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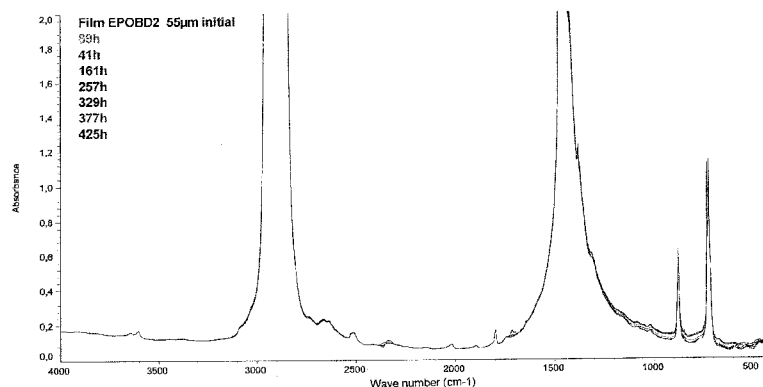
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(54) Title: OXO-BIODEGRADABLE ADDITIVES FOR USE IN FOSSIL FUEL POLYMER FILMS AND ONCE-USED PACKAGING

ENER Plastics - Sample EPOBD2 55µm
Analysis IRTF in transmission - Ventilated oven at 60°C

Figure: 1



(57) Abstract: The disclosed technology pertains to an Oxo-Biodegradation additive (OBD), composed of several chemical compounds, which when added to plastic polymers (such as polyethylene or polypropylene), imparts to them a property whereby they fragment and bio-assimilate after a programmed in-use life cycle, when exposed to sunshine and heat in the natural environment. The final i.e. end-user product, may be certifiable under the CNEP/SEESIB Protocols for Oxo-biodegradability or equivalent International Standard with a pass-fail criterion, to qualify being designated as an OBD product. The compounds described herein used with such polyolefin polymers include Stearates of Transition metals such as Manganese, Iron and Cobalt in varying combinations along with Calcium Carbonate and/or Titanium Dioxide balanced with a phenolic anti-oxidant to ensure a service life of a specified duration. Optionally, the compositions described in the application do not require the use of any bio-based substances.

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Title: OXO-BIODEGRADABLE ADDITIVES FOR USE IN FOSSIL FUEL POLYMER FILMS AND ONCE-USED PACKAGING

Related Applications

- 5 [0001] This Application claims priority to Canadian Patent Application No. 2,726,602 filed December 30th, 2010.

Field of the Invention

- [0002] The invention broadly falls in the technical field of Chemistry. More particularly the present invention is in the technical field of Oxo-
10 biodegradability of fossil fuel polymers. More specifically the invention relates to chemical compounds used in defined percentage composition to achieve Oxo-biodegradability of polyolefins such as polyethylene and polypropylene, as defined by a unique predictive 'pass-fail' criterion set out in accelerated laboratory testing procedures by France's leading Research Institutes-CNEP
15 and SEESIB.

Background of the Invention

- [0003] It was in the early 1930s that scientists discovered polyethylene, the first of the series in plastic polymer substances. Polyethylene was synthesized by polymerizing gaseous hydrocarbon ethylene. The product of
20 such polymerization resulted in a material, which has a high strength to weight ratio. It was found that this solid material offered many commercially interesting properties: it was cheap, durable, flexible and chemically resistant. Almost immediately after its discovery, the use of plastics has given rise to a variety of packaging products such as polyethylene and polypropylene plastic
25 films, bottles, cups, disposable snack food containers and such.

- [0004] Unfortunately, one of the main strengths of Polymers i.e. their long lasting strength and hydrophobicity became their greatest weakness as the world saw alarming increase in environmental pollution caused by virtually indestructible discarded plastic objects. Besides the fact that available landfill
30 space is becoming scarce, plastic poses many problems even after disposal as they tend to remain in the environment without degradation. It has been

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estimated that more than half of the manufactured synthetic polymers are used in packaging materials and 90% ultimately end up as garbage. The rate of accumulation of non-degradable plastic components is estimated to be 25 million tons per year. Most plastics available in the market are non biodegradable.

[0005] There is general agreement amongst the world-wide scientific community on the theory and results of photo and thermo oxidation on fossil fuel polymers. Extensive research over the last two decades has led to the discovery that polymers in presence of certain organic salts of transition metals, can accelerate the process of photo and thermo oxidation. A general chemical reaction for polyethylene degradation in the presence of organic salts of transition metals is shown in Figure 11, courtesy Centre Nationale d'Evaluation de Photoprotection (CNEP). The issue was to establish accelerated and predictive laboratory testing protocols to certify that a given recipe would perform as required/claimed, through commercially viable and usable testing procedures.

[0006] Until recently there was only one known guideline connected to Oxo-biodegradability of plastic polymers – known as the ASTM 6954. (*ASTM D 6954–04: Standard Guide for exposing and Testing Plastics that Degrade in the Environment by a Combination of Oxidation and Biodegradation*) However, ASTM 6954 itself states that it addresses test methods and procedures and is thus only a guideline recommended for comparative study for polymer performance on Oxo-biodegradation and therefore does not provide a pass/fail criterion or product specification for Oxo-biodegradability of products. A similar standard has been adopted by the British Standards Institute, BSI earlier this year. (*BS 8472:2011 - Methods for the assessment of the oxo-biodegradation of plastics and of the phyto-toxicity of the residues in controlled Laboratory conditions.*)

[0007] This has led to a proliferation of self-claimed Oxo-biodegradable additives (containing organo-metallic with or without bio-based products) being marketed world-wide. While the approach of self-claiming bio-

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degradability has been a successful marketing tool for retailers to establish their 'eco-friendliness' to their clients, the products do not necessarily perform as such. There are no specifics as to the time required by them to fragment or bioassimilate. In fact the absence of acceptable scientific data on predictability
5 of performance results, such self-claims have negatively impacted on the fossil fuel based plastic industry being maligned as perpetuating false claims to further their commercial agendas.

[0008] Fortunately a logical and scientific approach to establish an accelerated laboratory test protocol for the abiotic and biotic degradation of
10 fossil fuel polymers has been developed by the Centre Nationale d'Evaluation de Photoprotection (CNEP) and Synthèse et Etudes de Systèmes à Intérêt Biologique (SEESIB) (CNEP/SEESIB Protocol). These protocols are consistent with actual field behavior observed over nearly two decades at CNEP/SEESIB, and was verified independently by other Researchers (See
15 for example Andrea Corti et. al.: *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 (2010) 1106-1114, herein incorporated by reference.)

Summary of the Invention

20 **[0009]** The present disclosure provides compositions and information relating to a balanced recipe of pro and anti oxidant additives, for use in polymer films and packaging material. In one embodiments, the disclosure provides compositions that predict the final product to qualify as Oxo-biodegradable under scientifically based 'Pass-Fail' test protocols in an effort
25 to solve the issue of certifiable Oxo-biodegradability when compared to self-claimed Oxo-biodegradable products in the markets. The disclosure provides chemical compounds which when added in defined percentage to conventional fossil fuel based polymers accelerate their degradation process after a pre-established 'in-use' life.

30 **[0010]** In one aspect, the end-user product (a polymer to which the developed additive or composition is added at a recommended dosage)

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containing one or more of the chemical components is then subjected to predictive test protocols developed by Centre National d' Evaluation de Photo protection (CNEP) and Synthese et Etude des Systemes á Interet Biologique (SEESIB), which would guarantee a desired service life at full mechanical
5 properties; thereafter to fragment within a few months and subsequently to oxo-biodegrade in a 2-3 years span, on exposure to normal environmental conditions. The CNEP/SEESIB's testing protocol, based on macro-molecular considerations, is currently the only accelerated pass/fail protocol where laboratory results are reflective of actual environmental conditions and bio-
10 assimilation time frame; established by them over a two decade study of actual environmental behavior versus accelerated laboratory testing, measuring and predicting such behavior.

[0011] In one embodiment, there is provided a composition comprising
i) a pro-oxidant comprising one or more organo-metallic stearates selected
15 from iron, manganese and cobalt; ii) one or more phenolic antioxidants; iii) a polymer base; and iv) an inert filler. In one embodiment, the composition is master batch or an additive master batch. In one embodiment, the master batch is a solid. In one embodiment, the master batch is useful for imparting properties to plastics, such as polymer based plastics.

[0012] In one embodiment, the composition comprises a polymer base. Optionally, the polymer base is a polyolefin, such as polyethylene or polypropylene. In one embodiment, the composition comprises a pro-oxidant such as organo-metallic stearates of Iron, Manganese and Cobalt. In one embodiment, the term "organo-metallic stearates" refers to compounds having
25 the formula $(C_{18}H_{35}COO^-)_2 X$, where X can be iron, cobalt or manganese. In one embodiment, the pro-oxidant consists of metallic stearates of Iron, Manganese and Cobalt. Optionally, the pro-oxidant comprises from about 15 to about 30 percent by weight of the total composition.

[0013] In one embodiment, the composition comprises a phenolic
30 antioxidant. Examples of suitable phenolic antioxidants for use with the composition described herein are marketed under trade names IRGANOX

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1010; RICHNOX 1010 and EVERNOX-10. In one embodiment, the phenolic anti-oxidant has the chemical formula of $C_{73}H_{108}O_{12}$. Optionally, the phenolic antioxidant comprises from about 10 to about 20 percent by weight of the total composition.

5 **[0014]** In another embodiment, the compositions provided herein include one or more inert fillers. In one embodiment, the inert filler is Calcium carbonate and/or Titanium Dioxide.

[0015] The present disclosure includes articles made using the compositions described herein. For example, one embodiment includes
10 polymer-based film or containers suitable for use as packaging prepared by diluting the compositions described herein. In one embodiment, the polymer-based film or containers suitable for use as packaging are made by diluting the compositions, such as a master batch, to about 1- 5 percent into a polymer resin. In one embodiment, the polymer resin is a polyolefin carrier
15 resin. In one embodiment, the articles made using the compositions described herein are Oxo-biodegradable products which meet the requirements of CNEP/SEESIB protocols. In one embodiment, the polymer-based film or container is an Oxo-biodegradable product which meets the requirements of CNEP/SEESIB protocols.

20 **[0016]** In one embodiment, the compositions and articles described herein include colour pigments and/or other additives known to a person of skill in the art.

[0017] Also provided are polymer-based films or containers used for any kind of packaging/carriage materials comprising the composition
25 described herein.

[0018] Also provided are methods for producing the compositions described herein comprising mixing a pro-oxidant with one or more phenolic antioxidants, a polymer base and an inert filler. Also provided are methods for producing an oxo-biodegradable product, such as a polymer-based film or

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container, comprising diluting the compositions described herein with a polymer base.

[0019] One aspect of the disclosure is a recipe for an oxo-biodegradable (OBD) master batch, consisting of 15 to 30 percent by weight
5 of a mix of organo-metallic stearates of Iron, Manganese and Cobalt, along with a phenolic antioxidant at 10 to 20 percent with the balance being a pure polymer with an inert filler like calcium carbonate and/or Titanium Dioxide.

[0020] Another aspect of the disclosure is to prepare and submit articles, such as films/ disposable food trays etc., identical to those in actual
10 everyday use by consumers, for testing under the CNEP/SEESIB protocols, in a shape and form as near to the actual article used by consumers in the market place (which ensures that the final product will behave as predicted in the accelerated lab tests under the protocols.). This involves diluting the OBD master batch at a specified dosage of between 1% to 5% into the same
15 polyolefin polymers that are in actual use by convertors and preparing samples that replicate the end-use, in thickness and weight.

[0021] Other features and advantages of the present invention will become apparent from the following detailed description. It should be understood, however, that the detailed description and the specific examples
20 while indicating preferred embodiments of the invention are given by way of illustration only, since various changes and modifications within the spirit and scope of the invention will become apparent to those skilled in the art from this detailed description.

Brief Description of the Drawings

25 [0022] Embodiments of the invention will now be described in relation to the drawings in which:

[0023] Figure 1 shows 55 μm EP OBD-2 film thermo-oxidized at 60 degrees Celsius characterized using FTIR for up to 425 hours.

[0024] Figure 2 shows 55 μm EP OBD-2 film thermo-oxidized at 60
30 degrees Celsius characterized using FTIR for up to 425 hours.

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[0025] Figure 3 shows absorbance at 1719 cm⁻¹ as a function of time (duration) for EPOBD2 samples.

[0026] Figure 4 shows FTIR of EP OBD-2 film exposed under SEPAP12/24 (60 degrees Celsius) conditions for up to 100 hours.

5 **[0027]** Figure 5 shows FTIR of EP OBD-2 film exposed under SEPAP12/24 (60 degrees Celsius) conditions for up to 100 hours. Absorbance at 1719 cm⁻¹ generally increases with the duration of treatment.

[0028] Figure 6 shows FTIR of samples of film containing EP OBD-2 pre-exposed in SEPAP 12/24 for 20 hours, and thermo-oxidized at 60
10 degrees Celsius in the aerated oven.

[0029] Figures 7 shows FTIR of samples of film containing EP OBD-2 pre-exposed in SEPAP 12/24 for 20 hours, and thermo-oxidized at 60 degrees Celsius in the aerated oven. Absorbance at 1719 cm⁻¹ increase with the duration of thermo-oxidation.

15 **[0030]** Figure 8 shows FTIR characterizing the chemical evolution of EP OBD-2 film after 70 hours of exposure in SEPAP 12/24 until fragmentation, after 96 hours of thermo-oxidation and the initial state of the film.

[0031] Figure 9 shows FTIR characterizing the chemical evolution of
20 EP OBD-2 film after 70 hours of exposure in SEPAP 12/24 until fragmentation, after 96 hours of thermo-oxidation and the initial state of the film. Absorbance at 1715 cm⁻¹ is highest in the 96h E60C sample, followed by 70h SEPAP while the initial sample shows only a relatively small absorbance at ~1715 cm⁻¹.

25 **[0032]** Figure 10 shows variations of the ATP concentration expressed in p mol/ml vs. the biotest duration.

[0033] Figure 11 shows a general chemical reaction for polyethylene degradation in the presence of organic salts of transition metals.

Detailed Description of the Invention

[0034] In the following description, certain specific elements are provided in order to understand the various disclosed aspect of the technology. However, a person skilled in the art who reads the disclosure, the
5 figures and the claims will realize that the disclosed embodiments can be practiced with or without one or more of these specific details or with other elements without deviating from the scope of the inventions.

[0035] The recipe is for an Oxo-biodegradable (OBD) master batch, consisting of 15 to 30 percent by weight of a mix of organo-metallic stearates
10 of Iron, Manganese and Cobalt along with 10 – 20 percent phenolic antioxidant with the balance being a pure polymer along with an inert filler like calcium carbonate and/or Titanium Dioxide, which will ensure in-use mechanical integrity of 1 year.

[0036] The aforementioned OBD master batch is then diluted between
15 1-5 percent into the same polyolefin polymers. This process facilitates the replication of samples, such as films/ disposable food trays etc for end-use in thickness and weight. The final sample, which resembles the actual article used by consumers in the market place, may then be subjected to the accelerated lab tests under the protocols. For reference, in one embodiment
20 the final sample is termed as EP OBD2 (trade name).

[0037] In one aspect there is provided a composition comprising: i) a pro-oxidant comprising one or more organo-metallic stearates selected from iron, manganese and cobalt; ii) one or more phenolic antioxidants; iii) a polymer base; and iv) an inert filler. In one embodiment, the composition is an
25 additive master batch.

[0038] In one embodiment, the composition comprises a pro-oxidant consisting of organo-metallic stearates selected from iron, manganese and cobalt. In one embodiment, the composition comprises a polymer base such as a polyolefin. In one embodiment, the polyolefin is polyethylene. IN one
30 embodiment, the polyolefin is polypropylene.

[0039] In one embodiment, the composition comprises a pro-oxidant comprising metallic stearates of Iron, Manganese and Cobalt from about 15 to about 30 percent by weight of the total composition. In one embodiment, the composition comprises a phenolic antioxidant from about 10 to about 20
5 percent by weight of the total composition. In one embodiment, the composition includes an inert filler such as calcium carbonate and/or titanium dioxide.

[0040] In one embodiment, there is provided a polymer-based film or container suitable for use as packaging prepared by diluting a composition
10 described herein from 1 to about 5 percent into a polyolefin carrier resin.

[0041] In one embodiment, the polymer-based film or container is an Oxo-biodegradable product which meets the requirements of CNEP/SEESIB protocols.

[0042] In one embodiment, the compositions described herein may also
15 include colour pigments and/or other additives. In one embodiment, the polymer-based film or container described may also include colour pigments and/or other additives.

[0043] In one embodiment, there is provided a polymer-based film or container used for any kind of packaging/carriage materials comprising the
20 composition described herein.

[0044] A summary of the CNEP/SEESIB experimental protocol is described hereunder:

1. Evaluation of Abiotic Oxidabilities

[0045] **1.1** – Since the pro-oxidants introduced in the polymers tend to
25 initiate immediately in contact with the environment, it is necessary to 'control' this rapid oxidation step so that the final article can be used over a time span for the purpose it was originally made. This is achieved by balancing the composition or recipe by the addition of additional anti-oxidants. Thus to ensure and guarantee a useful life, say of one year, a film prepared from a
30 polymer containing the OBD additive, at a pre-selected mix ratio, should

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show that it will not be thermo-oxidized when exposed in an aerated oven at 60°C for 400 hours. This is established by observing the absorbance curve of the oven-exposed film in a Fourier Transform Infrared Spectroscopy (FTIR) where an increase at a wave number of 1715 cm⁻¹ should not be higher than 5 (x/1000) (where x is the film thickness in microns).

[0046] 1.2 - Thereafter, having served its useful life, the test specimen requires to demonstrate that it will start to fragment rapidly. For this a film sample (containing the OBD additive), is required to be exposed in a SEPAP 12/24 unit for 150 hours, operating under the following experimental 10 conditions:

- the incident light emitted by 4 medium pressure mercury arcs and filtered by the borosilicate envelope of the 4 lamps does not contain any wavelength shorter than 290 nm ;
- the temperature of the exposed surface is controlled and 15 maintained at (60±1)°C. -no external water is allowed to come into contact with the exposed sample. However, internal water is formed in the matrix through the decomposition of primary hydro-peroxides.

[0047] After an exposure of 150 hours, the film needs to demonstrate an absorbance increase equal or higher than (x/100) in an FTIR operating in 20 the transmission mode. Achieving this level confirms that the film will fragment within 3 to 4 months after the completion of its in-use life.

[0048] 1.3 - To establish the state of the specimen in terms of its total fragmentation and the destruction of its Hydro-carbon chain, the photo-oxidized film needs to be thermo-oxidized in the dark, such that the increase 25 in absorption seen on the FTIR operating in the transmission mode is equal to or higher than (2x/100). Since the antioxidants added to the film to ensure a given in-use life, needs to be first eliminated (so that it assumes a state identical to what it would achieve under photo-oxidative conditions in sunlight.), a pre-exposure of the specimen for a short duration in a SEPAP 30 12/24 is recommended.

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[0049] To follow the Protocol procedure, a fresh film, identical to that used above, is:

- 5 - exposed briefly (less than 30 hours) in the SEPAP 12/24 to convert the stabilizers (such as anti-oxidants both in the virgin polymers and in the OBD additive) into inert substances.
 - the SEPAP 12/24 pre-exposed film is then exposed to thermo oxidation at 60°C in an aerated oven for 300 hours. To satisfy the Protocol criteria the film should demonstrate an increase in absorbance equal to or higher than 2x/100 when examined in an FTIR run in the transmission mode.
- 10

[0050] In summary, the abiotic protocols require that for a film to be considered Oxo-biodegradable i.e. meet the 'pass/fail' criterion for Oxo-biodegradation, it (a). should not oxidize and the absorbance should not be more than (x/1000) (b). be oxidized to an extent equal to (x/100) (exposure to daylight) + (2x/100) (soil thermo oxidation) + (3x/100) to acquire biodegradability.

15

2. Evaluation of Biodegradability.

[0051] 2.1 - Pre-oxidation treatment

[0052] A large surface of the specimen film requires to be exposed in SEPAP 12/24 under identical conditions as set out by the Protocol for Abiotic conditions at A(b) above. The results must demonstrate that the increase in absorbance has been a minimum of x/100.

20

[0053] The photo-oxidized film is submitted to thermo-oxidation exposure under identical test parameters as set out in the Protocol for Abiotic conditions at A(c) above. The results must demonstrate an increase in absorbance of a minimum of 2x/100.

25

[0054] 2.2 - Biodegradability assay

[0055] For the biodegradability assay, the sample needs to be uniform and of a smaller particle size. Thus the already highly fragmented and very

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fragile film from step B (a) above, are pressed through a sieve with hole diameters about 1 mm. For the incubation with microorganism a conventional mineral medium of a composition given in the Biotic Protocols need to be followed.

- 5 **[0056]** The powdered specimen is then placed in several glass vials and flasks in measured quantities, along with specified volumes of the liquid medium, to which is added defined amount of microorganism or spores and allowed to incubate under specified temperature and controlled agitation, and the incubation is allowed to proceed for 180 days.
- 10 **[0057]** Periodically a single vial is removed and tested for microbial activity through an ATP assay conducted by a series of tests under optical microscopy, FTIR spectrophotometry, Gel Permeation Chromatography (GPC) and electron microscopy (as detailed in the CNEP/SEESIB Protocols).

Results of Abiotic and Biotic Tests Conducted on EPOBD-2 (trade-name)

- 15 **[0058]** **Evaluation of the Thermo-oxidability**
- [0059]** EP OBD-2 film thermo-oxidized in aerated oven at 60°C during 425 hours and film was characterized with FTIR during 0, 41, 89, 161, 257, 329, 377 and 425 hours. The only spectral variations observed were assigned to the loss of stearic acid associated to the Fe (III) stearate which absorbed at
- 20 1714 and 1697cm⁻¹. This guarantees a minimum one year lifetime in storage and use without mechanical detriment. (See Figures 1, 2 and 3)

[0060] $\Delta \text{abs } 1710 \text{ cm}^{-1} = \text{zero}$ (after 425 hours of exposure in the aerated oven).

[0061] Evaluation of the Photo-oxidability

- 25 **[0062]** EP OBD-2 film exposed in experimental conditions of SEPAP 12/24, temperature of exposed surface 60°C, no external water brought on the exposed samples. The film was exposed for 100 hours and chemical evolution was characterized with FTIR during 0, 10, 20,40,60,80, and 100

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hours. After 100 hours of exposure the absorbance increase were: (see Figures 4, 5 and 3)

[0063] $\Delta \text{abs } 1710 \text{ cm}^{-1} = 2.04 (\text{X}/100)$ (after 100 hours of exposure in SEPAP 12/24).

- 5 **[0064]** This guarantees that the film, accidentally scattered into the environment, should fragment after approximately 3 month of exposure to sunlight.

Evaluation of the Total Fragmentation and Destruction of the Hydrocarbon Chain.

- 10 **[0065]** The film containing EPOBD-2 was first pre-exposed in SEPAP 12/24 and after 20 hours of exposure, the sample was submitted to thermo-oxidation at 60°C in the aerated oven. After 140 hours of thermo-oxidation the absorbance increase observed on the FTIR was: (See Figures 6, 7 and 3)

15 **[0066]** $\Delta \text{abs } 1710 \text{ cm}^{-1} = 1.92 (x/100)$ (after 140 hours of thermo-oxidation)

[0067] This ensures that film will achieve biodegradability in soil in 2-3 years.

Evaluation of Biotic Degradation.

20 **[0068]** The biotest included in the CNEP-SEESIB protocol was carried out on the oxidized particles prepared from the film EP OBD2 through pre-exposure in SEPAP 12/24 for 70 hours (until fragmentation) and the fragments thermo-oxidized for 96 hours at 60°C. (See Figures 8 & 9), present the FTIR spectra which characterized the chemical evolution of EP OBD2 film after 70 hours of exposure in SEPAP 12/24, until fragmentation
25 (red spectrum) and after 96 hours of thermo oxidation of the photo-oxidized fragments (green spectra). The blue spectrum characterized the initial state of (EP OBD2) film whose thickness was 55 μm .

[0069] The absorbance increases at 1715 cm^{-1} were respectively 1.35 after exposure in SEPAP 12/24 and 2.00 after 96 hours of thermo-oxidation

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at 60°C. The extent of oxidation of the particles, determined by the absorbance increase at 1715 cm^{-1} equal to 3.63 (x/100), was high enough for carrying out the biotest. The oxidation particles were stored in sterile vials at 4°C.

5 **[0070]** The evaluation of the acquired biodegradability of the oxidized particles was based on the SEESIB protocol.

[0071] The plots presented in Figure 10 illustrates the variations of the ATP concentration expressed in p mol/ml vs. the biotest duration expressed in day (up to 180 days)

10 - the black plot illustrates the variation of the ATP concentration (i.e. of the population of active cells) in the medium in which the oxidized polymer particles were added. After a fast development of the cell population (in 4 days), a stationary state was observed until 120 days and a new development of the cell population was observed between 120
15 and 180 days;

- the dotted red plot illustrates the variation of the ATP concentration in the medium in which no oxidized polymer particles were added. The dotted plot evidenced the fact that no increase in the cell population was observed, the initial population of cell being
20 around 10^4 cells per each ml of solution.

[0072] The comparison between the black plot and the dotted red plot demonstrated that the cell population developed using the oxidized polymer as the sole source of carbon, while the cells were not surviving in the absence of the oxidized polymer. Under the SEESIB Protocols the
25 biodegradability of the oxidized polymer is certified when the ATP content in the medium containing the polymer is at least 4 times higher than the ATP content in the medium containing no polymer, both incubated under the same conditions. The results show that EP OBD-2 confirms to the SEESIB Protocols and is certified thereunder.

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[0073] It should also be noted that the results obtained in that biotest also show that the pro-oxidant additives introduced in the polymer matrix and the oxidized groups formed onto the macromolecular chain, did not present any toxicity towards *Rhodococcus rhodochrous*.

5 **[0074]** The unique combination of aforementioned components in defined percentage has made EP OBD-2 fully oxo-biodegradable under the CNEP/SEESIB protocols for Oxo-biodegradability and fully qualifies for being designated as an OBD product.

10 **[0075]** It has to be appreciated that the various changes, rearrangements and modifications can be made therein, without departing from the scope of the present invention and it is obvious that, within the scope of the invention, many disparities are possible for a person skilled in the art. The scope of the invention is not limited to the description hereinabove. The invention embodies in each novel element and each combination of elements.

15 **[0076]** While the present invention has been described with reference to particular embodiments and examples, the invention is not limited to the disclosed embodiments and examples. The invention is intended to cover various modifications and equivalent arrangements included within the spirit and scope of the appended claims.

20 **[0077]** All publications, patents and patent applications are herein incorporated by reference in their entirety to the same extent as if each individual publication, patent or patent application was specifically and individually indicated to be incorporated by reference in its entirety.

Claims:

1. A composition comprising:
 - i) a pro-oxidant comprising one or more organo-metallic stearates selected from iron, manganese and cobalt;
 - ii) one or more phenolic antioxidants;
 - iii) a polymer base; and
 - iv) an inert filler.
2. The composition of claim 1, wherein the composition is an additive master batch.
3. The composition of claim 2, comprising a pro-oxidant consisting of organo-metallic stearates selected from iron, manganese and cobalt.
4. A composition according to any one of claims 1 to 3, wherein the polymer base is a polyolefin.
5. A composition according to claim 4, wherein the polyolefin is polyethylene.
6. A composition according to claim 4, wherein the polyolefin is polypropylene.
7. A composition according to any one of claims 1 to 6, wherein the pro-oxidant comprising metallic stearates of Iron, Manganese and Cobalt is from about 15 to about 30 percent by weight of the total composition.
8. A composition according to any one of claims 1 to 7, wherein the phenolic antioxidant is from about 10 to about 20 percent by weight of the total composition.
9. A composition according to any one of claims 1 to 8, wherein the inert filler is calcium carbonate and/or titanium dioxide.

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10. A polymer-based film or container suitable for use as packaging prepared by diluting the composition according to any one of claims 1 to 9 from about 1 to about 5 percent into a polyolefin carrier resin.

11. The polymer-based film or container according to claim 10, wherein the polymer-based film or container is an Oxo-biodegradable product which meets the requirements of CNEP/SEESIB protocols.

12. A composition according to any one of claims 1 to 9 which may also include colour pigments and/or other additives.

13. The polymer-based film or container of claims 10 or 11, which may also include colour pigments and/or other additives.

14. A polymer-based film or container used for any kind of packaging/carriage materials comprising the composition of any one of claims 1 to 10 or claim 12-13.

ENER Plastics - Sample EPOBD2 55µm
Analysis IRTF in transmission - Ventilated oven at 60°C

Figure:

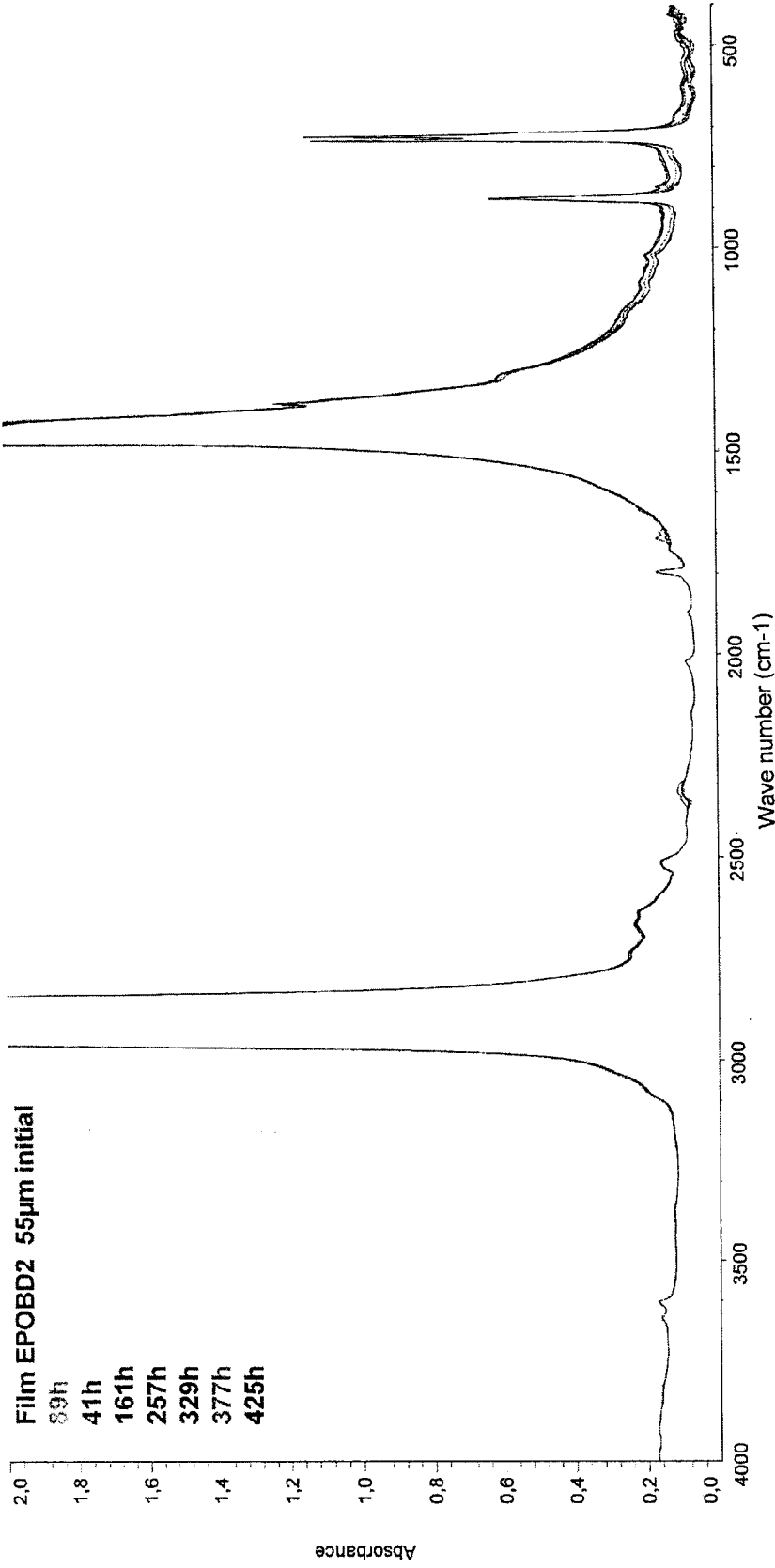


Figure: 2

ENER Plastics - Sample EPOBD2 55µm
Analysis IRTF in transmission - Ventilated oven at 60°C

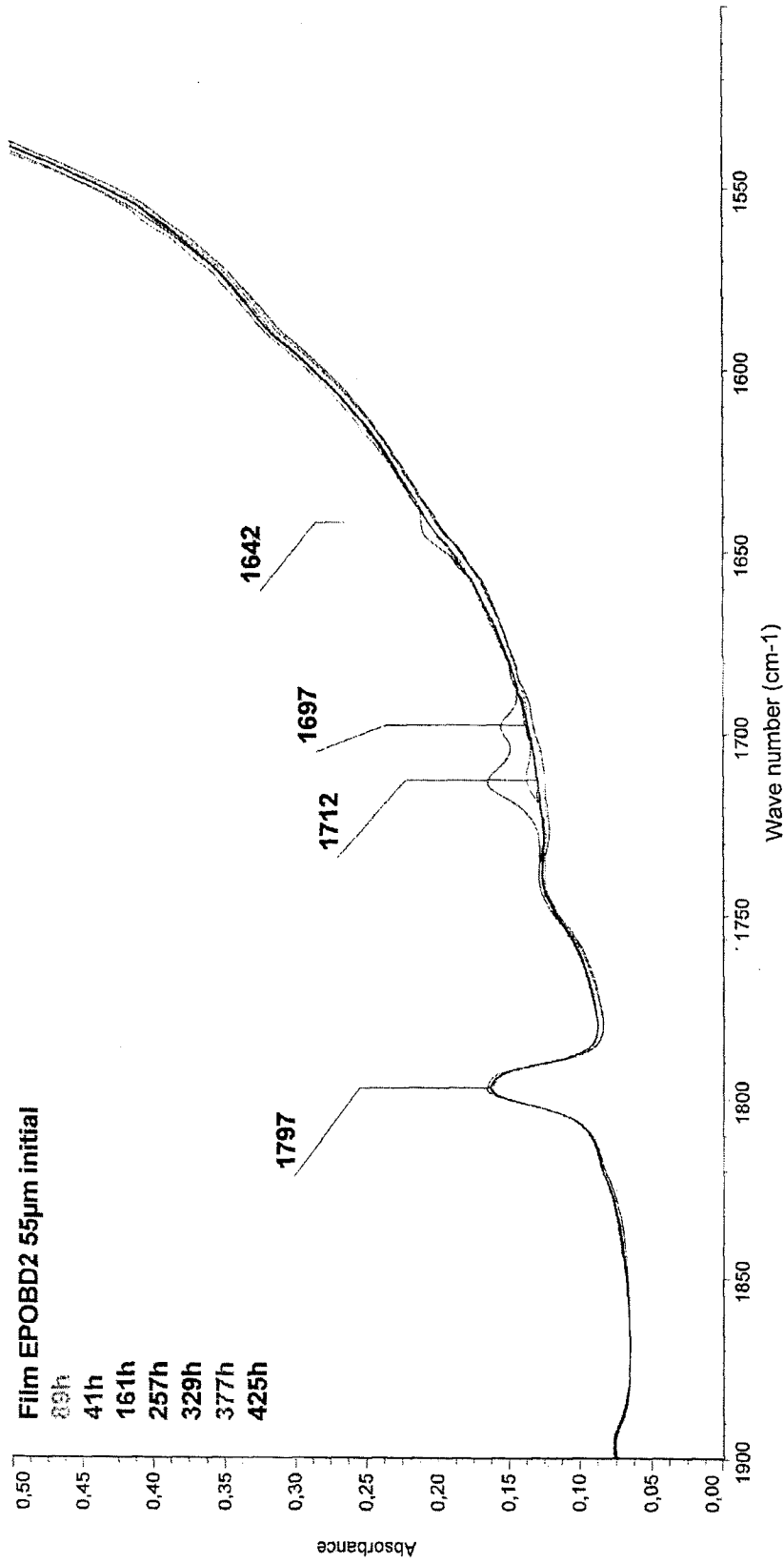
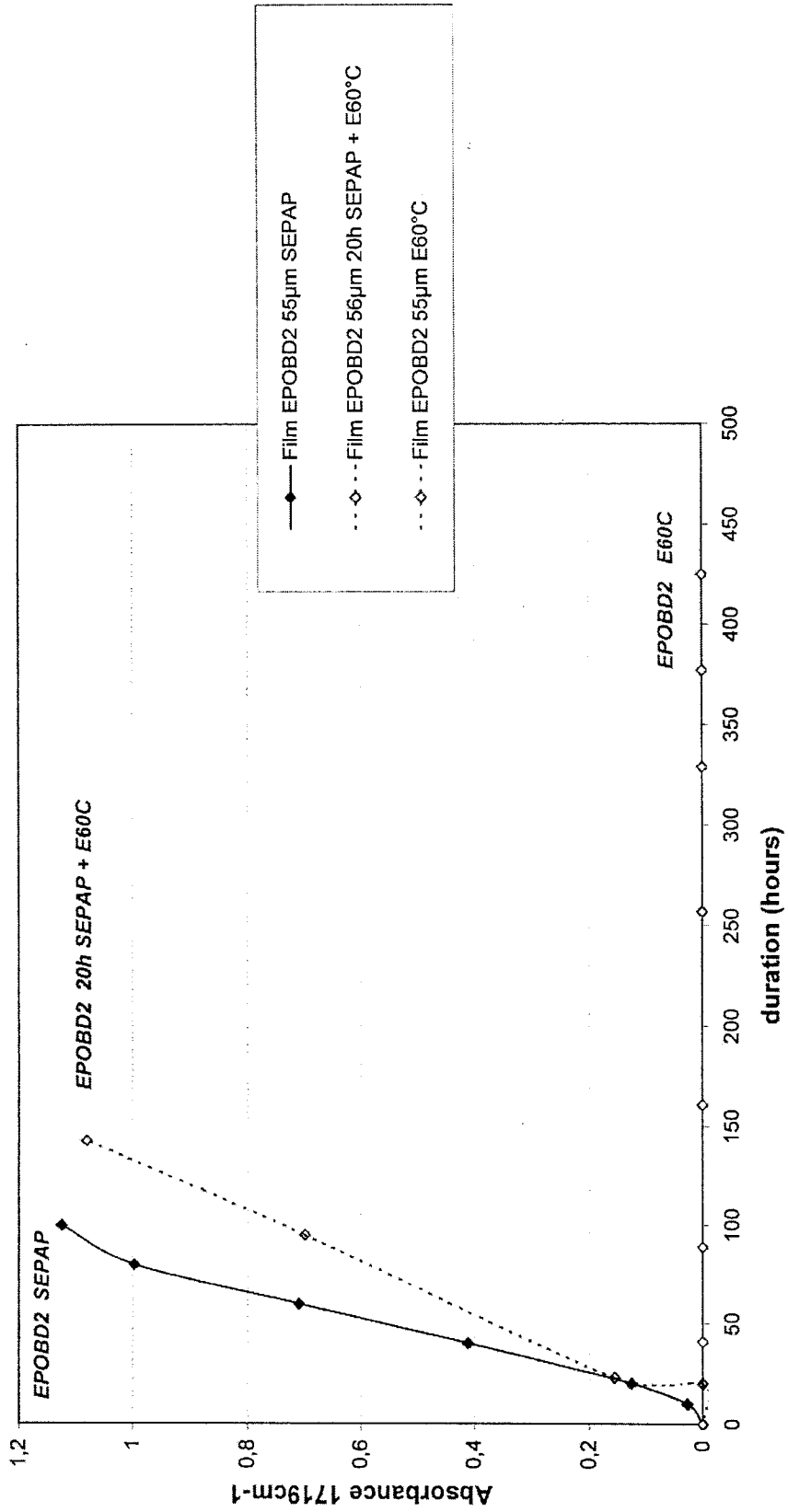


Figure: 3

ENER Plastics - Sample EPOBD2



4
Figure:

ENER Plastics - Sample EPOBD2 55µm
Analysis IRTF in transmission - Irradiation in SEPAP 12/24 60°C unit

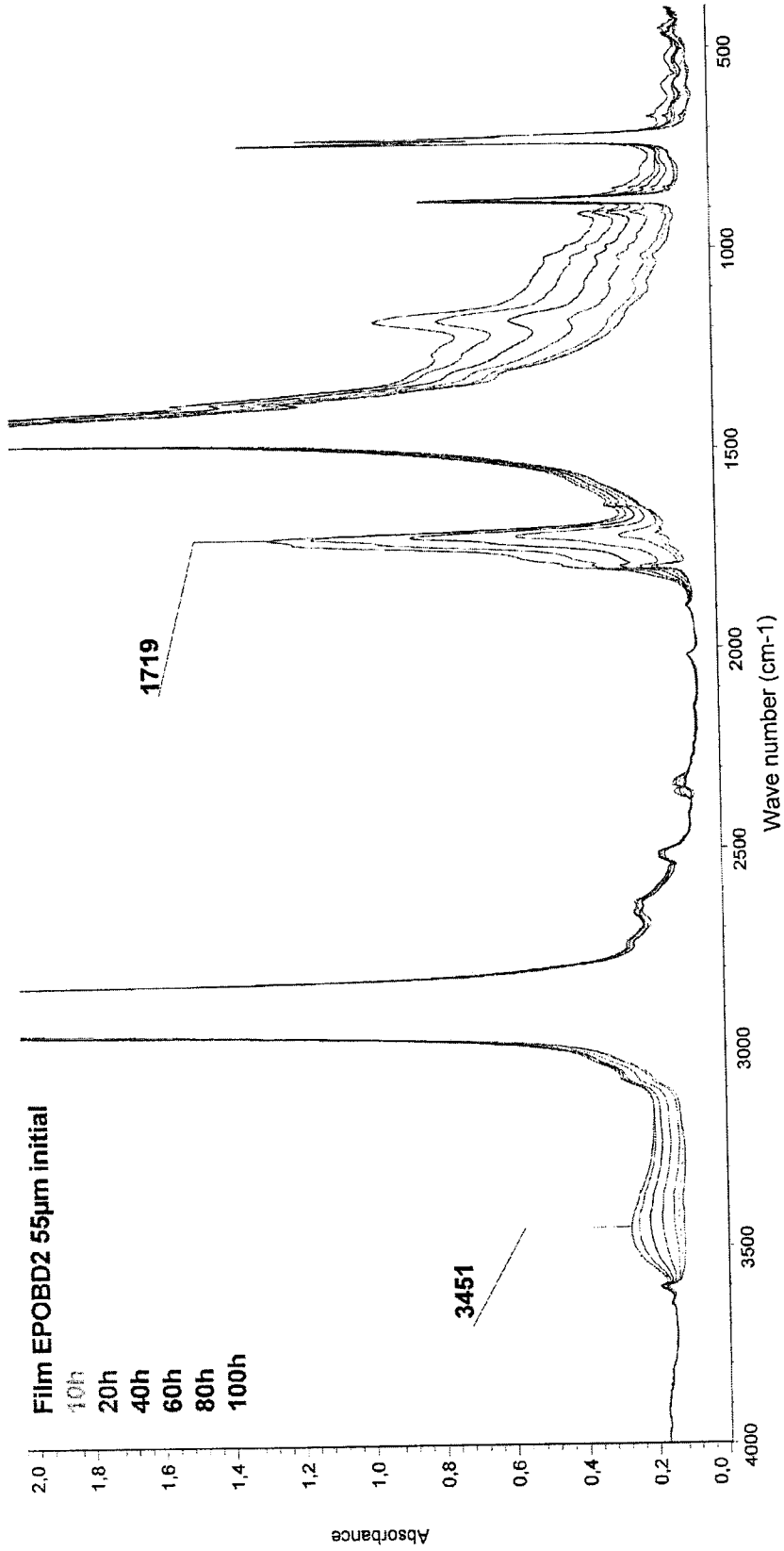


Figure: 5

ENER Plastics - Sample EPOBD2 55µm
Analysis IRTF in transmission - Irradiation in SEPAP 12/24 60°C unit

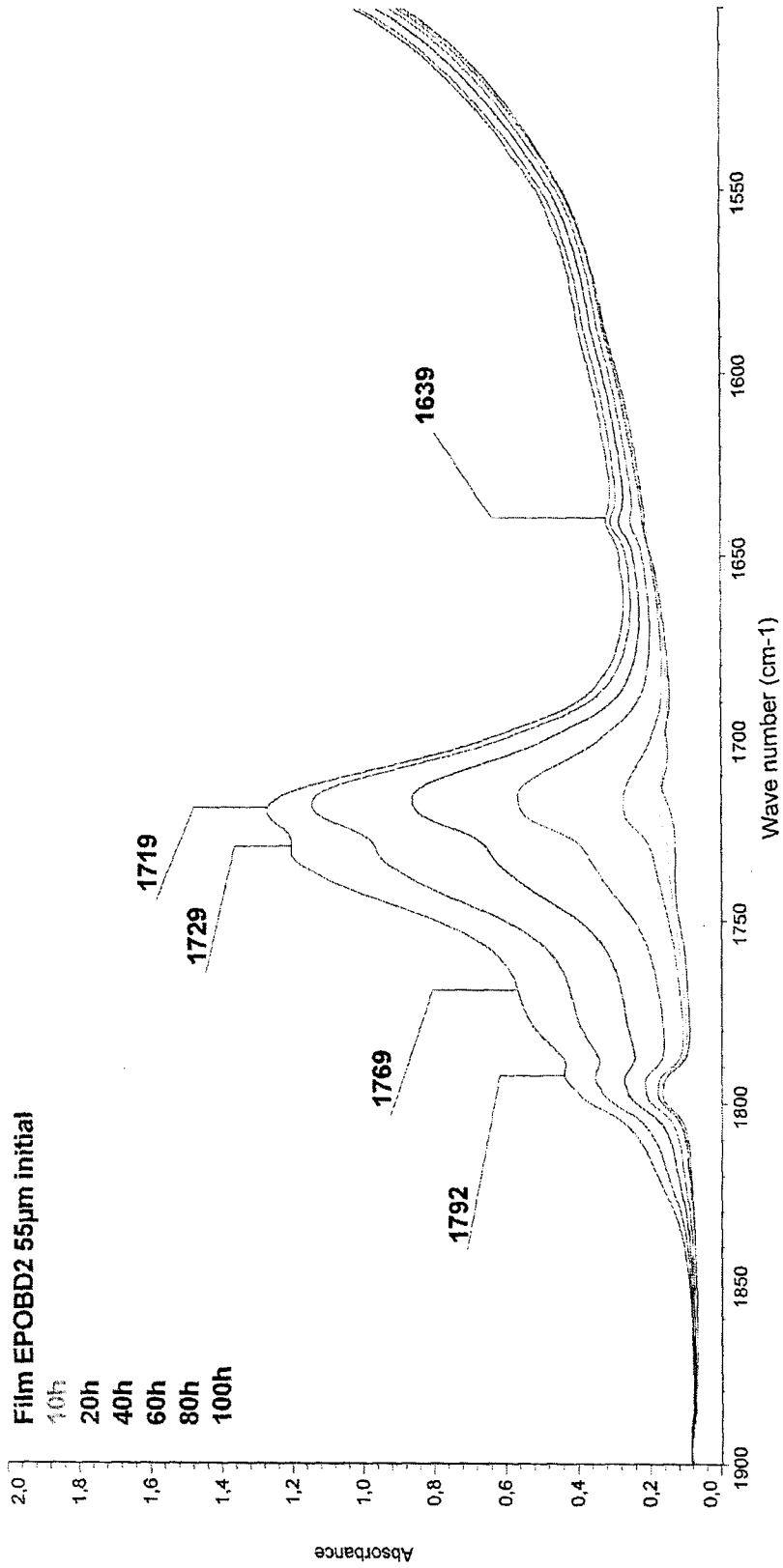


Figure: 6

ENER Plastics - Sample EPOBD2 55 μm

Analysis IRTF in transmission - 20h irr.in SEPAP 12/24 unit and Ventilated oven at 60°C

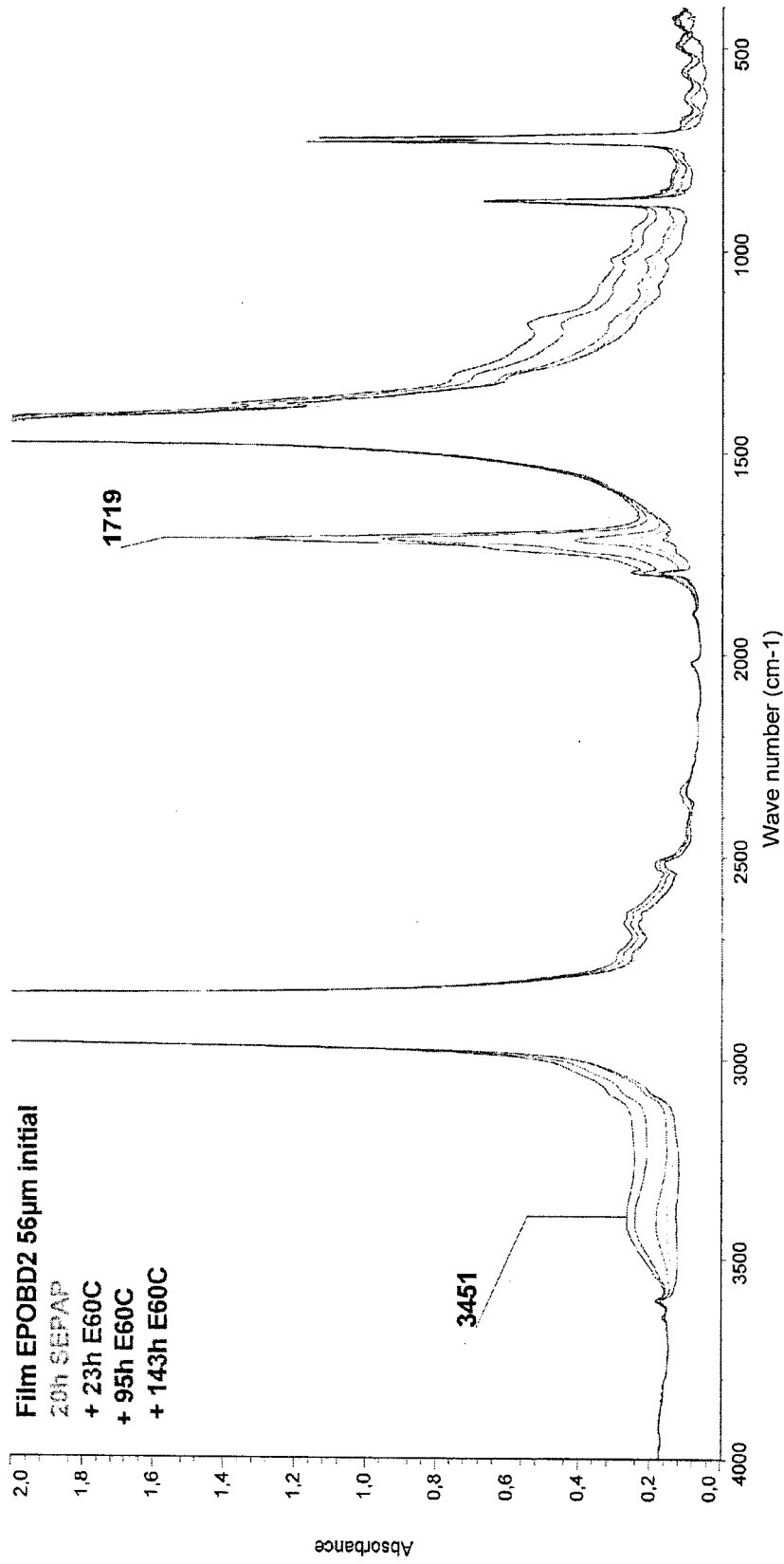


Figure: 7
ENER Plastics - Sample EPOBD2 55µm
Analysis IRTF in transmission - 20h irr.in SEPAP 12/24 unit and Ventilated oven at 60°C

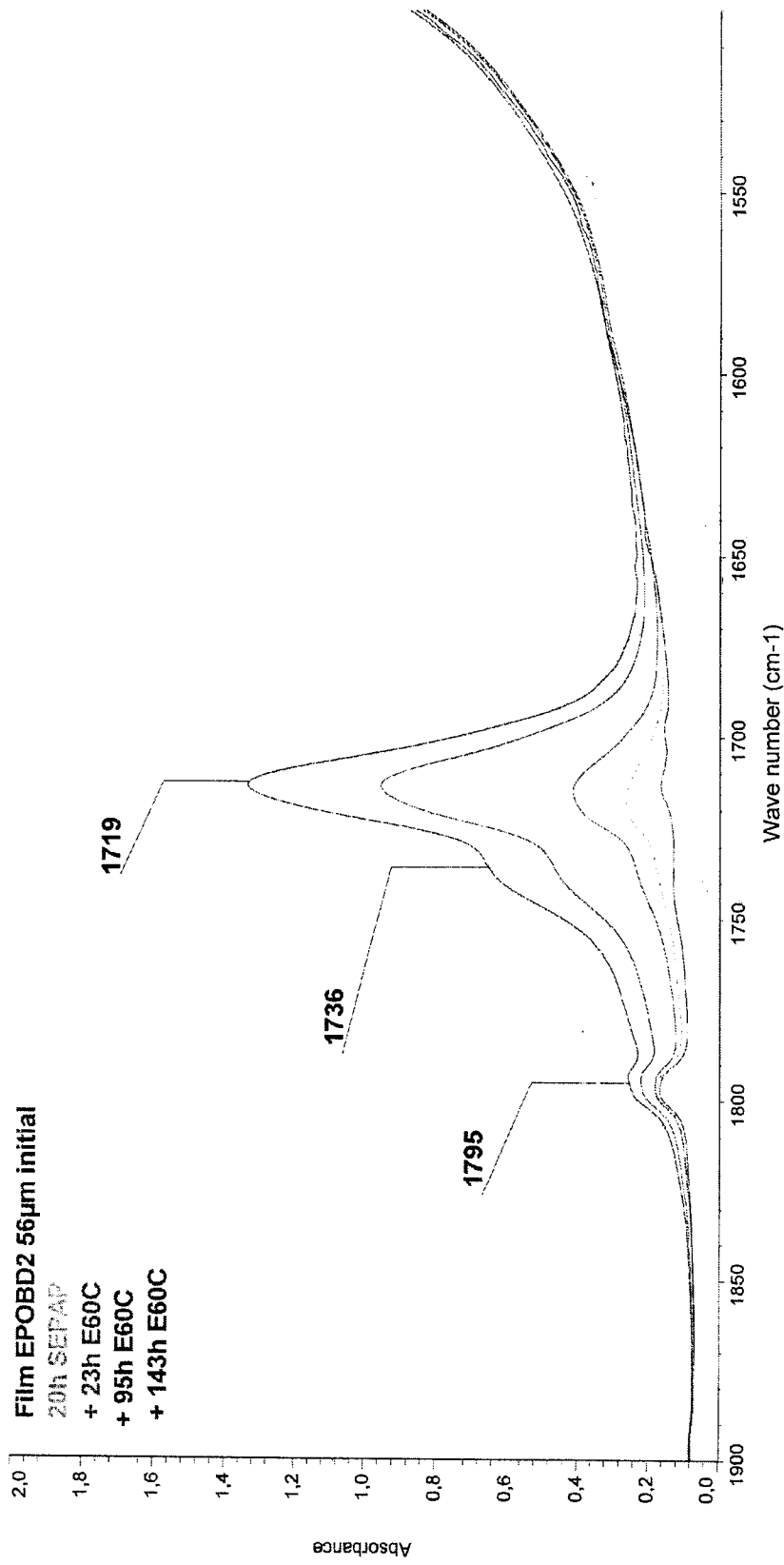
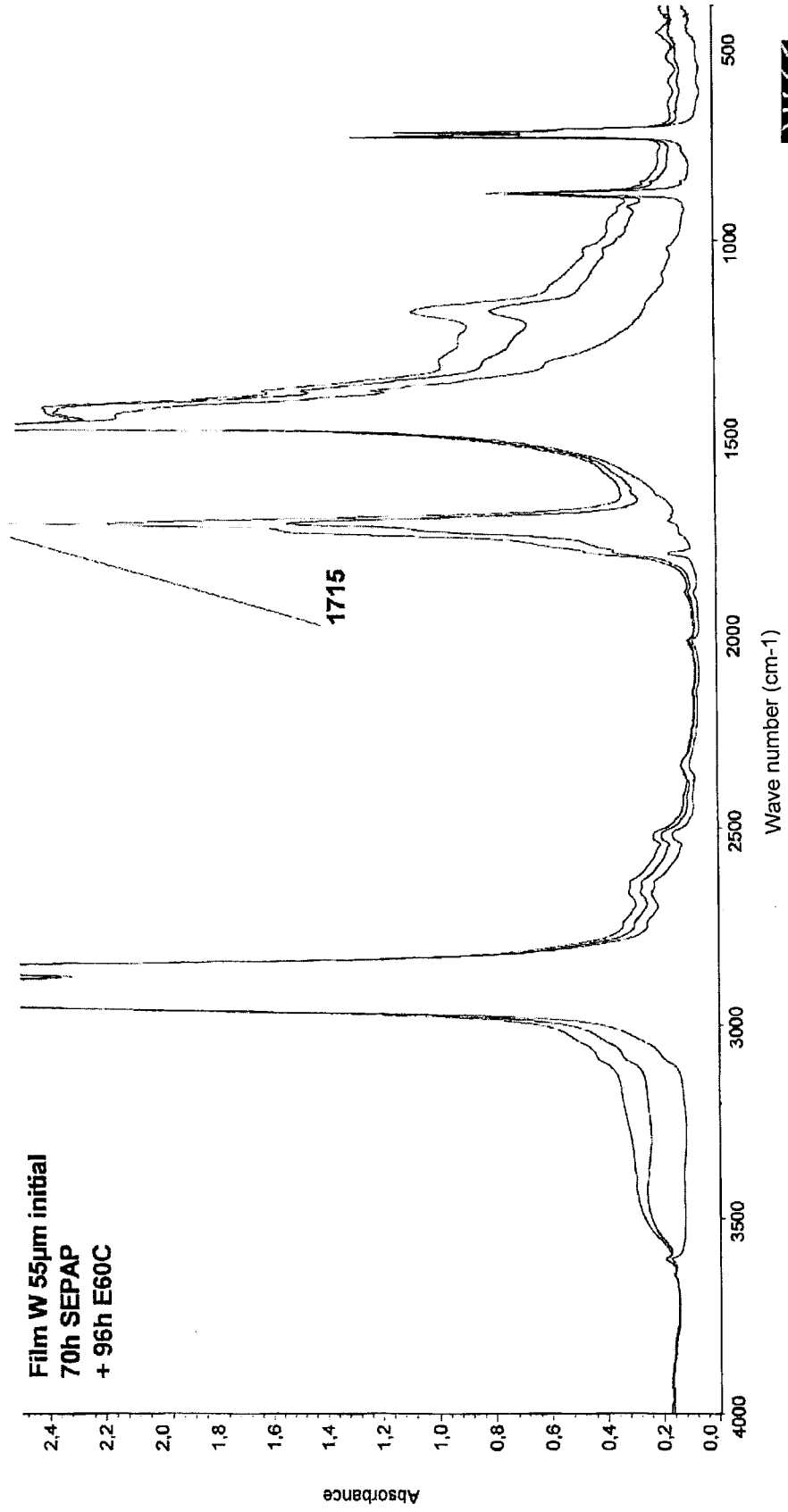


Figure 8

EP-ODB2 ENER plastics -

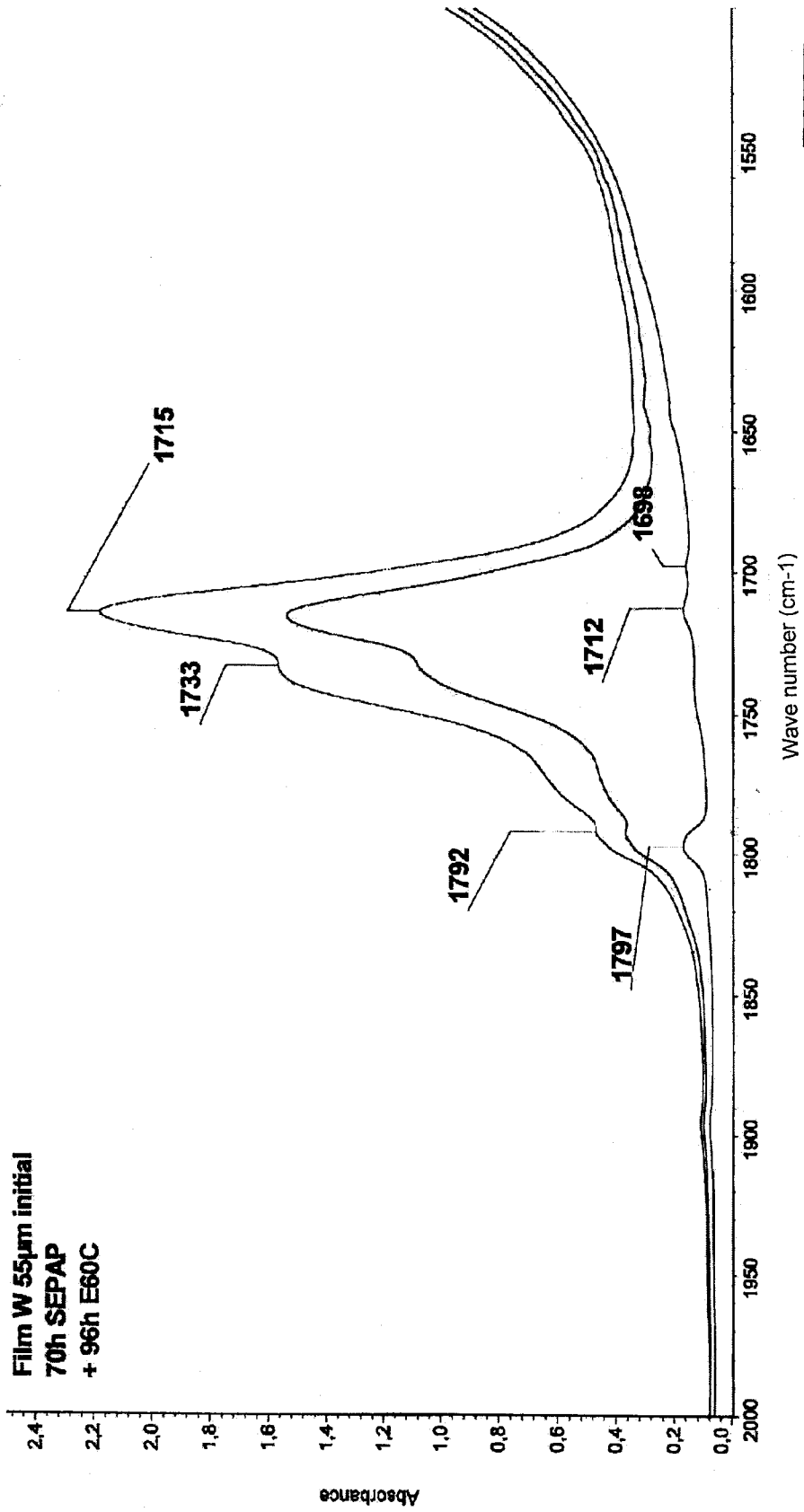
Analyse IRTF en mode transmission



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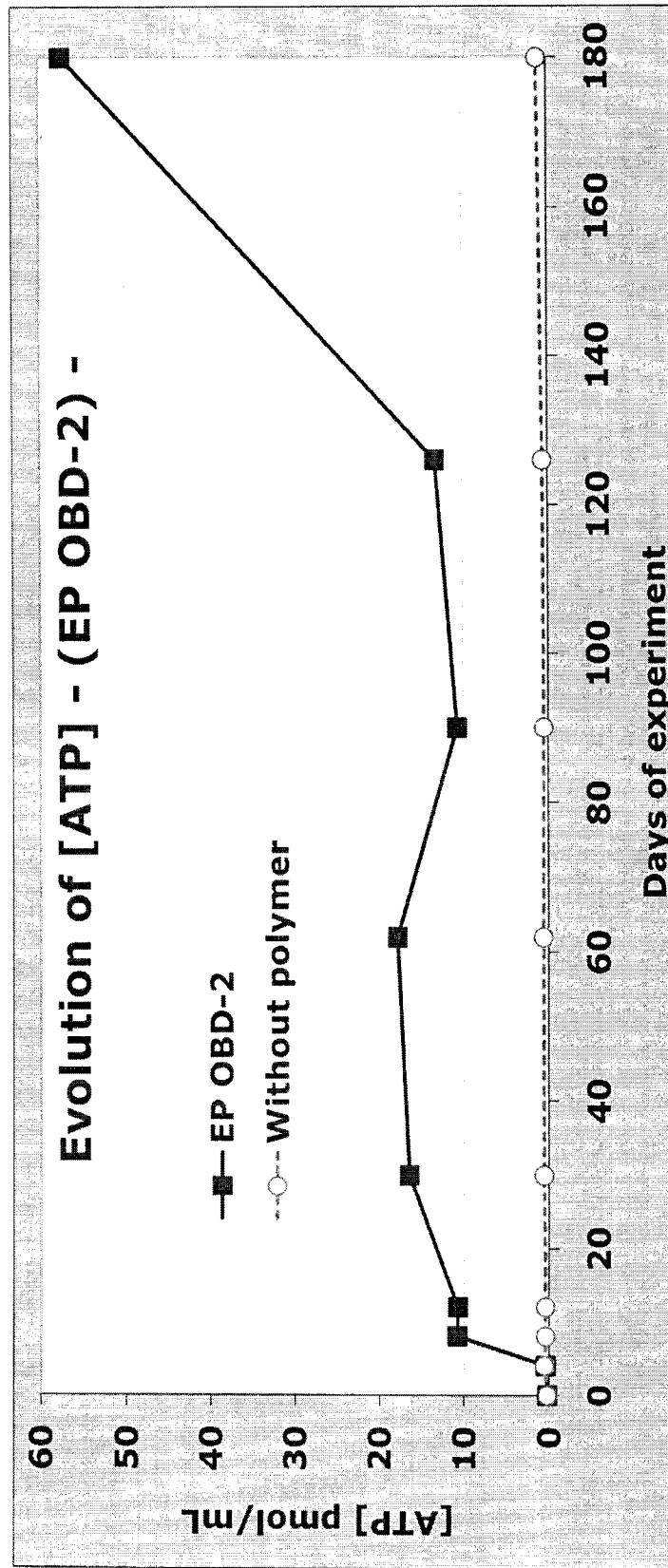
EP-ODB2 ENER plastics -
Analyse IRTF en mode transmission

Figure 9



10/11

FIGURE 10



INTERNATIONAL SEARCH REPORT

International application No.
PCT/CA2011/001385

<p>A. CLASSIFICATION OF SUBJECT MATTER IPC: <i>C08L 23/12</i> (2006.01), <i>C08J 3/22</i> (2006.01), <i>C08K 3/22</i> (2006.01), <i>C08K 3/26</i> (2006.01), <i>C08K 5/098</i> (2006.01), <i>C08K 5/13</i> (2006.01), <i>C08L 23/06</i> (2006.01) According to International Patent Classification (IPC) or to both national classification and IPC</p>																	
<p>B. FIELDS SEARCHED</p> <p>Minimum documentation searched (classification system followed by classification symbols) IPC: <i>C08L 23/12</i> (2006.01), <i>C08J 3/22</i> (2006.01), <i>C08K 3/22</i> (2006.01), <i>C08K 3/26</i> (2006.01), <i>C08K 5/098</i> (2006.01), <i>C08K 5/13</i> (2006.01), <i>C08L 23/06</i> (2006.01)</p> <p>Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched</p> <p>Electronic database(s) consulted during the international search (name of database(s) and, where practicable, search terms used) Canadian Patent Database, EPOQUE, Google Scholar Key words: pro-oxidant, phenolic antioxidant, polyolefin, filler</p>																	
<p>C. DOCUMENTS CONSIDERED TO BE RELEVANT</p> <table border="1"> <thead> <tr> <th>Category*</th> <th>Citation of document, with indication, where appropriate, of the relevant passages</th> <th>Relevant to claim No.</th> </tr> </thead> <tbody> <tr> <td>X</td> <td>CA 2,649,965 (GARDI, S. et al.) 22 November 2007 (22-11-2007) *see entire document</td> <td>1-14</td> </tr> <tr> <td>X</td> <td>CA 2,523,250 (MAENNLE, F. et al.) 04 November 2004 (04-11-2004) *see entire document</td> <td>1-7, 9-12, 14</td> </tr> <tr> <td>X</td> <td>US 5,258,422 (CHANG, P. I. et al.) 02 November 1993 (02-11-1993) *see entire document</td> <td>1-7, 9-12, 14</td> </tr> <tr> <td>X</td> <td>US 3,921,333 (CLENDINNING, R. A. et al.) 25 November 1975 (25-11-1975) *see entire document</td> <td>1-7, 9-12, 14</td> </tr> </tbody> </table>			Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.	X	CA 2,649,965 (GARDI, S. et al.) 22 November 2007 (22-11-2007) *see entire document	1-14	X	CA 2,523,250 (MAENNLE, F. et al.) 04 November 2004 (04-11-2004) *see entire document	1-7, 9-12, 14	X	US 5,258,422 (CHANG, P. I. et al.) 02 November 1993 (02-11-1993) *see entire document	1-7, 9-12, 14	X	US 3,921,333 (CLENDINNING, R. A. et al.) 25 November 1975 (25-11-1975) *see entire document	1-7, 9-12, 14
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.															
X	CA 2,649,965 (GARDI, S. et al.) 22 November 2007 (22-11-2007) *see entire document	1-14															
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<p><input type="checkbox"/> Further documents are listed in the continuation of Box C. <input checked="" type="checkbox"/> See patent family annex.</p> <table border="1"> <tbody> <tr> <td>* Special categories of cited documents :</td> <td>"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</td> </tr> <tr> <td>"A" document defining the general state of the art which is not considered to be of particular relevance</td> <td>"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone</td> </tr> <tr> <td>"E" earlier application or patent but published on or after the international filing date</td> <td>"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art</td> </tr> <tr> <td>"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</td> <td>"&" document member of the same patent family</td> </tr> <tr> <td>"O" document referring to an oral disclosure, use, exhibition or other means</td> <td></td> </tr> <tr> <td>"P" document published prior to the international filing date but later than the priority date claimed</td> <td></td> </tr> </tbody> </table>			* Special categories of cited documents :	"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention	"A" document defining the general state of the art which is not considered to be of particular relevance	"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone	"E" earlier application or patent but published on or after the international filing date	"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art	"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	"&" document member of the same patent family	"O" document referring to an oral disclosure, use, exhibition or other means		"P" document published prior to the international filing date but later than the priority date claimed				
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<p>Date of the actual completion of the international search 27 February 2012 (27-02-2012)</p>		<p>Date of mailing of the international search report 16 April 2012 (16-04-2012)</p>															
<p>Name and mailing address of the ISA/CA Canadian Intellectual Property Office Place du Portage I, C114 - 1st Floor, Box PCT 50 Victoria Street Gatineau, Quebec K1A 0C9 Facsimile No.: 001-819-953-2476</p>		<p>Authorized officer Erin Todd (819) 934-6733</p>															

INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.
PCT/CA2011/001385

Patent Document Cited in Search Report	Publication Date	Patent Family Member(s)	Publication Date
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