.2 months at 20°C), the green PE stretch film sample containing Reverte™ can be seen to have lost all of its significant physical properties, is exhibiting extreme friability and breaking up when handled.

These observations are commensurate with the measurements taken and contained within the body of this report.

END OF APPENDIX