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WELLS
PLASTICS LIMITED

BD Report

BD795/12 **02/05/2012**

Sample from Shandong Kingson Packing Tech Co Ltd

Received 06/03/2012

Sample Details

Description	Thickness	Base Colour	Print	OBD Additive
POF shrink film	~20 µm	Clear	None	Reverte BD 92771

Tested at Wells Plastics Ltd.'s laboratory, Stone, Staffordshire, U.K.

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Evaluation of the Oxo-Biodegradable Characteristics of a POF shrink film sample supplied by Shandong Kingson Packing Tech Co Ltd.

1. Background

Shandong Kingson Packing Tech Co Ltd interested in using Wells' "Reverte" oxo-biodegradable masterbatch in POF shrink film they produce.

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

The formulation has been developed for a 1% addition to give thin section polyolefin films maintained at 20°C 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.

One sample was provided for evaluation of its oxo-biodegradable properties. The sample was a clear POF shrink film which was understood to contain Reverte BD 92771. The sample was labelled with a description and an internal testing number to identify it.

2. Sample 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, microfragmentation 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 then the range given, but the actual critical carbonyl index is readily determined empirically during the testing procedure.

The test 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 50^{o}C according to the test method.

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 between 2 - 3 weeks in the cabinet; so we can see that the actual UV exposure was relatively slight and that the acceleration of the ageing process should be largely attributed to the higher temperature (50^{o}C) 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 50^{o}C, transposing them into the real-time results that would be expected at 20^{o}C.

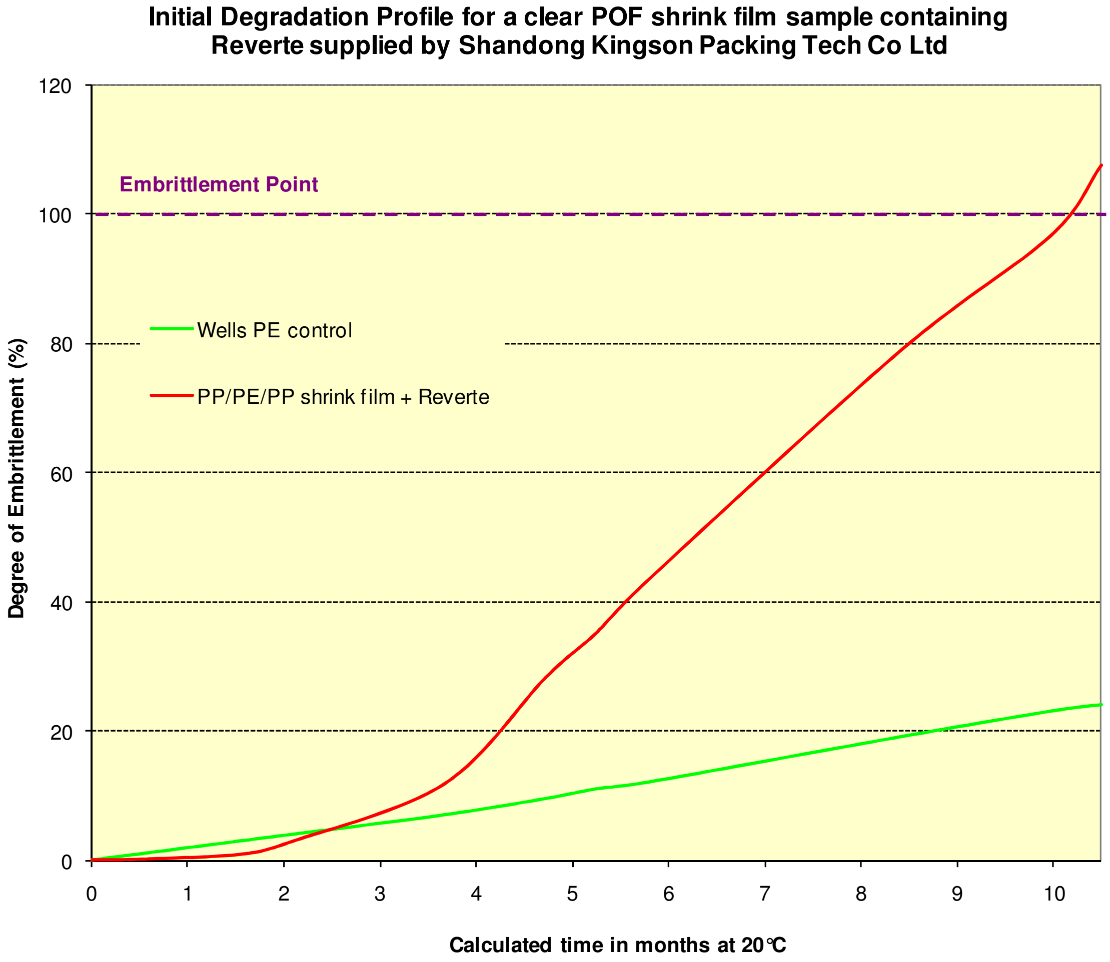
4. Results

4.1 Degree of Embrittlement.

	Degree of Embrittlement (%)										
Accelerated Ageing Time (hours at 50°C)	**0**	**24**	**72**	**96**	**168**	**216**	**240**	**264**	**384**	**456**	**480**
Calculated Time (months at 20°C)	**0.0**	**0.5**	**1.6**	**2.1**	**3.7**	**4.7**	**5.2**	**5.8**	**8.4**	**10.0**	**10.5**
Wells PE control	0.0	1.0	3.0	4.0	7.0	9.5	11.0	12.0	19.0	23.0	24.0
PP/PE/PP shrink film + Reverte	0.0	0.1	0.9	2.9	11.8	28.2	35.1	43.1	78.7	96.7	107.6

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 20°C in sunlight.

The clear POF shrink film sample containing Reverte demonstrated a greatly enhanced degradation profile. It showed a marked "dwell time of ~124 accelerated ageing hours (calculated to around 2.7 months at 20°C) 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 a point of 100% embrittlement after about 462 accelerated ageing hours (around 10.1 months at 20°C).

Chemical analysis of the clear POF shrink film sample indicated that the Reverte masterbatch is appropriate. The multi-layer structure of the film and the appropriate of Reverte will have impacted on the proper time taken for the sample to reach 100% embrittlement.

It can be seen that the sample 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 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 the Reverte additive does and it is this, together with the secondary phase biodegradation promoter, 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 POF shrink film sample supplied has been shown to be effective in introducing an oxo-biodegradable characteristic, giving a readily distinguishable dwell time after photo initiation followed by a steady progression towards embrittlement.
2. Analysis of the POF shrink film sample showed that the addition of Reverte masterbatch had been appropriate and this together with the multi-layer structure of the film will have impacted on the proper time taken for the film to reach 100% embrittlement.

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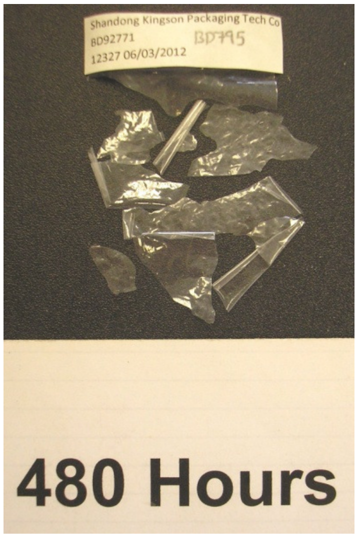
Appendix I

A photograph of the sample after ageing



Following its ageing process of 480 accelerated ageing hours (calculated to around 10.5 months at 20°C) the clear POF shrink film sample can be seen to have lost 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.