Title: BIODEGRADABLE POLYESTER COMPOSITION

Abstract: Disclosed is a biodegradable polyester composition, where the weight content of a cyclic ester as structurally represented by formula (I) is 100 ppm to 950 ppm based on the total weight of the biodegradable polyester composition, and the weight content of cyclopentanone is 0.5 ppm to 85 ppm based on the total weight of the biodegradable polyester composition. The present invention, by adding the cyclic ester and the cyclopentanone to the composition, controls the content of the ester and that of the cyclopentanone in the composition within a certain range, thus increasing the thermal-oxidative aging resistance of the biodegradable polyester composition; also, a film produced by blow molding or a workpiece produced by injection molding produces a small amount of surface precipitates after being boiled in 95% ethanol at 40 °C for 240 h, has great surface appearance properties, and improves the degree of lubrication for the film during the blow molding process of the biodegradable polyester composition, when the rate of film blowing is at 176 kg/h, the range of the thickness of the film is < 0.2 μm, and the relative deviation in the thickness of the film is < 1%, thus ensuring the stability of film bubbles and the continuity of film blowing.
本发明公开了一种可生物降解聚酯组合物，其中，基于可生物降解聚酯组合物的总重量，具有如式(1)所示结构的环状酯化物的重量含量为100ppm-950ppm；基于可生物降解聚酯组合物的总重量，环戊酮的重量含量为0.5ppm-85ppm。本发明通过在组合物中添加环状酯化物和环戊酮，将组合物中环状酯化物和环戊酮的含量控制在一定范围内，可以极大提高可生物降解聚酯组合物的抗热氧老化性能，并且吹塑所得的薄膜或注塑所得的制件，经95%的乙醇40℃煮240h，表面析出物少，具有优异的表面外观性能，且可以改善可生物降解聚酯组合物在吹塑过程中膜的润滑程度，在吹膜速度为176Kg/h时，膜厚极差<0.2μm，膜厚相对偏差<1%，保证了膜泡的稳定性和吹膜的连续性。
BIODEGRADABLE POLYESTER COMPOSITION

TECHNICAL FIELD

The present invention belongs to a field of modification of macromolecule materials, and specifically relates to a biodegradable polyester composition with excellent anti-thermal oxidative aging property, surface appearance property and bubble stability.

BACKGROUND

Biodegradable polyester is a kind of macromolecule material using biological resources as raw materials. With respect to a petroleum-based polymer using petrochemical resources as raw materials, the biodegradable polyester can be degraded during a process of biological or biochemical effect or in a biological environment, being a very active degradable material in the present biodegradable plastic research and one of the best degradable materials in market application.

At present, biodegradable polyester film takes one of the most important application fields of biodegradable polyester, mainly including grocery bag, garbage bag, shopping bag, mulching film and the like. During blow molding process of the biodegradable polyester in preparing films, it frequently appears that the film is not lubricating enough to adhere to a roll or is too lubricating to roll up. Thus it results in poor bubble stability and large range of film thickness during film blowing which severely affect a continuity of film blowing. In CN 101622311 A, by adding 0.05-5wt% of biodiesel into a biodegradable polyester mixture, a viscosity of the polyester mixture is decreased, to some extent leading to less adherence of film to the roll, which guarantees the continuity of film blowing. However, a decrease of the viscosity of the polyester mixture indicates that an addition of biodiesel damages performances of the polyester to some extent, resulting in an increased melting index and a decreased viscosity of the polyester mixture.

In addition, owing to effects of microorganism, illumination, radiation, atmosphere and contacted physical environment, a molding product prepared with the biodegradable polyester is relatively easy to age and degrade during storage and use, which hugely influences service performance of the product. A conventional method that solves the aging and the degradation of macromolecule material includes adding an antioxidant, a UV absorbent, a HALS stabilizer and the like into the material. For example, patent WO 2009/071475 discloses a mulching film of
polyethylene which contains hydroxyphenyltriazines as the stabilizer. CN 103687902 introduces the UV absorbent and the HALS stabilizer, or a light stabilizer combining both, for providing the mulching film with UV stability. Although the above stabilizers can provide certain stabilization, they are definitely not satisfactory for a transparent mulching film, especially for that with a relatively low wall thickness.

Besides, under a condition that the molding product prepared with the biodegradable polyester composition is digested with 95% ethanol, there will be a precipitate separating out of a surface of a film or a part, which thus influences a surface appearance property of the film or the part.

The present invention surprisingly finds by research that by adding a trace amount of a cyclic ester compound and cyclopentanone into the biodegradable polyester composition, an anti-oxidative property of the biodegradable polyester composition can be greatly enhanced, and meanwhile it guarantees that the biodegradable polyester composition has an excellent surface appearance property and enables the biodegradable polyester composition to have obviously improved film blowing properties. When a velocity of film blowing is relatively high, it presents good bubble stability as well as relatively small range of film thickness and guarantees the continuity of film blowing production.

**SUMMARY OF THE INVENTION**

An object of the present invention is to provide a biodegradable polyester composition. By adding a trace amount of a cyclic ester compound and cyclopentanone into the composition, the prepared biodegradable polyester composition may have excellent anti-thermal oxidative aging property, surface appearance property and bubble stability.

The present invention is realized by following technical solution:

a biodegradable polyester composition comprises following components in parts by weight:

i) 60 to 100 parts of biodegradable aliphatic-aromatic polyester;

ii) 0 to 40 parts of polylactic acid;

iii) 0 to 35 parts of an organic filler and/or an inorganic filler;

iv) 0 to 1 part of a copolymer which contains epoxy group and is based on styrene, acrylate and/or methacrylate.

In particular, based on a total weight of the biodegradable polyester composition, a weight
content of a cyclic ester compound having a structure shown as formula (I) is 100ppm-950ppm;

![formula (I)](image)

and based on the total weight of the biodegradable polyester composition, a weight content of cyclopentanone is 0.5ppm-85ppm.

Preferably, based on the total weight of the biodegradable polyester composition, the weight content of the cyclic ester compound is 160ppm-750ppm, preferably 210ppm-540ppm; and the weight content of cyclopentanone is 5ppm-50ppm, preferably 10ppm-35ppm.

Preferably, the biodegradable polyester composition comprises the following components in parts by weight:

i) 65 to 95 parts of the biodegradable aliphatic-aromatic polyester;

ii) 5 to 35 parts of the polylactic acid;

iii) 5 to 25 parts of the organic filler and/or the inorganic filler;

iv) 0.02 to 0.5 part of the copolymer which contains epoxy group and is based on styrene, acrylate and/or methacrylate.

The biodegradable aliphatic-aromatic polyester is one or more of poly(butyleneadipate-co-terephthalate) (PBAT), poly(butylenesuccinate -co-terephthalate) (PBST) and poly(butyleneadipate-co-terephthalate) (PBSt).

Addition of the cyclic ester compound helps to extend a service life of the biodegradable polyester composition. Cyclopentanone added into the biodegradable polyester plays a lubricant-like part. In research, the present invention found that controlling the content of the cyclic ester compound as 100ppm-950ppm and the content of cyclopentanone as 0.5ppm-85ppm in the biodegradable polyester composition, may not only guarantee the biodegradable polyester composition having good anti-thermal oxidative aging property, but also guarantee the prepared film or the prepared part having excellent surface appearance property. Besides, a lubrication degree of the film during a blow molding process of the biodegradable polyester may be improved. When the velocity of film blowing is 176 Kg/h, a range of a film thickness is less than 0.2μm and a relative deviation of the film thickness is less than 1%. The bubble stability and continuity of film blowing are guaranteed.
However, if the content of the cyclic ester compound in the biodegradable polyester composition is too high, the cyclic ester compound will separate out of a surface of a film or a part under a condition of being digested with 95% ethanol, which influences the surface appearance property of the film or the part. If the content of cyclopentanone in the biodegradable polyester composition is too high, during a film blowing process at high velocity, a film is too lubricating to roll up well on a roll, and also it would results in an unstable film bubble. Therefore, based on the total weight of the biodegradable polyester composition, the weight content of the cyclic ester compound is preferably 160ppm-750ppm, and more preferably 210ppm-540ppm; and the weight content of cyclopentanone is preferably 5ppm-50ppm, and more preferably 10ppm-35ppm.

The organic filler is selected from a group consisting of natural starch, plasticized starch, modified starch, natural fiber and wood flour, or a mixture thereof. The inorganic filler is selected from a group consisting of talcum powder, montmorillonite, kaolin, chalk, calcium carbonate, graphite, gypsum, conductive carbon black, calcium chloride, ferric oxide, dolomite, silicon dioxide, wollastonite, titanium dioxide, silicate, mica, glass fiber and mineral fiber, or a mixture thereof.

A route of acquiring the cyclic ester compound and cyclopentanone in the present invention may be by means of adding the cyclic ester compound and cyclopentanone directly during blending, extruding and processing the biodegradable polyester composition.

According to different needs of use, the biodegradable polyester composition according to the present invention may be further added with 0 to 4 parts of at least one of following substances: plasticizer, release agent, surfactant, wax, antistatic agent, pigment, UV absorbent, UV stabilizer and other plastic additives.

The plasticizer is one of or a mixture of two or more of citric esters, glycerol, epoxidized soybean oil and the like.

The release agent is one of or a mixture of two or more of silicone oil, paraffin, white mineral oil and Vaseline.

The surfactant is one of or a mixture of two or more of polysorbate, palmitate and laurate.

The wax is one of or a mixture of two or more of erucamide, stearamide, behenamide, beeswax and beeswax ester.

The antistatic agent is a permanent antistatic agent, specifically listed as one of or a mixture of two or more of PELESTAT-230, PELESTAT-6500 and SUNNICO ASA-2500.
The pigment is one of or a mixture of two or more of carbon black, black masterbatch, titanium
dioxide, zinc sulfide, phthalocyanine blue and fluorescent orange.
The UV adsorbent is one or more of UV-944, UV-234, UV531 and UV326.
The UV stabilizer is one or more of UV-123, UV-3896 and UV-328.
The other plastic additives may be nucleating agent, antifogging agent and the like.
The biodegradable polyester composition according to the present invention may be used for preparing shopping bag, compost bag, mulching film, protective cover film, silo film, film strip, fabric, non-fabric, textile, fishing net, bearing bag, garbage bag and the like.
Compared to the prior art, the present invention has following beneficial effects:
In the present invention, by adding the cyclic ester compound and cyclopentanone into the composition and controlling the content of the cyclic ester compound in a range of 100ppm-950ppm and the content of cyclopentanone in a range of 0.5ppm-85ppm in the composition, not only the anti-thermal oxidative aging property of the biodegradable polyester composition may be greatly improved, but meanwhile the film prepared by blow molding or the part prepared by injection molding has little precipitate separating out of the surface and has excellent surface appearance property after being digested with 95% ethanol at 40°C for 240 hours. In addition, the lubrication degree of film during the blow molding process of the biodegradable polyester may be improved. When the velocity of film blowing is 176 Kg/h, the range of film thickness is less than 0.2μm and the relative deviation of film thickness is less than 1%. The bubble stability and the continuity of film blowing are guaranteed.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENT
The present invention will be further described below by way of specific implementations, and the following embodiments are preferred implementations of the present invention, but the implementations of the present invention are not limited by the following embodiments.
In the embodiments of the present invention, PBAT is chosen as a component i), ADR4370 is chosen as a component iv), starch is chosen as an organic filler, talcum powder and calcium carbonate are chosen as inorganic filler, citric esters is chosen as a plasticizer, palmitate is chosen as a surfactant, and stearamide is chosen as a wax. The above-mentioned promoters, PBAT, ADR4370, PLA, cyclic ester compound and cyclopentanone are commercially available.
Embodiments 1-16 and Comparative Embodiments 1-4:

According to formulas shown in Table 1, PBAT, PLA, ADR4370, organic fillers, inorganic fillers, promoters such as plasticizer, surfactant, wax and the like, a cyclic ester compound and cyclopentanone were mixed evenly and put into a single screw extruder. After being extruded at 140°C-240°C and prilled, the compositions were obtained. Data of performance tests is shown in Table 1.

Performance evaluation method:
(1) Evaluation method for an anti-thermal oxidative aging property of a biodegradable polyester composition:

the biodegradable polyester composition was sealed in a non-vacuum aluminum foil bag. The aluminum foil bag was put in an air dry oven at 70°C to perform a thermal oxidative aging test. Samples were taken every 3 days for testing a melting index (190°C/2.16kg, according to ISO 1133). When the melting index of the sample was beyond a normal melting index range of the biodegradable polyester composition, it indicated that an obvious thermal oxidative aging degradation had occurred in the biodegradable polyester composition. A test time that the obvious thermal oxidative aging degradation occurred in the biodegradable polyester composition was recorded. The shorter the test time was, the poorer the anti-thermal oxidative aging property of the biodegradable polyester composition was indicated.

(2) Evaluation method for a surface appearance property of a molding product:

A 2mm palette was injection molded and put into a solution of 95% ethanol at 95°C for being digested for 240 hours, followed by being placed in a standard laboratory with an atmosphere temperature of (23±2) °C and a relative humidity of 45%-55%. After the palette was adjusted for 48 hours, ∆L, a variation of L-value of the palette before treated and after treated, was measured via a colorimeter. The greater the ∆L was, the more the precipitate separated out of the surface and the poorer the surface appearance property was.

(3) Evaluation method for bubble stability of the biodegradable polyester composition:

The bubble stability of the biodegradable polyester composition during film blowing was evaluated by a method of a range of a film thickness and a relative deviation of the film thickness:

The film thickness was measured via a screw micrometer: 10 measurement points were taken evenly on a film of 1m*1m to measure the film thickness.

The range of the film thickness was a difference value between a maximum thickness and a
minimum thickness among the 10 measurement points.

The relative deviation of the film thickness was calculated according to the following formula:

\[
\text{relative deviation of film thickness}\% = \frac{\text{range of film thickness}}{\text{average film thickness}} \times 100\%
\]

wherein, the average film thickness was calculated as an arithmetic average of the thicknesses measured respectively at the 10 measurement points which were taken evenly on the film of 1m*1m.

(4) Determination method for the cyclic ester compound:

1.2000g of the biodegradable polyester composition was weighed accurately, added into a 25ml volumetric flask, and dissolved by adding chloroform. After the biodegradable polyester composition was dissolved completely, it was diluted to 25mL. A peak area of the cyclic ester compound in the prepared solution was measured by a GC-MS test. The content of the cyclic ester compound in the biodegradable polyester composition was calculated according to the peak area of the cyclic ester compound in the prepared solution and a standard curve of the cyclic ester compound. The standard curve of the cyclic ester compound was calibrated by a solution of the cyclic ester compound/chloroform.

Models and parameters for GC-MS are as follows:

Agilent Technologies 7693 AutoSampler;
Agilent Technologies 5975C inert MSD with Triple-Axis Detector;
Chromatographic column: J&W 122-5532 UI: 350°C; 30m x 250μm x 0.25μm
Sample injection: front SS injection port He (helium)
Sample production: vacuum.

(5) Determination method for cyclopentanone:

1) Drawing a standard curve of cyclopentanone:

Cyclopentanone/methanol solutions in concentrations of 0.0001 g/L, 0.001 g/L, 0.01 g/L, 0.1 g/L, 5.0 g/L, 10.0 g/L and 20.0 g/L were prepared, respectively. Peak areas of cyclopentanone in the cyclopentanone/methanol solutions in different concentrations were measured respectively by a static headspace method. The standard curve of cyclopentanone was drawn, with the peak area of cyclopentanone as an ordinate and the concentration of cyclopentanone as an abscissa.

2) Measurement of a content of cyclopentanone in the biodegradable polyester composition:

Approximate 1.2000g of biodegradable polyester composition was weighed accurately and put into a static headspace test flask; the peak area of cyclopentanone in the biodegradable polyester
composition was measured by the static headspace method; and the content of cyclopentanone in the biodegradable polyester composition was calculated according to the peak area of cyclopentanone in the biodegradable polyester composition and the standard curve of cyclopentanone.

Instrument models and parameters for static headspace are as follows:

Agilent Technologies 7697 Headspace Sampler;
Agilent Technologies 7890A GC System;
Chromatographic column: J&W 122-7032: 250°C; 30m x 250μm x 0.25μm
Sample injection: front SS injection port N₂
Sample production: front detector FID.

Conditions for static headspace test are as follows:

Temperature:
Heater: 105°C
Quantitative loop: 135°C
Transmission line: 165°C
Time:
Balance for sample bottle: 120 minutes
Duration for sample injection: 0.09 minute
GC circulation: 30 minutes.

Table 1 Test data of Comparative Embodiments 1-4 and Embodiments 1-16 (parts by weight)

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<th></th>
<th>Embodiment 1</th>
<th>Embodiment 2</th>
<th>Embodiment 3</th>
<th>Embodiment 4</th>
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<td>0.98</td>
<td>0.89</td>
<td>0.90</td>
<td>0.95</td>
</tr>
</tbody>
</table>

It can be seen from Table 1 that, in the biodegradable polyester composition, when the content of the cyclic ester compound is 100-950ppm and the content of cyclopentanone is 0.5-85ppm, the
biodegradable polyester composition has better anti-thermal oxidative aging property. Besides, after the biodegradable polyester composition is digested with 95% ethanol at 40°C for 240 hours, $\Delta L$ is less than 0.80, which indicates that the composition has excellent surface appearance property. When the velocity at film blowing is 176 Kg/h, the range of the film thickness is less than 0.2μm and the relative deviation of the film thickness is less than 1%. It indicates that the composition has better bubble stability. However, in Comparative Embodiment 1, in which the content of the cyclic ester compound is less than 100ppm and the content of cyclopentanone is 0, though $\Delta L$ of the composition is relatively low, the time for thermal oxidative aging of the composition is relatively short, the range of the film thickness is more than 0.2μm, and the relative deviation of the film thickness is more than 1%. In Comparative Embodiment 2, in which the content of the cyclic ester compound is over 950ppm and the content of cyclopentanone is over 85ppm, $\Delta L$ is more than 1.0, the range of the film thickness is more than 0.2μm, and the relative deviation of the film thickness is more than 1%. It indicates that there is more precipitate separating out of the surface, and the surface appearance property and the bubble stability of the composition are poor. In Comparative Embodiment 3, in which the velocity at film blowing is below 176 Kg/h and in Comparative Embodiment 4, in which the velocity at film blowing is over 176 Kg/h, the range of the film thickness is more than 0.2μm, the relative deviation of the film thickness is more than 1%, and the film bubble of the composition is relatively unstable either.
What is claimed:

1. A biodegradable polyester composition, characterized in that, it comprises following components in parts by weight:
   i) 60 to 100 parts of biodegradable aliphatic-aromatic polyester;
   ii) 0 to 40 parts of polylactic acid;
   iii) 0 to 35 parts of an organic filler and/or an inorganic filler;
   iv) 0 to 1 part of a copolymer which contains epoxy group and is based on styrene, acrylate and/or methacrylate; wherein, based on a total weight of the biodegradable polyester composition, a weight content of a cyclic ester compound having a structure shown as formula (I) is 100ppm-950ppm;

   \[
   \text{(I)}; \\
   \]

   and based on the total weight of the biodegradable polyester composition, a weight content of cyclopentanone is 0.5ppm-85ppm.

2. The biodegradable polyester composition according to claim 1, wherein based on the total weight of the biodegradable polyester composition, the weight content of the cyclic ester compound is 160ppm-750ppm, preferably 210ppm-540ppm; and the weight content of cyclopentanone is 5ppm-50ppm, preferably 10ppm-35ppm.

3. The biodegradable polyester composition according to claim 1 or 2, wherein it comprises the following components in parts by weight:
   i) 65 to 95 parts of the biodegradable aliphatic-aromatic polyester;
   ii) 5 to 35 parts of the polylactic acid;
   iii) 5 to 25 parts of the organic filler and/or the inorganic filler;
   iv) 0.02 to 0.5 part of the copolymer which contains epoxy group and is based on styrene, acrylate and/or methacrylate.

4. The biodegradable polyester composition according to any one of claims 1-3, wherein the
weight content of the cyclic ester compound is measured by following method: 1.2000g of the biodegradable polyester composition is weighed accurately, added into a 25ml volumetric flask, and dissolved by adding chloroform; after the biodegradable polyester composition is dissolved completely, it is diluted to 25ml; a peak area of the cyclic ester compound in the prepared solution is measured by a GC-MS test; the content of the cyclic ester compound in the biodegradable polyester composition is calculated according to the peak area of the cyclic ester compound in the prepared solution and a standard curve of the cyclic ester compound; and the standard curve of the cyclic ester compound is calibrated by a solution of the cyclic ester compound/chloroform;

the weight content of cyclopentanone is measured by following method: 1.2000g of the biodegradable polyester composition is weighed accurately and added into a static headspace test flask; a peak area of cyclopentanone in the biodegradable polyester composition is measured by a static headspace method; the content of cyclopentanone in the biodegradable polyester composition is calculated according to the peak area of cyclopentanone in the biodegradable polyester composition and a standard curve of cyclopentanone; and the standard curve of cyclopentanone is calibrated by a solution of cyclopentanone/methanol.

5. The biodegradable polyester composition according to any one of claims 1-3, wherein the biodegradable aliphatic-aromatic polyester is one or more of poly(butyleneadipate-co-terephthalate) (PBAT), poly(butylensesuccinate-co-terephthalate) (PBST) and poly(butyleneesbacate-co-terephthalate) (PBSeT).

6. The biodegradable polyester composition according to any one of claims 1-3, wherein the organic filler is selected from a group consisting of natural starch, plasticized starch, modified starch, natural fiber and wood flour, or a mixture thereof; and the inorganic filler is selected from a group consisting of talcum powder, montmorillonite, kaolin, chalk, calcium carbonate, graphite, gypsum, conductive carbon black, calcium chloride, ferric oxide, dolomite, silicon dioxide, wollastonite, titanium dioxide, silicate, mica, glass fiber and mineral fiber, or a mixture thereof.

7. The biodegradable polyester composition according to any one of claims 1-3, wherein it further comprises 0 to 4 parts of at least one of following substances: plasticizer, release agent,
surfactant, wax, antistatic agent, pigment, UV absorbent, UV stabilizer and other plastic additives.

8. The biodegradable polyester composition according to any one of claims 1-7, wherein the biodegradable polyester composition is sealed in a non-vacuum aluminum foil bag, and a time for thermal oxidative aging for putting the aluminum foil bag in an air dry oven at 70°C for conducting a thermal oxidative aging test is equal to or more than 10 days.

9. The biodegradable polyester composition according to any one of claims 1-7, wherein a ΔL value of the biodegradable polyester composition is less than 0.80 after being digested with 95% ethanol at 40°C for 240 hours.

10. The biodegradable polyester composition according to any one of claims 1-7, wherein when an extrusion velocity at film blowing of the biodegradable polyester composition is 176Kg/h, a range of a film thickness is less than 0.2μm and a relative deviation of the film thickness is less than 1%.