

**DEFRA Project EV0422, 'Assessing the environmental impacts of
oxo-degradable plastics across their life cycle'**

INDUSTRY RESPONSE TO THE REPORT

Independent Scientific and Technical Evidence researched by the principal industrial stakeholders in the oxo-biodegradable additives sector;
EPI Europe, Symphony Environmental and Wells Plastics.

Submitted through the offices of the British Plastics Federation to the UK Department for Environment, Food and Rural Affairs (DEFRA) on 21st April 2011

Summary

DEFRA Project EV0422 was commissioned to evaluate the environmental impact of oxo-biodegradable plastics. Its publication raises a number of concerns for those who specialise in oxo-biodegradable additive technology, specific concerns are:

- The level of stakeholder input into the preparation of the report was significantly and unfairly lower than for similar projects conducted by DEFRA in this area. Industry was not given appropriate opportunities to comment and to supply supporting data and information during the development of the report.
- Concerns have been raised with regards to the expertise of the authors in the area of degradable plastics and biodegradation as the report gives inaccurate descriptions of OBD technology and mechanisms.
- The conclusions drawn for OBD plastics in relation to recycling, biodegradation, and other issues are not supported by available evidence.

This response aims to provide DEFRA with evidence, both from peer-reviewed academic literature and from studies in independent laboratories (both appended to this report), which support claims that:

- Properly formulated OBD plastic films can be made to degrade oxidatively at ambient temperature in short (< 1year) periods to materials whose molecular weights and polarities are such as to make them biodegradable.
- Oxidative degradation in biologically-active environments is faster than in the typical air-oven conditions used for laboratory investigations, so that laboratory testing over-, rather than under-estimates lifetime in the environment.
- Properly formulated OBD plastic films, once oxidatively degraded can be shown to mineralise to CO₂ and H₂O in soil contact; greater than 90% mineralisation has been demonstrated in independent laboratory testing.
- There is no evidence of any ecotoxicity from OBD plastics at any stage of their degradation, and a great deal of detailed evidence that there are no ecotoxicity implications to their use.
- Claims of accumulation of undegraded or partially degraded plastics in the environment are wholly unjustified.
- The OBD plastics industry is working hard to establish appropriate standards for evaluation and control of the performance of its materials.

Introduction

Plastic packaging is essential to modern life; it would not be possible to feed sixty million people three times a day in the United Kingdom without it. Supermarket shopping would be impossible without packaging, and food waste would increase dramatically if we were not able to deliver appropriate amounts of prepared foodstuff to the public. Some mock the concept of an individual cucumber being wrapped in plastic shrink-wrap but that simple action permits a shelf life of 14 days in comparison to 3 days without wrapping.

Polyolefins in particular have been an enormous success in packaging and agricultural applications requiring mechanically tough films, because they are cheap, easy to process and tough, and offer excellent barrier properties against moisture, micro-organisms and oxygen. They have come to dominate a highly competitive market simply because they are the best solution.

The current issues surrounding waste management in the UK are well known but plastic material often cannot easily be collected for reuse or recycling. A possible solution where waste management is problematic is to render the packaging material degradable and ultimately biodegradable. That way, whichever disposal route is taken the plastic will not interrupt the process. In the case of any plastic which misses the correct disposal routes, e.g. by becoming litter, then biodegradability becomes an important element.

One approach is oxo-biodegradable plastics. These work through an additive system that renders conventional polyolefins bio-degradable.

The opportunity to convert oil-derived packaging into a biodegradable material and maintain all the advantages of economical production and safe, hygienic packaging has been recognised in Central and Latin America, Africa, the Middle East and India with regional and national legislation recognising and in some cases stipulating the use of oxo-biodegradable technology.

DEFRA Project EV0422 was commissioned to evaluate the environmental impact of OBD plastics across their full life cycle. Its publication raises a number of concerns which we feel must be resolved before this report can be considered complete.

In the following sections we attempt to what we perceive as the failings of Project EV0422.

A Preliminary Note on Relevant Standards

The DEFRA report places considerable weight on the European standard EN13432 and the ASTM standard D6400 as standards for specifying biodegradability of plastics. In referring to these (and other) standards, the report states (p. 8) that “it is stated explicitly that degradation is brought about by the action of living organisms rather than physical or chemical processes”. This is a fundamental misunderstanding of the nature of biodegradation of polymers of whatever origin.

Biodegradation is the chemical dissolution of materials by bacteria or other biological means. It is the transformation of a substance into new compounds through the action of microorganisms such as bacteria. However, all polymers derive their mechanical properties, especially toughness from the entanglement of their long chains. Even when isolated from each other, in solution, the chains are too large to be able to cross the cell walls of bacteria or fungi. All biodegradation of polymers requires some extra-cellular chemical process to cleave the chains and to release them from the entangled mass as fragments small enough to be transported into the cell and metabolised. This may occur by hydrolysis, as in the case of polyesters like the poly(hydroxyalkanoate)s or it may be oxidation, as in the case of natural rubber. In either case it may be purely chemical or it may be mediated by enzymes released from the cell. Both hydrolytic enzymes (typically esterases) and oxidising enzymes (e.g. cytochrome systems) are well known. There is certainly nothing in nature to exclude oxidative scission as a precursor to biodegradation; it is the way that nature disposes of both natural rubber latex and the lignin fractions of wood and other plant matter. There is a complete absence of this significant distinction in the report which results in the total confusion of biodegradability and compostability of plastics.

These two well-recognised biodegradation pathways for plastics, depend on the mechanism (which may be purely chemical or biologically mediated) by which the chains are cleaved to fragments small enough for bio-assimilation. The terms hydro- and oxo-biodegradation are used to emphasise that biodegradation of a plastic is always a two-stage process; there are oxidative and/or hydrolytic stages leading in the first instance to macromolecular chain breakdown to materials able to sustain attack by environmentally ubiquitous microorganisms. Both mechanisms are influenced by the environments to which the materials are exposed.

One false impression that permeates much of the report is that biodegradability and compostability are the same thing; they are not. This is demonstrated by standards

organisations such as BSI and ISO who have separate definitions for these two characteristics.

A special issue with standard specifications in the context of the DEFRA report is that they refer back to EN 13432 / ASTM D6400 assuming that these are the major relevant specifications, but these specifications actually refer only to composting under a set of conditions that have been pre-assigned.

A fundamental problem of the report is that it uses the D6400 or EN 13432 compost specification standards as the yardstick to conclude that OBD plastics are not biodegradable because they do not conform to this specification. The report correctly points out that the biodegradation of any material should always be qualified by rate, time and conditions of exposure. Hence, it is true that compostable plastics are plastics that biodegrade to EN 13432 specifications. However, it is equally true that plastics that do not meet composting standards may be perfectly acceptably biodegradable in other environments.

It is also relevant to point out that there are materials that meet EN 13432 or ASTM D6400 that are *not* biodegradable in many circumstances. Poly(lactic acid) (PLA) is the obvious example; it hydrolyses and biodegrades in industrial composting which is moist and hot, but does not biodegrade in dry soil or sand burial, or even in composting, if the temperature remains low, as is common in e.g. home composting.

Industrial composting is not the same as natural biodegradation. Standards like EN 13432 (and similar ones, such as ISO 17088 and ASTM D6868) are not appropriate for OBD plastics. Indeed, EN 13432 explicitly acknowledges that it is not appropriate for plastic wastes which may end up in the environment through uncontrolled means.

It is important to recognise the context of the development of EN 13432. Pressure from the EU to remove organic material from the landfill waste stream is leading to intensive efforts to develop ways of processing organic wastes to achieve value recovery. One method suggested for this is large-scale industrial composting, using closed-vessel processes capable of generating the high temperatures needed to kill pathogens and seeds. A few years ago composting of plastic packaging was seen as a possible route to waste disposal. EN 13432 was developed as a mandated standard, explicitly to standardise plastics to be labelled as “compostable”. This created a conflict with the composting industry, which is highly resistant to the idea that plastic of any kind is acceptable in its input streams and is rightly concerned about the possibility of plastics as contaminants in its products. EN 13432

was designed to place a very high barrier to the entry of any plastic into industrial composting.

Critics of EN 13432 and of attempts to make it more prescriptive, argue that very rapid mineralisation under composting conditions fails to meet the aim of “recovery” of waste material as required by EU legislation. Rapid biodegradation to CO₂ is effectively slow incineration without energy recovery. It is also pointed out that much of the material which normally goes into industrial composting will not meet the requirements of EN 13432 for biodegradability and that the compost product would have limited value if it did.

ASTM Standard D 6400-04 is also a specification for compostable plastics. It is designed for plastics that are composted in municipal or industrial aerobic composting facilities. It is similar in most respects to EN 13432. Both identify acceptable labelling practice for plastics claimed to be compostable. Rate and extent of composting must be similar to a selected natural standard; the extent of biodegradation is expected to be 60% in 6 months (as compared to 6 months for 90% conversion in EN 13432). For land application of the compost product, the metals analysis should meet all accepted norms and the compost must pass standard ecotoxicity tests.

These two standards are relevant *only* to the composting environments for which they were developed. They are not relevant to home composting (which hardly ever reaches the temperatures mandated in industrial processes and is often both cold and anaerobic). They are not relevant to the marine environment, for example and certainly not to materials that biodegrade slowly.

It is *not* claimed that oxo-biodegradable plastics meet the compostability requirements of EN 13432 or ASTM D6400. Indeed the industry is at pains to emphasise the view that these standards are wholly inappropriate for analysis of oxo-biodegradable plastics.

It is perfectly possible to formulate OBD plastics so that they will embrittle, fragment and become invisible to the naked eye during the high-temperature, in-vessel phase required of all industrial composting in the EU. However, current OBD plastics were not developed to be composted and current producers, fully recognising the concerns of the compost industries, make no claims for compostability of these products.

Other applications, like carrier bag film, require a longer lifetime before degradation begins and are formulated accordingly. The DEFRA report places significant weight on a study (Annex B1) published from the University of California at Chico, which concluded that OBD

plastics were not biodegradable. The report's authors fail to recognise that the OBD film used was never formulated or claimed to degrade under the conditions of open windrow composting and would not have been expected to show any degradation, rendering the results meaningless.

Much the same is true of most of the pictures cited in the report as evidence for non degradation. The films used were never formulated or claimed to degrade under the conditions tested; they are intended to have a useful life, to be recyclable and to degrade only on ultimate disposal. A fundamental advantage of OBD plastics is this ability to delay the onset of degradation to allow a useful service life.

The OBD plastics industry fully recognises the need for proper standards for testing and specifying the performance of its products, and is currently actively supporting progress towards new standard methodologies and specifications underway in several standards organizations, including ASTM, BSI and CEN.

The landmark standard for testing of OBD plastics is currently ASTM D6954 which is a Standard Guide for testing plastics that degrade in an environment by multiple steps. It prescribes testing these plastics using a battery of standard methods to follow both abiotic degradation and biodegradation. It includes toxicity testing at all degradation stages to ensure that partial or slow biodegradation does not introduce toxicity issues. The Standard Guide references existing standards that may be used at each degradation stage to determine extent and rate of degradation, and commits to further standards development or improvements where needed.

At BSI level, a test methods standard (BS 8472) has been under development for a number of years and has now been approved for publication. Like ASTM D6954, it includes toxicity testing to ensure that partial or slow biodegradation does not introduce toxicity issues. It also uses existing standards at each degradation stage to determine extent and rate of degradation.

At CEN level, TC249 WG9 has been working for several years to develop a test methods standard for plastics which will intentionally or accidentally end their life in soil contact or burial (this includes items like shotgun cartridges, plastic "clays" for shooting and agricultural twines as well as mulch films). This test guide is currently awaiting the setting up of a "round robin" test to ensure that its procedures are practical.

All of these standards are for test methods. They describe what tests are needed, how they shall be performed and how the results are to be reported.

The next stage is to develop standard specifications which set values to the performance of materials in different tests. Such a method is currently in development in the US for landfill environments and will permit quantification of landfill degradation of all plastics. Other standards are anticipated to follow, particularly standard specifications for OBD plastics in a given environment. This will differentiate oxo-biodegradable from hydro-biodegradable plastics and set expectations of performance. The industry is fully supporting these efforts, though progress is frustratingly slow.

There are a number of other standards which are often cited in discussions of biodegradable plastics. Whilst the methods of biodegradation testing which they specify may be applicable to developments of standards for OBD technology, they are not directly applicable to the exposure conditions expected or intended for OBD plastics. They include:

ASTM D7081-05; a specification for Non-floating Biodegradable Plastics in the marine environment. It is based on D6400 for meeting an acceptable degree of biodegradation under marine conditions, water and sediment, but not composting.

ASTM D5338-98; a Standard Test Method for Determining Aerobic Biodegradation of Plastic under controlled composting conditions in a laboratory approximation. The rate and degree of biodegradation are recorded. It is the basis for the D 6400 Specification standard.

ASTM D5511; a Standard Test Method for Determining Anaerobic biodegradation of plastics under high-solids (30%) anaerobic digestion conditions. It is a simulation of anaerobic digestion in an industrial or municipal facility, very similar to D5526. The method compares plastics relative to a standard for rate and degree of biodegradation using gas evolution measurements.

ASTM D5526-94; a Standard Test Method for determining the anaerobic biodegradation of plastics under accelerated landfill conditions – but only after anaerobic conditions prevail. It is a laboratory test in which the evolution of gas with time is used to compare and contrast different plastics. No specification has been set.

ASTM D5988-03; a Standard Test Method for determining aerobic biodegradation of plastic in soil or of residual plastic in a compost after composting. This is not a specification but simply a method showing how to test and record the rate and degree of biodegradation relative to similar materials and standards.

ASTM D6776-02; a Standard Test Method for determining anaerobic biodegradation of radio-labelled materials in a laboratory-scale simulated landfill. This is a minor modification of D5526 (above) where radio-labels allow greater accuracy in gas measurements.

Oxo-biodegradable Plastics; Technology and Mechanisms

The basic technology

All polymers derive their mechanical properties, especially toughness, from the entanglement of their long chains. Polymer chains long enough to confer useful mechanical properties are usually too large to be able to cross the cell walls of bacteria or fungi. All polymer biodegradation thus requires that there be some extra-cellular chemical process to cleave the chains and to release them from the entangled mass as fragments small enough to be transported into the cell and metabolised.

There are two well-recognised biodegradation pathways for plastics, hydro- and oxo-biodegradation. The hydro- and oxo- prefixes are inserted to emphasise that biodegradation of a plastic always involves two stages, and both mechanisms are influenced by the environments to which the materials are exposed.

In natural hydro-biodegradable (HBD) polymers, chain scission occurs by hydrolysis, as in the case of polyesters like the poly(hydroxyalkanoate)s. In natural oxo-biodegradable (OBD) polymers, scission is by oxidation, as in the case of natural rubber and lignin.

In either case, this initial degradation may be purely chemical or it may be mediated by enzymes released from the cell. Both hydrolytic enzymes (typically esterases) and oxidising enzymes (e.g. cytochrome systems) are well known. There is certainly nothing in nature to exclude oxidative scission as a precursor to biodegradation; it is the way nature disposes of both natural rubber latexes and the lignin fractions of wood and other plant matter.

Aside from the basic distinction between hydrolytic and oxidative cleavage, the main differences between OBD and HBD technologies are;

- a) The lifetime of an OBD polymer, before biodegradation starts, can be regulated by varying the antioxidant:pro-oxidant ratio.
- b) Because of the induction period required for oxidation to produce biodegradable materials, biodegradation of an OBD material is inevitably slower than that of an HBD. Although this excludes OBD plastics from applications requiring, or merely specifying, very rapid bio-assimilation, there are equally many applications where the rapid and uncontrolled biodegradation of HBD plastics is a problem.

In summary, the basic technology of an OBD material involves:

- a) An induction period during which oxidation catalysis by the pro-oxidant(s) is prevented by the antioxidant(s). *During this period there is no change in the polymer but the antioxidants are consumed.*
- b) A rapid oxidation of the polymer during which chain scission produces low molecular weight fragments, which are oxidised, hydrophilic, dense and polar.
- c) A period of bioassimilation of the oxidised fragments leading to mineralisation to CO₂.

It is important to emphasise that these are overlapping processes. In particular, once significant oxidation starts, it is faster in biotic than in abiotic environments, so that lifetime predictions from simple oven ageing or light exposure testing will tend to predict over-long breakdown times in natural exposure.

In the following sections we summarise the evidence for the various processes;

Mechanism of oxidation

Annex A of the DEFRA report gives a summary of the technology as seen by the report's authors. The authors accept (p. ix) that "the evidence for the mechanism of breakdown is highly robust".

Whilst broadly correct, the summary is deficient in some ways and shows the fundamental lack of familiarity with the details of polyolefin oxidation which characterises much of the report.

The oxidation of any polyolefin is an unavoidable consequence of the molecular structure and, in particular of the presence of impurities in the polymer as a result of manufacture and, especially, processing. All polymers derive their mechanical properties, especially toughness, from the entanglement of their long chains and cleavage of those chains in a polyolefin results in embrittlement.

The mechanism of oxidation has been the subject of innumerable studies since the pioneering work of Bolland and Gee in the 1940s on oxidation of rubbers. Despite the often repeated, but entirely incorrect, view that plastics have essentially infinite lifetimes, the modern polyolefins industry would not be possible without the use of antioxidants to protect the polymers from the inevitability of oxidative degradation. The peer-reviewed work of Ohtake's group in particular [1-4] has shown quite conclusively that even "ordinary" polyethylene, both film and thicker (bottle) materials, recovered from a waste disposal site after 30+ years shows substantial oxidative degradation and that "biodegradation of thin LDPE film in soil was unexpectedly fast because of the synergistic action of oxidative and/or

photo-oxidative degradation on biological activity, which is probably due to the increasing hydrophilicity of the film surface.”

The DEFRA report quite correctly points out that the function of the transition metal catalyst in oxo-biodegradable plastics is to accelerate the normal oxidative degradation reactions. There is *no change in the reaction mechanism*, merely in the rate of breakdown of the polymer.

The statement (underlined on p. 1 of Annex A) that transition metals do not catalyse the initial cleavage of the polymer chain is perfectly correct but wholly irrelevant. It has been accepted in all scientific studies of polyolefin oxidation for at least thirty years that the oxidation of any polyolefin is initiated by the decomposition of traces of peroxides produced during processing, not by direct chain scission. It is this decomposition of peroxides which drives the auto-accelerating oxidation of the polymer and it is this decomposition which is accelerated by the transition metal catalysis. Direct chain scission is irrelevant whether or not a catalyst is present, except in so far as it may act as a new source of free radicals (and hence peroxides) arising from mechanical stresses on the polymer in use.

There appears to be no real need for us to present more evidence that the oxidation catalysts used in OBD technology do indeed function to accelerate the normal degradation processes, but it is worthwhile to cite important peer-reviewed papers which have appeared since the DEFRA report's reviewers completed their literature survey.*

Thus, Fontanella et al. [5] (Annex 1 #1) recently reported an extensive study of HDPE, LDPE and LLDPE films of thickness 43 to 59 μm containing pro-oxidants and enough phenolic antioxidant to give a thermal induction period of more than 400 h at 60 °C in the dark. They showed that heat treatment “equivalent to 2-3 years of thermal oxidation at room temperature in the dark (particles buried in the soil or particles not exposed to sunlight) reduced Mw typically to around 4000. Similarly, Corti et al. [6] (Annex 1 #2) used a commercial 15 μm LLDPE mulch film and showed that “Films with pro-oxidant additives exhibited a higher level of oxidation as revealed by increase in their carbonyl index.” Husarova et al. [7] (Annex 1 #3) also recently demonstrated very rapid oxidation of 55 μm LDPE film containing a manganese based pro-oxidant; a sample with an initial Mw of 260,000 was reduced to Mw of 6400 in 40 days ageing at 70°C.

* New academic papers of direct relevance cited here are appended as Annex 1

Independent testing of commercial samples of OBD plastics[§] has also routinely shown that these materials can be formulated to degrade rapidly, both thermally or under UV solar irradiation to Mw values of well below 10,000. As an example, Fig. 1 (adapted from Annex 2 #1) shows the reduction of Mw with irradiation time for a commercial PE film aged according to the requirements of ASTM D6954 by a combination of heat and UV light. The requirements of ASTM D6954 Tier 1 (Mw <5000) and the UAE 5009/2009 standard (Mw<5000 in no more than 4 weeks) are achieved within 360 h of exposure, with the Mw falling from an initial value of 139,000 to 970 in 1392 h. It is also notable that the initial polydispersity index (Mw/Mn) fell from 8.6 to 2.3, consistent with completely random scission of the polymer chains. Figure 1 also shows data (adapted from Annex 2 #2) for a commercial PP film similarly aged. The requirement of Mw <5000 is achieved within 960 h of exposure, with the Mw falling from an initial value of 360,000 to 4290. The initial polydispersity index (Mw/Mn) fell from 7 to 2, again consistent with completely random scission of the polymer chains. Other independent studies are available and are appended as Annex 2 #3, and #4. All show the same behaviour; the initially high Mw of PE or PP is rapidly reduced to below 5000 by oxidation.

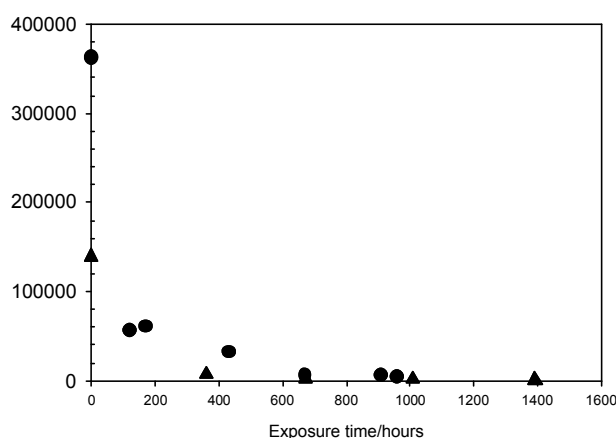


Fig. 1: Mw v time data for commercial OBD films aged as required by ASTM D6954 (? = polyethylene; ●= polypropylene) (adapted from Annex 2 #1 and #2)

Oxidation products and their biodegradability

In Annex 1 of the DEFRA report it is stated that “it is claimed that the species RO· can lead to the production of biodegradable intermediates”. This is far more than a claim. It is a fact supported by large amounts of robust experimental evidence.

[§] Independent studies of commercial materials of direct relevance cited here are appended as Annex 2

It is important to recognise that the fragmentation of a polyolefin by oxidation-induced embrittlement is not simply scission in the sense of cutting the film into pieces. It happens because the molecular weight of the polymer is reduced to the point where it is no longer sufficiently entangled to be able to act in a tough manner. Because this molecular weight reduction is caused by oxidative chain scission it leads to polymer fragments which not only have low molecular weights but are also significantly polar.

In their original study [8], Chiellini et al., studied oxidation of a commercial OBD film, without any pre-treatment. The LDPE film was thermally degraded in air in an oven at 55 °C for 44 days. As shown in Fig. 2, the weight of the sample increased due to oxygen absorption, but, more significantly, at the end of oxidation the weight fraction soluble in acetone had risen to over 25%;

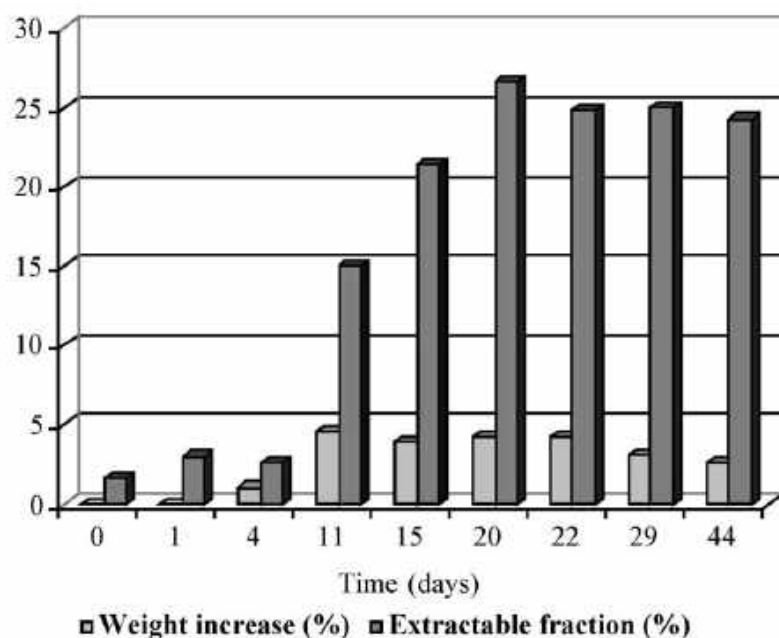


Fig. 2: Increase of dry weight and acetone extractable fraction of LDPE film with thermal degradation at 55°C [8].

Similar results were obtained by the same authors [9] in a study of thermal oxidation of several commercial OBD films of LDPE at different temperatures (Table 1); acetone soluble fractions of up to ca. 30% were recorded.

Sample	CO _i ^a	Acetone extract		
		(%)	Mw ^b (kDa)	ID ^b
FCB-ZSK10	0.453	6.5	1.52	1.49
	0.534	7.7	1.47	1.46
	3.583	17.9	1.30	1.39
	6.816	27.1	0.92	1.32
FCB-ZSK15	0.212	5.9	1.58	1.46
	2.864	9.2	1.67	1.52
	5.193	23.8	1.27	1.43
	7.256	22.6	1.03	1.36
LDPE-DCP540	0.627	5.5	1.08	1.27
	2.243	11.3	1.49	1.41
	4.818	21.1	1.08	1.37
	5.441	27.7	0.89	1.33

^a Evaluated by FT-IR as $D_{11640-1840}/D_{11435}$

^b Determined by HT-GPC.

Table 1: Relationship between carbonyl index (CO_i) and percentage extractable with acetone from original and thermally treated LDPE film [9].

PE is insoluble in acetone and these high weight fractions of extractable material, coupled with their low molecular weights and high carbonyl content as measured by FTIR, show that this material is no longer in any real sense PE; it is a highly oxidised and polar material.

These highly oxidised fractions are highly biodegradable. Fig. 3 shows mineralisation data obtained in soil burial experiments at 25 °C [8, 10]. They clearly show that these polar, oxidised fractions are as biodegradable as cellulose, the material used as a positive standard in biodegradation testing.

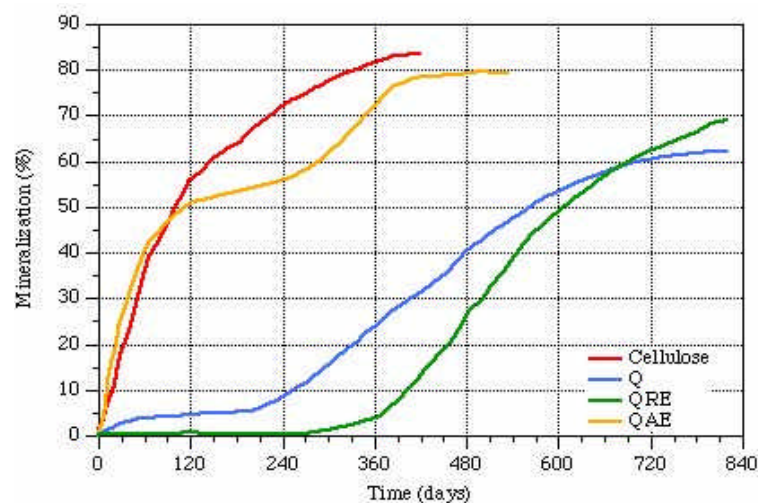


Fig. 3: Mineralisation in soil burial at 25 °C. QAE is the acetone extract from an oxidised LDPE film. [10]

Timescale of oxidative breakdown

There is very robust peer-reviewed evidence that:

- a) Commercial OBD films do degrade by oxidative chain scission to produce low molecular weight products.
- b) The products of oxidation are highly biodegradable.

The real issues are:

- a) What is the timescale of the oxidation in real-world applications? and;
- b) What is the evidence for completeness of biodegradation?

The report is critical of claims on the timescale of oxidative breakdown of OBD films to the point of biodegradability. They cite the paper of Jakubowicz, [11] who used the Arrhenius approach to predict that his Mn-activated samples would have degraded to the point of biodegradability in between 2.5 and 4.5 years and use this to conclude that the breakdown time of OBD polymers exposed outdoors will inevitably be long.

It is important to recognise that the oxidative lifetime of any OBD material depends upon the amounts of antioxidant and of pro-oxidant incorporated and that lifetimes can be very variable depending upon the formulation. This is a major strength of OBD technology; it allows sensible useful life without degradation and it also allows the materials to be recycled.

There are good reasons for believing that OBD materials can be made to degrade in shorter times than those criticised by the reports authors as unacceptably long.

In one recent paper [12] (Annex 1 #4), Ojeda et al studied degradation of a commercial HDPE/LLDPE blend containing a pro-oxidant additive and taken from the market as a supermarket bag. They showed that oxidation reduced the weight average molecular weight from its initial value of 183,000 to 8300 after only 280 days of outdoor exposure in Porto Alegre, (Brazil).

Jakubowicz [13,14] recently presented data derived from testing of some commercial OBD films using Mn based pro-oxidant. As in previous work, they aged the samples thermally, in the dark at slightly elevated (40 to 75 °C) temperatures and used the Arrhenius equation to predict lifetimes to oxidative failure at 25 °C ranging from 65 to 330 days (Fig 4).

It should be noted that:

- a) These data were produced at SP laboratories in Sweden, a wholly independent testing body with no connection to commercial producers of OBD film,

- b) the samples used were commercial products, and
- c) the predictions of lifetime are in reasonable agreement with actual test data in soil exposure (discussed below).

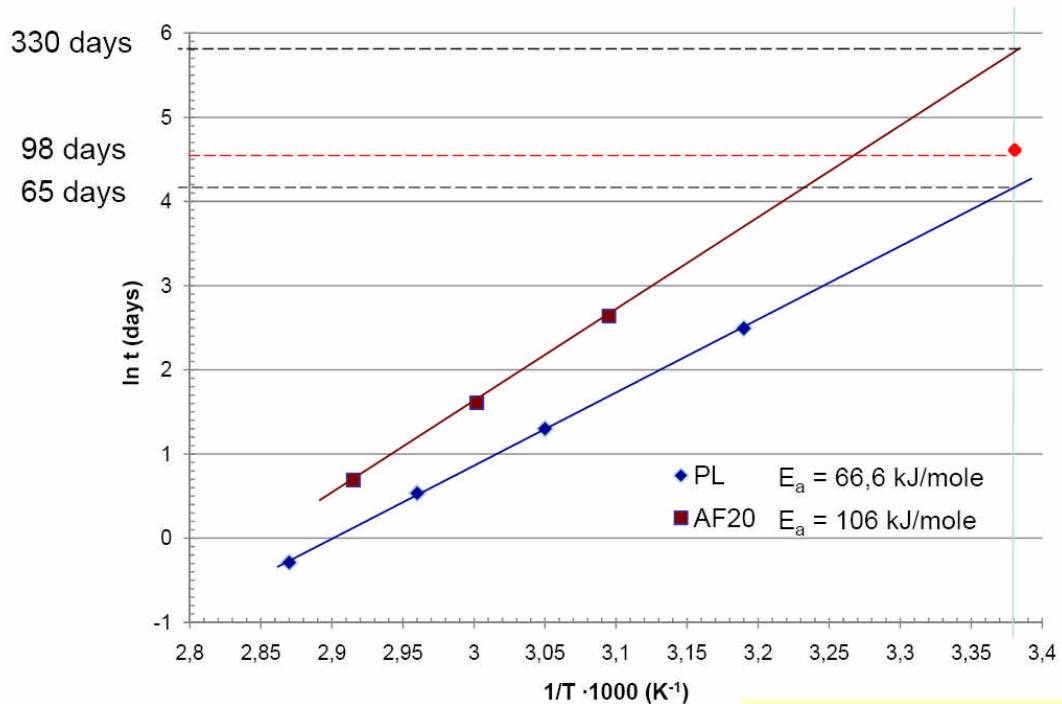


Fig. 4: Arrhenius plots for degradation of commercial OBD films. Reproduced with permission from [13].

Another factor is that oven ageing tests ignore the synergism between oxidation and biodegradation in the real world. Thus, Corti et al. [6] recently showed that once a commercial mulch film sample exposed to light began to oxidise, subsequent oxidation was significantly faster in a biotic than an abiotic environment at 23 °C. They suggest that abiotic oxidation of a polymer's carbon backbone produced metabolites which supported metabolic activities in fungal cells leading to further biotically-mediated polymer degradation. Thus, the combined impact of abiotic and biotic factors promoted the oxidation/biodegradation of the films.

A similar study formed part of an evaluation of commercial materials, also conducted by Chiellini's laboratory and presented here as annex 2 #5. FTIR measurements of oxidation in an oxidised OBD film which had been incubated in the presence of an organism (*S. griseus*) capable of secreting oxidising enzymes showed that the material underwent significant oxidation as compared to the same film without exposure to the micro-organism.

These results clearly show that OBD materials can be degraded by oxidation to the point of total embrittlement at ambient temperatures in periods of less than a year if properly formulated.

What is needed is the development of proper standards against which commercial products can be evaluated.

Biodegradation studies

One false impression that permeates much of the report is that biodegradability and compostability are the same thing. This is far from true. The environment of an industrial composting facility is quite different from that to which OBD polymers are expected to be exposed in any normal use. Both the temperatures and the likely microbiological activity are quite different.

EN 13432 and other composting standards were developed to place a very high barrier to the entry of any plastic into industrial composting and, as far as we are aware, no reputable manufacturer makes claims of compostability for OBD plastics. The most likely disposal routes for OBD plastics are recycle, landfill and soil surface exposure/burial (litter and mulch films), so that degradation in soil contact or burial is more relevant.

Because of market requirements, commercial OBD materials have been independently tested in other countries, mainly in China. For example, Annex 2 #6 is a report from the Chemical Industry Institute of Shihezi University, on the performance of a PE mulching film. The material was recovered from a field after one growing season and tested according to ISO 14855:1999 in a controlled composting environment; it was shown to reach 77% conversion of carbon to CO₂ in 45 days. Annex 2 #7 is a report from the China National Centre for Quality Supervision and Testing of Plastics Products, on the testing of oxo-biodegradable polypropylene lunch boxes, again to ISO 14855:1999, after pre-oxidation in a controlled composting environment. The requirement was for 30% conversion of carbon to CO₂, hence the test was stopped when this was achieved. This material achieved 33% conversion in 126 days.

Annex 2 #8 is a report from a Spanish Technology Centre, commissioned by a Spanish supermarket chain to investigate claims of degradability of T-shirt bags taken from the market place. Degradability testing of the oxidised material in a controlled composting environment at 58 °C showed 28% mineralisation to CO₂ in 67 days.

These independent tests of commercial products were all conducted under composting conditions, according to international standard methods. In practice, OBD materials are not intended for composting applications but are more likely to end their lives in soil contact or burial.

In the work of Chiellini et al. on a commercial material, presented in Annex 2 #5, respirometric measurements of CO₂ production from an oxidised OBD film which had been incubated in the presence of an organism (*S. griseus*) capable of secreting oxidising enzymes showed that biodegradation was very much faster than that of the same film without exposure to the micro-organism, reaching 80% of the theoretical CO₂ yield in 130 days of soil exposure at 28 °C. This result was attributed to the demonstrable increase of oxidation by the biological attack by the selected microorganisms once the oxidation has been initiated by thermal ageing.

Jakubowicz [15] has shown that some OBD plastics may degrade more slowly in active composting than in air. However, the published studies by Chiellini et al. show *faster* bioassimilation of OBD plastic in soil at 25 °C than in compost at 58 °C and the most recent work of Jakubowicz, reported below has confirmed this.

In this most recent study, Jakubowicz et al. performed a detailed analysis of biodegradation of some commercial OBD films [13, 14] (Ref. 14 is Annex 1 #5). They carried out mineralisation testing in both compost (58 °C) and soil burial (25 °C) environments. The sample used was a commercial film which had been oxidised (10 days at 65 °C in air) to an Mw of 8,800 from its original value of 131,500. In composting, the thermally oxidised material reached 40 – 50% mineralisation in 700 days, showing intermediate values comparable with the data of Chiellini and of Fontanella et al. [5] (Fig. 4).

However, studies in soil burial support the results of Chiellini about faster bioassimilation in soil than in compost; when the same samples were tested in soil burial at 25 °C (Fig. 5), they had an induction time of around 300 days then achieved 80% mineralisation in a further 300 days. *The final mineralisation after 700 days was 91%.*

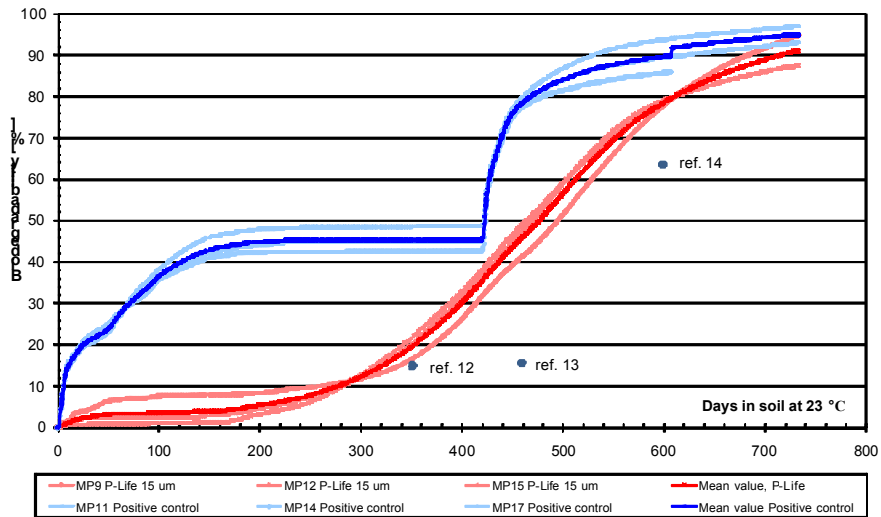


Fig. 5: Compost mineralisation of commercial OBD films. Reproduced with permission from [13,14].

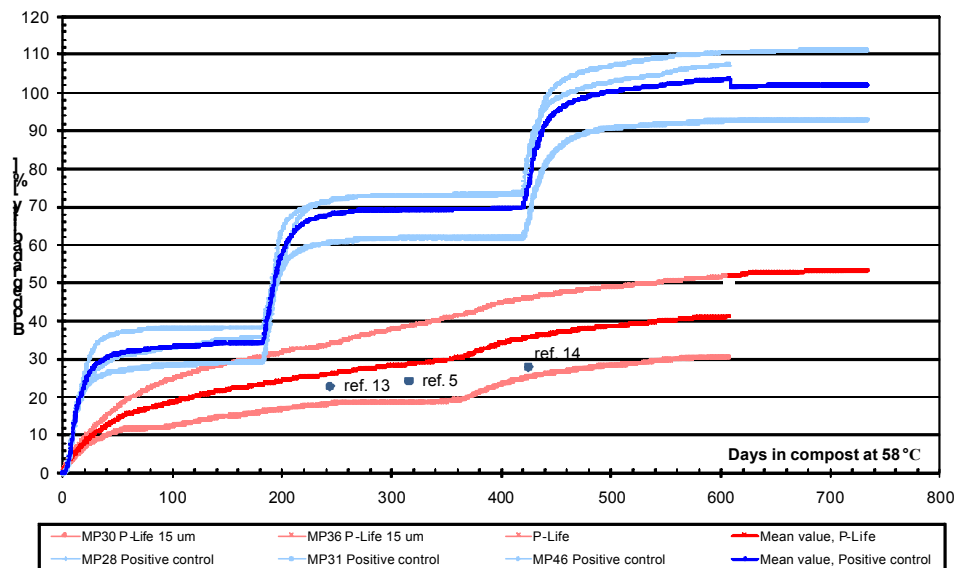


Fig. 6: Soil burial mineralisation of commercial OBD films. Reproduced with permission from [13,14].

A study of the bacterial and fungal populations of the soil and compost suggested that there were distinct differences which accounted for the ability of the soil microbes and fungi to metabolise the plastic more rapidly than did the compost.

Given that these results were obtained by a wholly independent Government Standards Laboratory of the highest reputation, using commercially sourced samples, it is worth quoting from the conclusions in ref. [14]:

“Results obtained after two years of biodegradation experiments have shown significantly higher levels of biodegradability in the soil environment at 23 °C compared with the compost environment at 58 °C. After two years in the soil mineralization experiment, 91% biodegradability was achieved without reaching a plateau phase. *This result has two important implications. The most important one is that it is possible to create LDPE based materials that will almost completely biodegrade in soil within two years. It also indicates that the risk of plastic fragments remaining in soil indefinitely is very low.* (Our italics)”

Again, these data show that it is perfectly possible to produce commercial materials which oxidise and mineralise in acceptable timescales. What is needed is the development of proper standards against which commercial products can be evaluated.

Toxicity

The authors of the DEFRA report concede that there is no evidence of any likely or actual toxic effects in the environment arising from the use of OBP materials so there is no real need to dwell on the issue. All manufacturers of OBD materials currently in the market have independent data verifying the absence of any restricted compounds from their products and of their compliance with food safety legislation relating to migration of additives from the plastic into contacting media. However some supplementary confirmation is available from work performed recently by Chiellini [10].

Chiellini submitted a pro-oxidant additive based on an aliphatic salt of a transition metal to a comparative biodegradation test with a linear saturated hydrocarbon (C₂₂H₄₆ – Docosane) in river water medium and found that the additive biodegraded with a profile strictly analogous to that recorded for docosane. A 90% level of comparable mineralization is reached in 140 days of incubation (Fig. 7).

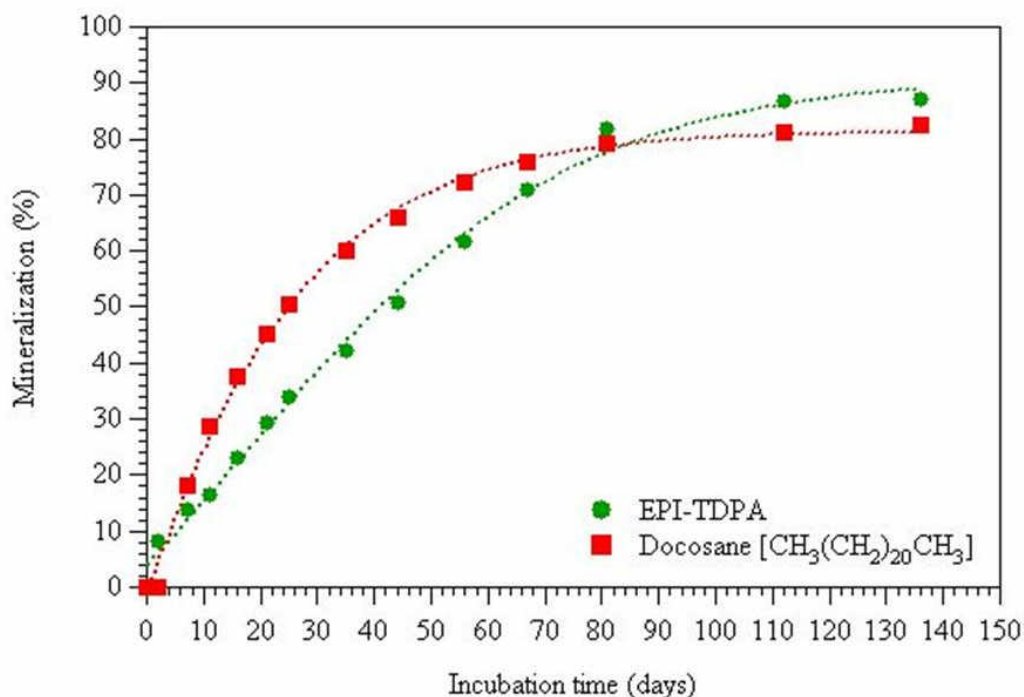


Fig. 7: Mineralisation in river water at 25°C of pro-oxidant additive (EPI-TDA) and docosane [10].

Moreover, cress seed germination testing (OECD 208) for ecotoxicity carried out on the incubation media (river water) of different pro-degradant additives based on transition metal organic salts, once 90% of mineralization was reached, when compared with incubation media at 90% docosane degradation level, blank (river water) and distilled water, gave analogous percentages of seed germination (Fig. 8, Table 2). These results confirm that the organic component of the additive is fully biodegradable and that the metal component has no toxic effects even when present in far higher concentrations than would ever be possible from a formulated OBD plastic, where the additive is typically used at 1-4% concentration.



Fig. 8: Picture of the cress seeds germination test

Test Culture	starting concentration	Germinated seeds
	(ppm)	(%)
Ad#1	258	96
Ad#2	269	98
Ad#3	301	94
Docosane	266	92
Blank	-	90
Water control	-	92

Table 2: Cress seeds germination test results [10].

Persistence in Soil

The biodegradation of OBD plastics in soil conditions is inevitably slow compared to materials like cellulose and starch, though it is entirely consistent with (and can be much faster than) biodegradation of most natural waste materials which are ligno-celluloses not pure cellulose. Critics of OBD technology argue (incorrectly) that the oxidised fragments of OBD plastics will stop degrading/biodegrading and persist indefinitely in soil or water, or that the particles which have oxidised but not yet mineralised are a hazard.

Another common criticism is that fragments of OBD plastics are harmful in the environment because they will blow around in the wind, adsorb pesticides or wash out into rivers or the sea.

These are all criticisms based on speculation rather than hard evidence.

There is no chain length or particle size limit below which oxidation chemistry stops. Conventional polyolefins oxidise relatively slowly in the environment whereas the oxo-biodegradable polyolefins oxidise much more rapidly. The products are the same, however, and degradation chemistry proceeds in both cases regardless of the size of the fragments.

The report presents no evidence that plastic particles are any more likely to attract toxins than are any other particles naturally present in the environment; pesticides are strongly adsorbed by humic materials in the soil. In any case, fragments of OBD plastic which have oxidised are a material with a completely different molecular structure. There is no evidence that such fragments would be harmful. Indeed there is substantial and robust evidence that they are not. The report acknowledges that there are no toxicity issues associated with the use of OBD plastics. Extensive ecotoxicity testing has shown no effects of the use of OBD plastics on soil fertility or on survival of microorganisms. Scott [16] has provided an extensive review of many years experience of the use of OBD plastics in agriculture, and Yang et al. [17] report an extensive Taiwanese Government study of OBD plastics in agriculture; both show no adverse effects and very substantial environmental benefits. As further examples, Annex 2 #9 and #10 are reports of ecotoxicity studies on commercial OBD materials, showing full compliance with relevant standards. These studies were carried out in an independent laboratory where the additive masterbatch was ground to powder and tested for ecotoxicity. Despite the fact that the pure additive is intended for use at a 1% addition rate in OBD film, and thus contains 100 times more metal salt than would be found in an OBD film, no ecotoxicity was observed.

Annex 2 # 11 and Annex 2 #12 are reports of ecotoxicity studies carried out on oxidatively degraded commercial OBD films, as required for Tier 2 and Tier 3 ecotoxicity testing according to ASTM6954 and showing neither short- nor long-term toxicity.

The absence of any ecotoxicity effects from partially degraded OBD plastics is also confirmed by the results of independent testing of commercial films subjected to industrial composting conditions [18]. Compost was produced incorporating commercial OBD plastic and contained partially-oxidised film as well as its oxidation products in the biomass.

Ecotoxicity was evaluated according to ASTM - approved methods, at independent laboratories and no ecotoxicity was found

In his recent study of a commercial material (Annex 2 #5), Chiellini used xylene extraction of the residues from biometric testing of a commercial OBD film to determine the presence of undegraded plastic and concluded that no significant hydrocarbon residues were present. They also showed no ecotoxicity of the residues from biometric testing “even at fairly high concentrations of potential contaminants”

OBD films have been widely used in agricultural mulching, in Israel, USA, Japan, China, Taiwan and some South American countries, since 1975 with no evidence of residual plastics particles or loss of soil fertility year on year. Partly-oxidised plastics are easily wetted by water and extremely adhesive to soil surfaces. They do not blow around in the wind.

OBD plastics at the very least provide a respite from the visual pollution problem by causing discarded plastics bags and films to disintegrate into invisibly-small fragments. However, fertile soils require organic carbon and it has been demonstrated by Chiellini's group [19] that part of the polymer carbon is fixed in the soil as cell biomass during biodegradation of OBD plastics. Discarded OBD plastics can be expected to contribute to soil enrichment while compromising neither use-life functions, reuse nor recycling.

Recyclability

The report claims that “Oxo-degradable plastics are not suitable for recycling with mainstream plastics”. This statement gives very little consideration of the available evidence showing that these materials can be safely recycled. In particular it is based upon a biased view of the report from the Environment Department of Quebec cited as reference 67 in the report.

Properly formulated and managed, OBD plastics will not compromise an oil-based recycling stream.

The length of the useful life of an OBD plastic product is determined by the ratio of the concentrations of the antioxidants and of the pro-oxidants contained in the particular formulation, which can be modified so that the plastic product degrades according to whatever timescale is required.

It is important to recognise exactly what is happening in an OBD plastic in the early stages of its life. As in all plastics, the presence of traces of peroxides initiates radical reactions in the material and this is accelerated by the pro-oxidant catalyst in an OBD material. The main effect of the antioxidants is to trap these radicals and prevent their attack on the polymer. *Until the antioxidants are consumed and oxidation of the polymer starts, there is no change in the polymer.*

The important point is that it is the ratio of pro-oxidant to antioxidant which is controlling. If OBD plastic is recycled as a minor component of a recycle stream the antioxidant concentration is unchanged because both the OBD and the conventional plastic contain antioxidants at similar concentrations. In contrast the pro-oxidant is massively diluted and its effect will be negligible as long as the proportion of OBD plastic is not too high.

Obviously if *any* plastic is to be recycled it must be collected and processed before it has become degraded. OBD products will normally have a useful life before embrittlement of at least 18 months, and if they have not been collected and recycled by then, they probably never will be.

If a new product to be made from recycle which contains, or might contain, a pro-oxidant, is intended for short-life products such as refuse-sacks, bin-liners, shopping bags, bread wrappers etc., the effect of any pro-degradant formulation is unlikely to manifest itself during the intended service-life. However, it is arguable that it would be sensible to add pro-oxidants to such items, since a proportion of them are likely to find their way into the environment, where they would otherwise subsist for decades after being discarded, and biodegradability for such items is desirable.

If the plastic is intended for long-term applications and is made from recycled material then re-stabilisation should be the norm.

The Quebec report concluded that at least one of the two OBD films which they tested was entirely compatible with recycling of PE film and had no adverse effects on the plastic during processing or on its lifetime afterwards. The poor performance of the other film tested suggests that the material was either not well specified or (more likely) that it was degraded before entering the recycle stream. This is a wholly independent study, commissioned by a Government Department.

Again, these data show that it is perfectly possible to produce commercial materials which are entirely compatible with recycling streams and have no deleterious effect on lifetime.

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Enclosed Supporting Literature

Annex 1: From Academia:

The following papers have been published since the DEFRA report was composed and therefore would not have been accessible to its authors at that time. These papers are referenced within this response.

1. Fontanella et al., 2010. Comparison of the biodegradability of various polyethylene films containing pro-oxidant additives. *Polymer Degradation and Stability*, 95, 1011 – 1021
[Shown as reference #5 in the response]
2. Corti et al., 2010. Oxidation and biodegradation of polyethylene films containing pro-oxidant additives: Synergistic effects of sunlight exposure, thermal aging and fungal biodegradation. *Polymer Degradation and Stability*, 95, 1106 – 1114
[Shown as reference #6 in the response]
3. Husarova et al., 2010. Aerobic biodegradation of calcium carbonate filled polyethylene film containing pro-oxidant additives. *Polymer Degradation and Stability*, 95, 1794 – 1799
[Shown as reference #7 in the response]
4. Ojeda et al., 2011. Degradability of linear polyolefins under natural weathering. *Polymer Degradation and Stability*, 96, 703 – 707
[Shown as reference #12 in the response]
5. Jakubowicz et al., 2011. Kinetics of abiotic and biotic degradability of LDPE containing pro-degradant additives. *Polymer Degradation and Stability*, 96, 919 – 928
[Shown as reference #14 in the response]

Annex 2: From Industry:

The following papers have been supplied by Symphony Environmental and Well Plastics and were produced as part of their in-house testing programmes. The inclusion of these papers is to provide additional information in support of the submission. EPI Europe carries out its testing programmes through its collaboration with Prof. Emo Chiellini at Pisa. The work of Corti et al. ref. [6] and of Chiellini et al. refs. [8,9] was carried out with commercial material supplied by EPI. EPI also provided the commercial material for the recyclability study described on p. 24.

1. RAPRA report 51806 (26th July 2010) – measurement of average molecular weight, by GPC, as required by ASTM6954 tier 1 and UAE S. no. 5009/2009 Section 5.3 Abiotic Degradation.

This report describes the reduction in molecular weight of LDPE material containing Wells Plastics Reverte grade BD92771 when exposed to a combination of light and temperature over a period of just over 8 weeks. It illustrates that the requirements of ASTM6954 Tier 1 (Mw <5000) and the UAE 5009/2009 (Mw<5000 in no more than 4 weeks) standard are achieved within 360hrs of exposure, with the Mw falling from an initial value of 139,000 to 2,860, and falling to 920 in 1392 hrs.

2. RAPRA report 50967 (22nd December 2009) – analysis of ASTM D6954 tier 1 aged samples of polypropylene.

This report describes the reduction in molecular weight of PP material containing Wells Plastics Reverte grade BD93470 when exposed to a combination of light and temperature over a period of just over 960 hours. It illustrates that the requirements of ASTM6954 Tier 1 (Mw <5000) standard are achieved within 960hrs of exposure, with the Mw falling from an initial value of 361,000 to 4275.

3. RAPRA report 47562 (16th April 2007) - testing of an oxodegradable LDPE film to ASTM D6954 tier 1.

This report describes the reduction in molecular weight of PE material containing Symphony Plastics d₂w additive when exposed to heat ageing.

4. RAPRA report 46303 (7th June 2006) – the comparative GPC analysis of aged and unaged polyethylene film samples.

This report describes the reduction in molecular weight of PE material containing Symphony Plastics d₂w additive when exposed to heat and light ageing.

5. Soil Burial Biodegradation Tests of Oxo-degradable Polyolefins; Final Report of a test programme for Symphony Environmental by University of Pisa.

This report describes a study of the environmental fate of oxo-biodegradable polyolefins in soil. It shows that the biodegradation of thermally oxidized oxo-biodegradable HDPE in soil proceeds at rates similar to that of natural rubber treated under the same conditions, that a certain level of oxidation, as promoted by abiotic pre-ageing (thermal and/or UV), will stimulate microbial attack, and that biodegradation can be significantly enhanced if the material is also submitted to the metabolic activity of a microbial species capable of secreting oxidative enzymes. This last result can be attributed to the increase of oxidation and degradation by the biological attack by the selected microorganisms once the oxidation has been promoted by the thermal ageing.

6. Chemical Industry Institute of Shihezi University, China, Report dated 08/12/2009. Test Report for the Biodegradation of BD Agricultural Film (Key Laboratory of Green Processing for Chemical Industry in the Xinjiang Production and Construction Corps. Sample supplied by: Xinjiang Kangrunjie Environmental Science and Technology Co., Ltd.)

This report describes the biodegradation of polyethylene agri-film containing Wells Plastics Reverte grade BD93253 after one growing seasons environmental exposure. The material was tested to ISO14855:1999; a controlled composting environment. The material was shown to achieve 77% conversion of carbon to CO₂ in 45 days. Please note: this report is in Chinese but an English translation is appended.

7. China National Centre for Quality Supervision & Test of Plastics Products, NTSQP report No. [2007]C0741-1 Testing of Oxo-Biodegradable Lunch Boxes to ISO14855:1999

This report describes the biodegradation of polypropylene lunch boxes containing Wells Plastics Reverte BD 93470 after pre-oxidation in a controlled composting environment. The test requirement was for 30% conversion of carbon to CO₂ hence the test was stopped when this was achieved. This material achieved 32.7% conversion in 126 days. Please note: this report is in Chinese but is overwritten with an English translation

8. Test Report from LGAI Technological Centre, S.A., Barcelona on degradation and mineralisation testing of a commercial plastic carrier bag (Symphony d₂w), carried out for Condis Supermercats S.A, Spain
9. Report from OWS to Symphony on ecotoxicity testing of d₂w products
10. Report from OWS to Symphony on ecotoxicity testing of d₂w products
11. Report No. 795-11046-1-T3 from Biosystems Atlanta to Wells Plastics on ecotoxicity testing of partially degraded commercial HDPE film according to the ecotoxicity requirements of tiers 2 and 3 of ASTM6954.
12. Report No. 795-11058-1-T3 from Biosystems Atlanta to Wells Plastics on ecotoxicity testing of partially degraded commercial LDPE film according to the ecotoxicity requirements of tiers 2 and 3 of ASTM6954.