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(57) Abstract: This disclosure relates to an oxygen scavenging composition and a packaging article prepared there from. The oxygen scavenging composition comprises a polyester component; an organic oxidizable polymeric component; and a sulfonated metal oxidation catalyst. The oxygen scavenging effect of the packaging article is controllable within a time period ranging between zero minutes and the lifetime of the packaging article in the configuration in which it is packed.



A PACKAGING ARTICLE

Field of the Disclosure:

The present disclosure relates to an oxygen scavenging composition and an article prepared there from.

Background

Exposure of oxygen to products such as Foods, beverages, and medicines promotes quicker degradation or de-naturation of these products. The shelf-life of these products is therefore directly governed by the container that is used for packaging. Apart from deterioration, flavor retention is another reason which calls for a special precaution while selecting a suitable container for packaging these products. However, mere selection of a container with high barrier may not be sufficient to ensure better shelf-life of these products. Even the manner in which these containers are sealed and packed also plays an important role in ensuring a better shelf-life of these products. This is particularly important in cases where the containers are plastic containers.

Therefore, it becomes imperative to limit the oxygen exposure of the oxygen-sensitive food articles in the packaging system for ensuring a better shelf life. Such packaging thus keeps the article in inventory longer, thereby reducing restocking costs, and costs incurred from waste.

The commonly known approaches for minimizing the oxygen exposure are the use of a passive barrier, use of an active barrier or use of a dual barrier material in the packaging material. The materials used for imparting passive barrier are known in the art and there are several disadvantages associated with them.

The approach involving the use of active barrier substances, which are also called as scavenging agents, is being increasingly preferred. This approach to protect oxygen-sensitive products is two-fold; besides providing barrier to ingress of external oxygen, the oxygen scavenging agent in the packaging consumes the oxygen contained in the packaging article either as residual dissolved oxygen in the packaged goods and/or in the void space within the packaging article not occupied by the packaged goods: i.e. "headspace". Headspace oxygen scavenging normally involves the removal of large quantities of oxygen from the interior of the package.

Serious drawbacks with the use of oxygen scavenging agent is that an extended "**induction time**" is often present before full scavenging activity

is achieved. Induction time is the time from the formation of the article until the time the oxygen transmission rate is significantly reduced.

Prior art (US7049359 and WO 2010/096430) indicates that the oxygen scavenging capacity may appear only after ageing of the blend, as the oxidation catalyst may be embedded in the wrong phase, i.e. away from the scavenger polymer phase.

It is desired that there is no ingress of oxygen through the packaging article into the contained oxygen sensitive product, and it is preferable that the packaging article is able to remove the oxygen from the contained-product/inside-environment, from a time right after the packaging of the oxygen sensitive item.

Alternative available options of active barrier based packaging products allow ingress of oxygen for about a week or even longer even after sealing of the item in the packaging unit. This time period is too long a time period and during this period the packaged oxygen sensitive food is quite vulnerable to the onslaught of oxygen exposure during this period.

It is therefore desirable to have a packaging article that allows no ingress/removes ingress of oxygen right from the time of packaging/sealing of the oxygen sensitive item inside the packaging unit. Furthermore, there also is a need for a packaging article wherein the oxidation catalyst is in a

form such that the possibility of leaching of the oxidation metal catalyst is completely obviated.

Objects of the Invention:

It is an object of the present invention to provide a non-toxic packaging article for oxygen sensitive products.

It is another object of the present invention to provide a packaging article that increases the shelf-life of oxygen sensitive products.

It is still another object of the present invention to provide a packaging article which efficiently protects the oxygen sensitive products from the exposure of oxygen right from the time of its packaging.

Definitions:

As used in the present disclosure, the following words and phrases are generally intended to have the meanings as set forth below, except to the extent that the context in which they are used to indicate otherwise.

PET means a polymer having primarily ethylene terephthalate repeat units..

CO-PET means polymer having other repeat units apart from ethylene terephthalate.

Co-PTSA means cobalt salt of p-Toluenesulfonic acid.

R-PET means recycled PET

The oxygen scavenging composition/article means a composition or article which consumes, depletes or reduces the amount of oxygen from a given environment.

The term “onset of oxygen scavenging activity” means beginning of removal of oxygen from the container, as opposed to ingress of external oxygen into the container.

Throughout this disclosure the word “comprise”, or variations such as “comprises” or “comprising”, will be understood to imply the inclusion of a stated element, integer or step, or group of elements, integers or steps, but not the exclusion of any other element, integer or step, or group of elements, integers or steps.

The use of the expression “at least” or “at least one” suggests the use of one or more elements or ingredients or quantities, as the use may be in the embodiment of the invention to achieve one or more of the desired objects or results.

When an amount, concentration, or other value or parameter is given as a range, or a list of upper and lower values, this is to be understood as specifically disclosing all ranges formed from any pair of any upper and lower range limits, regardless of whether ranges are separately disclosed. Where a range of numerical values is recited herein, unless otherwise stated, the range is intended to include the endpoints thereof, and all integers and fractions within the range. It is not intended that the scope of the present disclosure be limited to the specific values recited when defining a range. When the term "about" is used in describing a value or an end-point of a range, the disclosure should be understood to include the specific value or end-point referred to.

Summary:

In accordance with one aspect of the present disclosure there is provided an oxygen scavenging composition comprising:

a polyester component;
an organic oxidizable polymeric component; and
a sulfonated metal oxidation catalyst.

In accordance with one embodiment of the disclosure the oxygen scavenging composition of the present disclosure comprises a compatibilizing agent

prepared by copolymerizing a sulfoisophthalic acid based comonomer having the sulfonate group neutralized by a counter cation and PET, said counter cation being selected from the group consisting of alkali metal cation, alkaline earth metal cation, VIIIb group metal cation and non-metallic cation.

Typically, the polyester component is at least one selected from the group consisting of PET, CO-PET and R-PET.

Typically, the sulfonated oxidation metal catalyst is cobalt salt of one or more organic sulfonic acids represented by the formula



Wherein R is an alkyl, substituted alkyl, aryl or substituted aryl moiety.

In accordance with an exemplary embodiment of the present disclosure the sulfonated oxidation metal catalyst is cobalt salt of p-Toluenesulfonic acid.

Typically, the cobalt content of the composition is greater than 5 ppm.

Alternatively, the cobalt content of the composition is greater than 20 ppm.

In accordance with an exemplary embodiment of the present disclosure the organic oxidizable polymeric component is poly(m-xylene adipamide), the proportion of poly(m-xylene adipamide) being in the range of about 0.2 to 15 % with respect to the mass of the packaging article.

Typically, the organic oxidizable polymeric component is an olefin containing segment containing at least one olefinic unsaturation, the proportion of the olefin containing segment being in the range of about 0.2% to 8% with respect to the mass of the packaging article.

Typically, the proportion of the polyester component in the packaging article is in the range of about 85 % to about 99.8% with respect to the mass of the composition.

Typically, the organic oxidizable polymeric component comprises partially aromatic polyamide, the proportion of the partially aromatic polyamide being in the range of about 0.2% to 15% with respect to the mass of the composition.

In accordance with another aspect of the present invention there is provided an oxygen scavenging packaging article wherein the commencement of the oxygen scavenging effect is controllable within a time period ranging between zero minutes and the lifetime of the packaging article in the configuration in which it is packed with an intended item and sealed, wherein said packaging article is selected from the group consisting of monolayer bottle, sachet, pouch, blister container and thermoformed container, and wherein said oxygen scavenging effect is exhibited from the

commencement of the oxygen scavenging effect through the lifetime of the article.

Brief Description of the Drawing:

Figure 1 provides comparative oxygen scavenging activity of three different oxygen scavenging compositions comprising 5% polyamide + 25ppm cobalt, 5% polyamide + 50 ppm cobalt and 5% polyamide+ 25ppm cobalt + a compatibilizer prepared in accordance with this disclosure.

Description:

In accordance with one aspect of the present invention there is provided oxygen scavenging composition comprising:

- a polyester component ;
- an organic oxidizable polymer; and
- a sulfonated oxidation metal catalyst.

PET remains to be a preferred choice when it comes to packaging material.

In one of the embodiments, the polyester component in the composition is a homopolymeric condensate of polyethylene terephthalate. In another

embodiment, the polyester component is copolymer of PET comprising homopolymeric condensate of polyethylene terephthalate and ethylene isophthalate.

In one of the embodiments the polyester component of the composition of the present invention is recycled PET.

Intrinsic viscosity of the polyester component is an important parameter in the manufacturing of various packaging materials, especially bottles. Depending on the end application, the polyester component of varying intrinsic viscosities is employed in the composition of the disclosure. The intrinsic viscosity of the polyester typically varies between 0.6 and 1.00

The polyester component constitutes the bulk of the oxygen scavenging composition and its proportion in the composition varies between 85% to 99%.

The organic oxidizable polymer present in the oxygen scavenging composition of the present invention is an olefin containing segment containing at least one olefinic unsaturation. The proportion of the olefin containing segment typically varies between 0.2% and 8% with respect to the mass of the composition. The organic oxidizable polymeric component typically comprises partially aromatic polyamide. The proportion of the

partially aromatic polyamide ranges between 0.2% and 15% with respect to the mass of the packaging article.

In accordance with an exemplary embodiment of the present invention poly(m-xylene adipamide) is used as the organic oxidizable polymer. The proportion of the oxidizable polymer typically varies between 0.2 and 15%

Another essential component of the present invention is the oxidation metal catalyst. The inventors of the present disclosure in their pursuit for an improved metal oxidation catalyst that offers superior oxygen scavenging activity with a relatively shorter onset of action carried out several experiments and surprisingly found that cobalt salt of p-Toluenesulfonic acid offers improved oxygen scavenging activity and that too with a very short onset of action. In the context of the present disclosure the term onset of oxygen scavenging activity means the time period required for the oxygen scavenging material to actually start depleting the oxygen from the environment from the time of the formation of the packaging article using the oxygen scavenging composition.

It has been observed that the onset of oxygen scavenging activity varies from few days to few weeks depending upon the composition of the oxygen scavenging material. In case of oxygen scavenging materials that employ conventionally known cobalt carboxylates or wherein cobalt is introduced in

the compositions through high molecular weight carriers such as fatty acids or alcohols, it takes few days for the oxygen scavenging material to actually start depleting the oxygen from the immediate environment.

The inventors of the present disclosure have surprisingly found that when cobalt is used in the form of a salt of an organic sulfonic acid as an oxidation metal catalyst it facilitates faster interaction with oxidizable polymer as the former remains more strongly bonded with the oxidizable polymer phase through physical bonding due to higher solubility parameter mismatch with the polyester phase. The cobalt salt of an organic sulfonic acid which is used as an oxidation metal catalyst in accordance with the present disclosure is represented by the formula:



wherein R is an alkyl, substituted alkyl, aryl or substituted aryl moiety.

In accordance with an exemplary embodiment of the present disclosure cobalt is used as Co-PTSA as an oxidation metal catalyst. Co-PTSA can be incorporated through different techniques which include incorporating it as coating on PET pellets, compounding with PET or it may also be incorporated as crystals or as tablets.

In accordance with one of the embodiments of the present disclosure, the oxygen scavenging composition of the present invention further comprises a

compatibilizer prepared by copolymerizing a sulfoisophthalic acid based comonomer having the sulfonate group neutralized by a counter cation and PET. Typically, the counter cation is an alkali metal or alkaline earth metal or VIIIb metal cation or non-metallic cation

The amount of cobalt in the oxygen scavenging composition of the present disclosure typically varies between 5 and 2000 ppm.

In accordance with another aspect of the present disclosure there is provided an oxygen scavenging packaging article wherein the commencement of oxygen scavenging effect is controllable within a time period ranging between zero minutes and the lifetime of the packaging article in the configuration in which it is packed with an intended item and sealed, wherein said packaging article is selected from the group consisting of monolayer bottle, sachet, pouch, blister container and thermoformed container, and wherein said oxygen scavenging effect is exhibited from the commencement of the oxygen scavenging effect through the lifetime of the article.

The oxygen scavenging effect is typically controlled either by varying the particular sulfonate salt or by systematically varying the concentration of a particular sulfonate salt.

The specification will now be described with the help of following non-limiting examples.

In the examples and the results that follow, the metal content of the samples was calculated from the amount added during polymerization and from the loading of the copolymerization product during injection molding. Similarly, MXD6 content of the samples was calculated from the loading of the MXD6 chips during injection molding. Intrinsic viscosity (IV) was obtained according to ASTM D4603-03 using 0.5 g/cc solution of the polymer in phenol-tetrachloroethane solvent (60:40 wt ratio, 30°C).

Oxygen transmission rate (OTR) was determined for the 0.36 mm thick film cut out from the bottle using Mocon Ox-Tran 2/21 modular system at 23°C and at 752 mmHg pressure. A mixture of 98% nitrogen and 2% hydrogen was used as carrier gas and 100% oxygen was used as the test gas.

Intrinsic viscosity (IV) was obtained according to ASTM D4603-03 using 0.5 g/cc solution of the polymer in phenol-tetrachloroethane solvent (60:40 wt ratio, 30°C).

Example 1:

Synthesis of Co-PTSA

80.9 g of p-toluenesulfonic acid (having the structure: $\text{H}^+\text{SO}_3^- - \text{C}_6\text{H}_4\text{CH}_3$) was dissolved in 1654 g water. 59.1 g of Cobalt acetate ($\text{CoAc}_2 \cdot 4\text{H}_2\text{O}$) was

added and heated for 1 hr from 20 to 140⁰C while employing a condenser to collect the by product acetic acid, leaving cobalt sulphonate salt solution in water. The completion of the reaction was indicated from an increase in pH. Water in the product was removed using vacuum.

Preparation of a polyester component

Slurry of purified terephthalic acid (6 kg) in ethylene glycol (4.5 kg) was esterified for 3.5 hrs up to 260⁰C at 2 bar nitrogen pressure. Cobalt sulfonate catalyst prepared in Example 1 (containing 2000 ppm Co) was added to the molten esterification product. After an interval of 20 minutes, antimony trioxide catalyst (300 ppm Sb in PET) dissolved in ethylene glycol 250 ml was added. The mixture temperature was increased to ~ 285⁰C, while gradually reducing the pressure over 45 minutes to 1 mm of Hg to obtain polymeric product. The product was extruded out of the reactor in the form of a strand, quenched in a water bath and sliced into chips containing 2000 ppm of cobalt. The intrinsic viscosity of the polymer was determined as 0.30 (ASTM D4603). The copolymer chips were crystallized at 140 °C in air oven.

Manufacture of barrier polyester bottles with 5% MXD6 + 50 ppm Co

0.2 kg of the catalyst master batch chips, and 0.4 kg of MXD6 chips and 7.4 kg of base polyester (poly (ethylene terephthalate-co-ethylene isophthalate), IV = 0.84 dL/g) were tumble mixed, dried at 160 °C for 5 hr, and injection molded using 2 cavity Arburg injection molding machine (Model Allrounder 420C) operated at cylinder temperature of 275-280 °C and runner temperature of 290-287 °C, into 48 g perform. These performs were blown into bottles of 1.5 L volume using SIDEL SB01 single cavity blow molding machine. The OTR value was found to be $0.025 \text{ cm}^3 \cdot \text{m}^{-2} \cdot \text{day}^{-1}$ measured for film thickness of 0.30 mm.

Manufacture of polyester bottles with 5%MXD6

0.4 kg of MXD6 chips and 7.6 kg of base polyester (poly(ethylene terephthalate-co-ethylene isophthalate), IV = 0.84 dL/g) were tumble mixed, dried at 160 °C for 5 hr, and injection molded using 2 cavity Arburg injection molding machine (Model Allrounder 420C) operated at cylinder temperature of 275-280 °C and runner temperature of 290-287 °C, into 48 g perform. These performs were blown into bottles of 1.5 L volume using SIDEL SB01 single cavity blow molding machine. The OTR value was found to be $1.98 \text{ cm}^3 \cdot \text{m}^{-2} \cdot \text{day}^{-1}$ measured for film thickness of 0.30 mm

Manufacture of 'Control' polyester bottles without Co and MXD6

8 kg of base polyester (poly(ethylene terephthalate-co-ethylene isophthalate), IV = 0.84 dL/g) were dried at 160 °C for 5 hr, and injection molded using 2 cavity Arburg injection molding machine (Model Allrounder 420C) operated at cylinder temperature of 275-280 °C and runner temperature of 290-287 °C, into 48 g perform. These performs were blown into bottles of 1.5 L volume using SIDEL SB01 single cavity blow molding machine. The OTR value was found to be 6.8 cm³.m⁻².day⁻¹ measured for film thickness of 0.30 mm

Manufacture of barrier polyester bottles with 5% MXD6 + 25 ppm Co

0.1 kg of the catalyst master batch chips and 0.4 kg of MXD6 chips and 7.5 kg of base polyester (poly(ethylene terephthalate-co-ethylene isophthalate), IV = 0.84 dL/g) were tumble mixed, dried at 160 °C for 5 hr, and injection molded using 2 cavity Arburg injection molding machine (Model Allrounder 420C) operated at cylinder temperature of 275-280 °C and runner temperature of 290-287 °C, into 48 g perform. These performs were blown into bottles of 1.5 L volume using SIDEL SB01 single cavity blow molding machine.

Measurement of O₂ concentration in the bottle using Oxysense 4000B

Three bottles (1.5L capacity) were taken viz. 5%MXD6 + 50 ppm Co, and Control PET , 5% MXD6 + 25 ppm Co (as prepared using example 4,6,7

above) and the oxygen sensor called O₂xyDot was fixed inside the bottle using transparent glue. After curing, de-mineralized water was filled inside the bottle. Nitrogen was purged and the Oxygen level in the bottle was brought to optimum level before it was capped with beri-cap. The bottles were kept in conditioned room at 23°C. The OxySense 4000B non-invasive oxygen analyzer was used to measure the oxygen concentration inside the bottle. The following tables below provide change in concentration of oxygen for the different formulations.

Oxygen concentration in

ppb

Table 1

Days	5%MXD6 + 50 ppm Co	5%MXD6 + 25 ppm Co
0	1395	1200
1	1150	1090
2	935	1001
3	830	921
4	735	846
5	645	774

12	415	523
14	362	480
16	330	451
19	284	370
27	174	201
32	106	111

Table 2

Day	5% MXD6 + 25 ppm Co+ compatibilizer
0	1200
1	1086
2	1001
6	704
10	567
14	480
16	451
23	280
24	256
26	219
30	147
31	114
32	111
33	96
34	85
35	66
37	47
39	16
40	15

Example 2:

Recipe prepared by following the procedure as provided in example 1 except that the amount of MXD6 was 5% and the amount of cobalt was 38 ppm.

Time in min	O ₂ ppb - Oxysense
0	7406
30	7009
60	6702
120	6362

The numerical values given for various physical parameters, dimensions and quantities are only approximate values and it is envisaged that the values higher than the numerical value assigned to the physical parameters, dimensions and quantities fall within the scope of the invention and the claims unless there is a statement in the specification to the contrary.

While considerable emphasis has been placed herein on the specific ingredients of the preferred formulation, it will be appreciated that many additional ingredients can be added and that many changes can be made in the preferred formulation without departing from the principles of the invention. These and other changes in the preferred formulation of the invention will be apparent to those skilled in the art from the disclosure

herein, whereby it is to be distinctly understood that the foregoing descriptive matter is to be interpreted merely as illustrative of the invention and not as a limitation.

Claims:

1. An oxygen scavenging composition comprising:
 - a polyester component;
 - an organic oxidizable polymeric component; and
 - a sulfonated metal oxidation catalyst.
2. An oxygen scavenging composition as claimed in claim 1 further comprising a compatibilizing agent prepared by copolymerizing a sulfoisophthalic acid based comonomer having the sulfonate group neutralized by a counter cation and PET, said counter cation being selected from the group consisting of alkali metal cation, alkaline earth metal cation, VIIIb group metal cation and non-metallic cation.
3. An oxygen scavenging composition as claimed in claim 1, wherein the polyester component is at least one selected from the group consisting of PET, CO-PET, R-PET.
4. An oxygen scavenging composition as claimed in claim 1, wherein the sulfonated oxidation metal catalyst is cobalt salt of one or more organic sulfonic acids represented by the formula:



wherein R is an alkyl, substituted alkyl, aryl or substituted aryl moiety.

5. An oxygen scavenging composition as claimed in claim 1, wherein the sulfonated oxidation metal catalyst is cobalt salt of p-Toluenesulfonic acid.
6. An oxygen scavenging composition as claimed in claims 4 or 5 , wherein the cobalt content of the composition is greater than 5 ppm.
7. An oxygen scavenging composition as claimed in claim 4 or 5, wherein the cobalt content of the composition is greater than 20 ppm.
8. An oxygen scavenging composition as claimed in claim 1, wherein the organic oxidizable polymeric component is poly(m-xylene adipamide), the proportion of poly(m-xylene adipamide) being in the range of about 0.2 to 15 % with respect to the mass of the packaging article
9. An oxygen scavenging composition as claimed in claim 1, wherein the organic oxidizable polymeric component is an olefin containing segment containing at least one olefinic unsaturation, the proportion of the olefin containing segment being in the range of about 0.2% to 8% with respect to the mass of the packaging article.
10. An oxygen scavenging composition as claimed in claim 1, wherein the proportion of the polyester component in the packaging article is in the range of about 85 % to about 99.8% with respect to the mass of the composition.

11. An oxygen scavenging composition as claimed in claim 1, wherein the organic oxidizable polymeric component comprises partially aromatic polyamide, the proportion of the partially aromatic polyamide being in the range of about 0.2% to 15% with respect to the mass of the composition.
12. An oxygen scavenging packaging article wherein the commencement of oxygen scavenging effect is controllable within a time period ranging between zero minutes and the lifetime of the packaging article in the configuration in which it is packed with an intended item and sealed, wherein said packaging article is selected from the group consisting of monolayer bottle, sachet, pouch, blister container and thermoformed container, and wherein said oxygen scavenging effect is exhibited from the commencement of the oxygen scavenging effect through the lifetime of the article.

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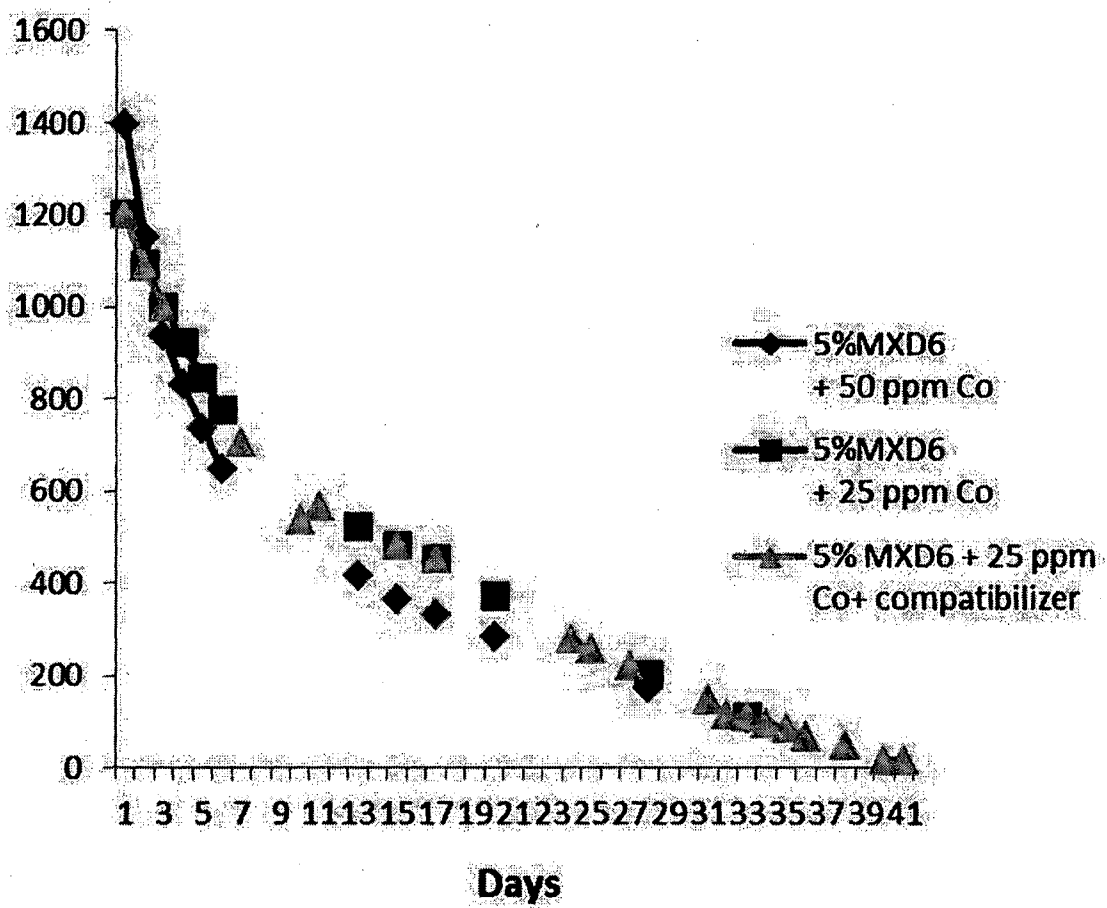


FIGURE. 1